Laboratory Manual

Preface

The goal of this laboratory manual is to provide the student with an enjoyable experience that is both informative and challenging. It is our hope that the thrill of "seeing" theoretical principles "come to life" in the laboratory will enhance your overall understanding, as well as stimulate and develop critical thinking skills. Although there has always been considerable debate over the most effective methods of instruction; many agree that lecture topics supported by practical exercises are a proven model to create a successful learning environment. We embrace the spirit of this model in the *Forensic Chemistry Laboratory Manual*. Our approach is to correlate laboratory exercises to the theoretical and investigative principles of forensic chemistry. This will provide the student with valuable hands-on experience while adding clarity and continuity to lecture topics. This laboratory manual was written within the framework of each of the following areas.

Level and Audience

The *Forensic Chemistry Laboratory Manual* covers the laboratory component of a one semester class in forensic chemistry. It is not designed to be a stand-alone laboratory manual. It was specifically written to complement *Basic Principles in Forensic Chemistry*, the required text for a one semester class offered as part of our forensic certificate program. The course requires no prerequisite and is designed for students with little, if any, background in chemistry or forensics. The laboratory exercises are designed to provide practical experience in forensic investigative techniques. Emphasis is on the development of proper technique, handling of evidence, and interpretation of data and results. Although there is brief exposure to more sophisticated chemical principles, it is not the main focus of the manual. It is possible to perform complex procedures and reliably interpret results without an in-depth understanding of the complex reaction mechanisms involved.

Forensic Investigation

Investigative techniques are developed using evidence and test results from actual case studies. Students learn to exercise due diligence in the formulation of hypotheses, preparation of courtroom testimony, and presentation of results. "Moot" courts are used to develop proper courtroom demeanor, i.e., giving testimony, presenting evidence, jury interaction, etc. In addition, students are exposed to proper format and writing techniques typically used in the submission of case reports.

Stockroom Preparation

It was important to develop experiments that require chemicals and laboratory equipment that is both inexpensive and readily available.

Safety

Forensic chemical analysis is often performed by highly trained scientists in a controlled environment. Consequently, a few procedures used in forensic investigation have been intentionally omitted. These omissions may be based on reagent cost or availability, lack of analytical instrumentation or specialized glassware, or safety concerns when working with potentially dangerous chemicals. In these few cases, data and/or test results are provided for interpretation and presentation purposes only. Strict adherence to all safety procedures is highly stressed.

J.I. Khan et al., *Basic Principles of Forensic Chemistry*, DOI 10.1007/978-1-59745-437-7,

To The Student

We wish you success as you begin your journey into forensic chemistry. This manual was specifically designed to illustrate principles and techniques commonly used in forensic investigation. All too often, students fail to realize (or appreciate) the importance of practical laboratory experience and its relationship to theoretical principles. Lost in the topics presented in lecture are the long hours scientists spend in the laboratory developing and proving these theories. As you perform the experiments in this manual, you will learn proper experimental technique and develop an appreciation of the correlation that exists between theory and practice. We hope that your laboratory experience is enjoyable and informative.

Strict adherence to safety procedures will create a relatively safe and hazard free laboratory environment. It is the responsibility of each student to contribute to this safe environment by following all safety rules and regulations. The following list of safety procedures should be followed at all times. Your laboratory may have specific safety rules and practices, in addition to those below, that will be thoroughly explained by your laboratory instructor.

Wear approved safety glasses or goggles at all times
If you have contact lenses, nonvented goggles are required.

Prepare for lab
Read the experiment carefully and be aware of potential hazards before coming to lab.

Dress for lab
No loose fitting cloths, no shorts, no open-toed shoes, no tank tops. Tie long hair back to prevent contact with an open-flame. Lab coats or aprons are highly recommended and may be available in lab.

No food or drinks are allowed in the laboratory
Chemicals may adhere to food or liquids and may cause illness. If you take a break to eat, wash your hands thoroughly.

Know the location and proper use of all safety equipment
Survey the lab and locate all exits, safety showers, fire extinguishers, fire blankets, eye wash facilities, emergency gas shut-off valves, emergency phones, etc.

No unauthorized experiments
Closely follow the instructions given in this manual. Do not deviate from the procedures or techniques explained.

Practice proper laboratory behavior at all time
Do not take unnecessary risks. Playing or “horsing around” in lab will not be tolerated and will result in your expulsion.

Handle all chemicals properly
Never taste chemicals or inhale chemical vapors.
Avoid direct contact of chemicals with skin.
Never pour excess chemicals back into the original container.
Your instructor will advise you in the proper disposal of waste material.

Keep your work area neat and organized
Do not clutter your work area with excessive chemicals, glassware, and books.

Smoking is not permitted in the laboratory

Report all accidents to your instructor
Report all accidents, no matter how small, to your laboratory instructor. This information may be used to further develop and/or refine existing safety procedures.

If you have questions ASK YOUR INSTRUCTOR

Your instructor is a trained professional who is very familiar with the procedures performed in each experiment. If you have questions or require clarification, do not hesitate to ask.

The above represents a list of minimum safety precautions that should be followed to create a safe laboratory environment. Following these procedures will not guarantee a safe, accident-free environment, nor are they intended to represent a complete list of all safety rules and regulations. The possibility of accident and/or injury is always present in the lab; however, strict adherence to proper safety procedures at all times will minimize the risk for such occurrences.

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Experiment # 2

Name _____

Forensic/Scientific Investigation and Atomic Structure

Reference: Chapters 1 and 2

Objectives: Students will gain practical experience using the scientific method to develop conclusions. Students will become familiar with atomic structure and writing electron configurations for ground state neutral atoms and ions.

Introduction:

Scientific discoveries are usually the result of a systematic approach to a good idea or an unexplained observation. Although many variations of this "systematic" approach exist, it often involves a stepwise process called the *scientific method*. The *scientific method* is a procedure used to develop technical theories and generally includes four phases; *observation, hypothesis, experimentation, and theory*. It begins with the observation of some type of unexplained phenomenon. A possible cause of the observation is proposed during the hypothesis phase. Experiments are then specifically designed in order to prove the hypothesis. If experimental results do not support the hypothesis another possibility is considered and tested. If experimental results confirm the hypothesis, and are consistently reproducible, a formal explanation is developed and subsequently offered as a theory. The theory is then presented to the scientific community where it may be accepted or rejected. If accepted, it may become a *principle* or a *law*.

Part: A: Forensic/ Scientific Investigation

Observation: Different types of cell phone ring tones.

Hypothesis: No cell phones are currently set to identical ring tones

Experiment: Each student that has a cell phone will play their ring tone. Record the results.

Theory:

Part B: Atomic Structure

Complete the following table

Symbol	# Protons	# Neutrons	# Electrons	Atomic Mass
H				1
	19		19	
K^+				39
B		6		11
	5		2	
Cl			17	
Cl^-	17			

Part C: Electron Configuration

Write the electron configuration for each of the following.

Mg (atomic mass = 24)

Mg^{2+} (atomic mass = 24)

C (atomic mass = 12)

N^{3-} (atomic mass = 14)

Ca (atomic mass = 40)

Cl^{-} (atomic mass = 35.45)

H (atomic mass = 1)

Experiment # 3

Name _____

Properties of Elements

Reference: Chapter 3

Objectives: Students will distinguish the difference between a chemical property and a physical property. Students will gain practical experience in the use of properties to identify elements and compounds.

Materials: 1M hydroiodic acid (HI), 1M hydrochloric acid (HCl), 0.3M hypophosphorous acid (H_3PO_2), 0.5M sulfuric acid (H_2SO_4), 1M acetic acid ($HC_2H_3O_2$), 3% silver nitrate solution ($AgNO_3$), 1% iodine solution in water, chloroform ($CHCl_3$), starch (sugar), sodium nitrite ($NaNO_2$), test tubes, matches, and wax pencils.

Introduction:

There are fundamental properties associated with all forms of matter. These *distinguishing characteristics* may be physical or chemical in nature, and are commonly used to identify and classify a particular substance. A *physical property* is anything that can be measured or observed without changing chemical composition. The melting point and boiling point of water are examples of physical properties because these temperatures can be measured without changing the chemical composition of water. A *physical change* is a change in the state of matter, but not its chemical composition. There are three accepted states of matter; solid, liquid, and gas (although some would argue *plasma* is also a state). Other physical properties commonly used in the forensic identification of elements and compounds are: color, odor, density, solubility, conductivity, and sublimation.

Chemical properties are a measure of the ability of a substance to produce new substances, or simply, a measure of the reactivity of a substance. *Chemical changes* are transformations that produce products chemically and physically different from the starting material. A solution containing silver nitrate will produce a white precipitate (solid) in the presence of chloride ions and a yellow precipitate in the presence of iodide ions. These observations illustrate the chemical changes that result from the reactivity (chemical properties) of silver nitrate. Physical and chemical properties are commonly used to identify elements and compounds in the field of forensic science. Consequently, these properties may be used to support or reject specific parts of an investigation.

Part A:

Clean five test tubes and label each with the name of an acid shown below. Place 10 drops (approx. ½ ml) of the corresponding acid into each of the labeled test tubes. Add 1–2 drops of 3% silver nitrate solution ($AgNO_3$) to <u>each</u> test tube and observe the results. If a precipitate (solid) forms, record the color below next to the corresponding acid. If no precipitate is observed, write "none."

Acid	Color of Precipitate
Hydroiodic acid	
Hydrochloric acid	
Hypophosphorous acid	
Sulfuric acid	
Acetic acid	

Conclusion:

Part B:
Strike a matchstick on the following surfaces and record your observations.

Nature of Surface	Ignite (yes/no)
Wood	
Cement	
Metal	
Plastic	
Paper	
Course sandpaper (matchbox)	

Conclusion:

Part C:
Clean three test tubes and label each with a reagent shown below. Place ten drops (approx. ½ ml) of 1% iodine solution in each test tube and add the corresponding reagent. Record your observations.

Reagent	Add	Observation
Starch	Approx. "½ pea size" of starch	
Chloroform	1 ml chloroform	
Acetic acid	1 ml acetic acid	

Conclusion:

Experiment # 4

Name_____

Mixtures and Compounds

Reference: Chapters 1, 2, and 3

Objective: Students will observe common properties of mixtures and compounds.

Materials: methanol, DI water, sugar, salt, analytical balance, watch glass, and oven.

Introduction:

Elements and compounds may exist as pure substances or as mixtures. *Pure substances* contain only one component and have the same composition throughout, i.e., pure gold, pure sugar, pure water, etc. *Mixtures* contain two or more pure substances and may be homogeneous or heterogeneous. *Homogeneous mixtures* have the same composition and properties throughout. However, they are not pure substances because they contain more than one component. *Heterogeneous mixtures* have distinctly different properties within the mixture; water and sand would be an example. In any *binary solution* (a solution that contains only two components), the *solvent* is the component present in greatest amount and the *solute* is the component present in least amount.

The following mixtures will be provided. Classify each mixture by circling homogeneous or heterogeneous.

Solution #1	Sugar in water (sat.)	homogeneous or heterogeneous
Solution #2	Salt in water (sat.)	homogeneous or heterogeneous
Solution #3	Sugar in methanol (sat.)	homogeneous or heterogeneous
Solution #4	Salt in methanol (sat.)	homogeneous or heterogeneous

Part A:

Clean and dry four watch glasses and label each 1, 2, 3, or 4. Weigh each empty watch glass on an analytical balance and record the mass in the table below under "watch glass." Be sure to weigh the watch glasses after they are labeled! Place 1.0 ml of the corresponding solutions above on each of the labeled watch glasses, i.e., place 1.0 ml of solution #1 on watch glass labeled 1, etc. Using the same analytical balance that was used to weigh the empty watch glasses, carefully weigh the watch glasses containing each solution. Determine the mixture mass for each solution by subtracting the mass of the empty watch glass from the mass of the watch glass containing solution. Record the mass of each mixture in the table below under "mixture mass." Save the watch glasses containing each solution for Part B. Clean and dry two small test tubes and place one test tube into a small beaker. Place the test tube/beaker on the analytical balance and *tare* the balance (zero the balance with the test tube/beaker on the pan). Place 1.0 ml of water into the test tube and record the mass in the table below under Solvent Mass for water. Repeat the procedure using the other test tube and 1.0 ml of methanol. Record the mass below under Solvent Mass for methanol (water is the solvent in solutions 1 and 2, methanol is the solvent in solutions 3 and 4). Subtract the solvent mass from the mixture mass and record the difference in the table below under "solute mass."

Watch Glass	Mixture Mass	Solvent Mass	Solute mass
#1-		Water-	
#2-		Water-	
#3-		Methanol-	
#4-		Methanol-	

What is the mass of sugar in mixture #1?
What is the mass of salt in mixture #2?
What is the mass of sugar in mixture #3?
What is the mass of salt in mixture #4?

Did the mixture mass exceed the solvent mass in any solution? If so, explain.

Did the solvent mass exceed the mixture mass in any solution? If so, explain.

Did the solvent mass equal the mixture mass in any solution? If so, explain.

Explain your observed results using your knowledge of homogeneous and heterogeneous mixtures.

Part B:

Carefully place the watch glasses containing each solution in the oven and evaporate the solvent to dryness. When evaporation is complete, weigh each watch glass and record the mass in the table below under "watch glass/residue." Determine the mass of the residue by subtracting the mass of the empty watch glass (measured in Part A) from the mass of the watch glass/residue. The residue is the actual mass of solute contained in each solution. Record the residue mass in the table below under "solute mass (actual)."

Watch Glass/Residue	Solute Mass (actual)

What is the actual mass of sugar in mixture #1?
What is the actual mass of salt in mixture #2?
What is the actual mass of sugar in mixture #3?
What is the actual mass of salt in mixture #4?

Conclusion: (Hint: Definition of solution; did any of the 1.0 ml solutions actually contain 1.0 ml of solvent?)

Experiment # 5

Name _____

Chemical Formulas and Nomenclature

Reference: Chapters 2 and 3

Objective: Students will gain experience writing chemical formulas for ionic compounds. Students will learn formal procedures used to name ionic and covalent compounds.

Introduction:

Substances are either elements or compounds. A *compound* is a substance that consists of two or more elements bonded together in a specific way. The forces that hold atoms together in a compound are called *chemical bonds.* An *ionic bond* involves the *transfer* of electrons from a *metal* to a *nonmetal*. A *covalent bond* consists of a pair of electrons *shared* between two *nonmetals*.

Ions and the Octet Rule

Atoms are electrically neutral because they have an equal number of electrons and protons. An atom can be converted into a charged particle called an *ion* by losing or gaining one or more electrons. The loss of electron(s) from a neutral atom produces a *positively* charged ion called a *cation* (pronounced cat-ion). The gain of electron(s) by a neutral atom produces a *negatively* charged ion called an *anion* (pronounced an-ion).

Generally, the charge on an ion can be predicted from the position of the element on the periodic table. The metals (on the left-hand side of the table) lose electrons to form cations. The Group IA elements lose ONE electron to achieve an octet and take a charge of 1 positive. This is correctly written using a superscript "+" attached to the upper right side of the elemental symbol, i.e., Na^+. Notice that the number "1" is *not* written when the cation carries a positive *one* charge. The Group IIA elements lose TWO electrons and take a charge of 2 positives. This is correctly written using a superscript "2+" attached to the upper right side of the elemental symbol, i.e., Mg^{2+}. When cations carry a charge greater than one, the number is written first, followed by the sign. The Group IIIA elements lose THREE electrons and take a charge of 3 positives which is written as a superscript "3+", i.e., Al^{3+}.

The nonmetals (on the right-hand side of the table) gain electrons to form anions. The Group VIIA elements gain ONE electron to achieve an octet and take a charge of 1 negative. This is written using a superscript "-" attached to the elemental symbol, i.e., Cl^-. Once again, the "1" is *not* written. The Group VIA elements gain TWO electrons and take a charge of 2 negatives which is written as a superscript "2-", i.e., O^{2-}. The Group VA elements gain THREE electrons and take a charge of 3 negatives which is written as superscript "3-", i.e., N^{3-}. Some transition metals and metals in Group IVA have variable charges (more than one positive ion is possible). See table below.

Some common ions and their location on the periodic table.

IA	IIA											IIIA	IVA	VA	VIA	VIIA	
H^+																	
Li^+	Be^{2+}													N^{3-}	O^{2-}	F^-	
Na^+	Mg^{2+}											Al^{3+}		P^{3-}	S^{2-}	Cl^-	
K^+	Ca^{2+}						Fe^{2+} Fe^{3+}	Co^{2+} Co^{3+}	Ni^{2+}	Cu^+ Cu^{2+}	Zn^{2+}					Br^-	
Rb_+	Sr^{2+}									Ag^+			Sn^{2+} Sn^{4+}			I^-	
Cs^+	Ba^{2+}										Hg_2^{2+} Hg^{2+}		Pb^{2+} Pb^{4+}				

Writing Formulas for Ionic Compounds

Ionic compounds are electrically neutral. Therefore, when writing formulas, the cations (positive) and anions (negative) must combine to produce a net charge of zero. In the formula, the cation (metal) is always written first, followed by the anion (nonmetal). The number and types of each element must be clearly shown in the formula; the type of element is indicated using the elemental symbol, and the number of each element is indicated using a subscript attached at the lower right side of the symbol. The number "1" is not written in cases requiring only a single element. Formulas for ionic compounds are called *formula units*.

The correct ratio required to produce a net charge of zero when Na^+ ions combine with Cl^- ions is one to one because one Na^+ cancels one Cl^-. Therefore, the formula is NaCl. Notice this is not written Na_1Cl_1.

The correct ratio when Na^+ ions combine with O^{2-} ions is two to one because two Na^+ are required to cancel one O^{2-}. The 2 atoms of Na are indicated in the formula using a subscript "2" directly attached to Na. The formula is Na_2O.

The correct combining ratio when Na^+ ions and P^{3-} ions combine is: Na_3P (three to one).

Practice Examples:

Write the formula for the ionic compound that is formed when each of the following pairs of ions interact:

a) K^+ and S^{2-}
b) Mg^{2+} and O^{2-}
c) Ca^{2+} and I^-
d) Li^+ and N^{3-}
e) Al^{3+} and S^{2-}

Solution

(a) The cation has a charge of 1+ because K is a member of Group IA. The anion has a charge of 2- because S is member of Group VIA. Thus, two positive ions (2+) are required for each negative ion (2-) to produce an electrically *neutra*l formula unit.

The formula is $\mathbf{K_2S}$.

(b) The cation has a charge of 2+ and anion has a charge of 2-. The ratio is 1:1. The formula is **MgO**.

(c) The cation has a charge of 2+ and anion has a charge of 1-. Two negative ions are required for each positive ion. The formula is $\mathbf{CaI_2}$.

(d) The cation has a charge of 1+ and anion has a charge of 3-. Three positive ions are required for each negative ion. The formula is $\mathbf{Li_3N}$.

(e) The cation has a charge of 3+ and anion has a charge of 2-. Two positive ions are required for three negative ions. Here, the lowest common factor of 3+ and 2- is 6 (without sign). The formula is $\mathbf{Al_2S_3}$.

Naming Ions

The names of cations and anions are determined by a system developed by the International Union of Pure and Applied Chemistry (IUPAC).

Metals That Form Only One Type of Positive Ion

Elements in Groups IA, IIA, IIIA, and some transition elements form only one type of cation. For these ions, the name of the cation is the elemental name of the metal followed by the word "ion." Cations can now be differentiated from their corresponding neutral forms using specific names. For example, K is potassium (neutral form) and K^+ is potassium ion (cationic form).

Na^+	sodium ion	K^+	potassium ion	Mg^{2+}	magnesium ion
Al^{3+}	aluminum ion	Ag^+	silver ion	Zn^{2+}	zinc ion

Metals That Form Two Different Positive ions

Metals in Group IVA, and most transition metals, form more than one type of cation and the charge must be included in the name. For these ions, the name of the cation is the elemental name of the metal followed by a Roman numeral in parentheses, *with no space* after the name. The Roman numeral indicates the positive charge on the ion. Technically, the names do not end with the word "ion," although some still prefer to include it.

Sn^{2+}	tin(II)	Sn^{4+}	tin(IV)
Pb^{2+}	lead(II)	Pb^{4+}	lead(IV)
Cu^{+}	copper(I)	Cu^{2+}	copper(II)
Fe^{2+}	iron(II)	Fe^{3+}	iron(III)
Co^{2+}	cobalt(II)	Co^{3+}	cobalt(III)
Hg_2^{2+}	mercury(I)	Hg^{2+}	mercury(II)

Naming Anions

Anions are named by replacing the last part of the elemental name with the suffix *–ide*, and adding the word "ion". Anions can now be differentiated from their corresponding neutral forms using specific names. For example, S is sulfur (neutral form) and S^{2-} is sulf*ide* (anionic form).

F^-	fluoride ion	Cl^-	chloride ion	Br^-	bromide ion	I^-	iodide ion
O^{2-}	oxide ion	S^{2-}	sulfide ion	N^{3-}	nitride ion	P^{3-}	phosphide ion

Polyatomic Ions

A *polyatomic ion* is an ion that contains two or more elements. It is recommended that you memorize the names and formulas of the following polyatomic ions:

NH_4^+	ammonium	SO_3^{2-}	sulfite
CN^-	cyanide	SO_4^{2-}	sulfate
OH^-	hydroxide	HSO_3^-	hydrogen sulfite
$C_2H_3O_2^-$	acetate	HSO_4^-	hydrogen sulfate
CrO_4^{2-}	chromate	PO_3^{3-}	phosphite
$Cr_2O_7^{2-}$	dichromate	PO_4^{3-}	phosphate
MnO_4^-	permanganate	HPO_4^{2-}	hydrogen phosphate
NO_2^-	nitrite	ClO^-	hypochlorite
NO_3^-	nitrate	ClO_2^-	chlorite
CO_3^{2-}	carbonate	ClO_3^-	chlorate
HCO_3^-	hydrogen carbonate	ClO_4^-	perchlorate

The common name for HCO_3^-, HSO_3^-, and HSO_4^- are bicarbonate, bisulfite, and bisulfate respectively.

Naming Ionic Compounds

Binary ionic compounds containing metals that form only one type of positive ion

These compounds contain only *two types* of elements; a metal ion and a nonmetal ion. Note: binary refers to element types, not total number of atoms. For example; $MgBr_2$ contains 3 total atoms, 1 Mg and 2 Br's, but it contains only two types, Mg and Br. Also, recall all elemental symbols begin with capital letters, if you have a binary compound, your formula contains only two capital letters. The metal is always named first using the elemental name of the metal. The nonmetal is named second using the anionic name of the nonmetal (elemental name modified with the suffix *–ide*).

Practice Examples:

Name the following binary ionic compounds:

NaCl	$MgBr_2$	AlP	K_2S	SrF_2	ZnI_2

Solution

NaCl	sodium chloride	K_2S	potassium sulfide
$MgBr_2$	magnesium bromide	SrF_2	strontium fluoride
AlP	aluminum phosphide	ZnI_2	zinc iodide

Binary ionic compounds containing metals that form two different positive ions

These compounds are essentially named using the same procedure developed for metals forming only one type of cation. The distinction is that the charge on the cation must be written as a Roman numeral in parentheses immediately after (with no space) the metal name.

Practice Examples:
Name the following binary ionic compounds:

$FeBr_3$	CoF_2	SnO	PbI_4	HgS	Cu_3P

Solution

$FeBr_3$	iron(III) bromide	PbI_4	lead(IV) iodide
CoF_2	cobalt(II) flouride	HgS	mercury(II) sulfide
SnO	tin(II) oxide	Cu_3P	copper(I) phosphide

Ionic compounds containing polyatomic ions
Identifying compounds containing polyatomic ions is somewhat simplified by the fact that all elemental symbols begin with capital letters. If you identify more than two capital letters in the formula, your compound contains a polyatomic ion that you must immediately recognize (the value of memorizing!). Naming these compounds is simply based on your familiarity with the polyatomic ions. The cation is named first using its elemental name, followed by the name of the polyatomic ion.

Practice Examples:
Name the following polyatomic ionic compounds:

$Ca(NO_3)_2$	$ZnSO_4$	NH_4CN
Li_3PO_4	Na_2CO_3	$Mg(HCO_3)_2$

Solution

$Ca(NO_3)_2$	calcium nitrate
$ZnSO_4$	zinc sulfate
NH_4CN	ammonium cyanide
Li_3PO_4	lithium phosphate
Na_2CO_3	sodium carbonate
$Mg(HCO_3)_2$	magnesium hydrogen carbonate

Compounds containing only nonmetals (molecular compounds)
This type of compound contains covalent bonds, so the concept of cations and anions is somewhat obscure and does not necessarily apply. Regardless, they are essentially named using the previously developed procedure for naming binary compounds containing a metal. The first element is named using its elemental name and the second is named using its anionic name. The difference is the use of Greek prefixes attached to each name, which indicate the number of each element present in the formula. There is one important exception; the prefix "mono" is never attached to the name of the first element in the formula. Let us look at SF_6 as an example. You should immediately notice that S and F are nonmetals because both are located in the nonmetal section of the periodic table (right side). This observation should immediately trigger "prefixes" in your mind. SF_6 is sulfur hexafluoride because the formula indicates one sulfur (note the absence of the subscript "1" attached to S) and 6 fluorides (subscript 6 attached to F). This is not monosulfur hexafluoride because "mono" is never used with the first element. It is possible, however, to attach all other prefixes to the name of the first element, i.e., N_2O_5 is dinitrogen pentoxide. The Greek prefixes are listed below, note that all end in a vowel. When the prefix ends with an "a" or "o", and the elemental name begins with an "a" or "o", the vowel of the prefix is usually dropped to simplify pronunciation. Notice in our N_2O_5 example above, the name of the anion is pentoxide, not pentaoxide.

Greek prefixes

1 (mono-)	2 (di-)	3 (tri-)	4 (tetra-)	5 (penta-)
6 (hexa-)	7 (hepta-)	8 (octa-)	9 (nona-)	10 (deca-)

Practice Examples:
Name the following binary molecular compounds:

N_2O_5	CO_2	P_4S_3	XeF_6	ICl	NH_3	I_4O_9	CO	H_2O	H_2O_2

Solution

N_2O_5	dinitrogen pentoxide	NH_3	nitrogen trihydride (ammonia)
CO_2	carbon dioxide	I_4O_9	tetraiodine nonoxide
P_4S_3	tetraphosphorous trisulfide	CO	carbon monoxide
XeF_6	xenon hexafluoride	H_2O	dihydrogen monoxide (water)
ICl	iodine monochloride	H_2O_2	dihydrogen dioxide

Exercises

Inspect the periodic table and list ALL elements with a ONE letter abbreviation.

Symbol	Name	Period number	Group number	metal, nonmetal, or metalloid

Formulas of Ionic Compounds

Complete the following table with the formula of the compound.

	Br^-	O^{2-}	NO_3^-	PO_4^{3-}
Na^+				
Mg^{2+}				
Al^{3+}				
Pb^{4+}				
NH_4^+				
Fe^{2+}				
Fe^{3+}				

Ionic Compounds

Write the correct name for each of the following ionic compounds.

Formula	Name
$Pb(HCO_3)_4$	
Al_2S_3	
$LiHSO_4$	
$Zn_3(PO_4)_2$	
CoF_3	
$Ca(CN)_2$	
SnO_2	
Na_2CrO_4	
K_2CO_3	
Cu_3P	
$Sr(OH)_2$	
$(NH_4)_2HPO_4$	
Hg_2Cl_2	

Formula	Name
$BaSO_4$	
$Sn(NO_3)_2$	
$AgClO_3$	
$Cu(HSO_3)_2$	

Molecular Compounds

Name each of the following compounds.

Formula	Name
BBr_3	
Br_3O_8	
CI_4	
C_3O_2	
Cl_2O_7	
IF_5	
I_2O_5	
NCl_3	
N_2O_5	
OF_2	
P_4S_3	
P_4S_9	
P_4O_{10}	
SF_6	
S_2Cl_2	
SiS_2	
$SiBr_4$	
XeF_2	
XeO_4	
XeF_6	

Experiment # 6

Name _____

Solubility

Reference: Chapter 3

Objective: Students will test the solvent properties of various liquids to observe and understand the chemical nature of solubility and miscibility.

Materials: acetone, chloroform, ammonia, methanol, water, test tubes, and pipettes.

Introduction:

Water is a common solvent in many solutions and substances like salt and sugar readily dissolve in water. Any substance that dissolves appreciatively in a specified solvent is said to be *soluble* in that solvent. Technically, the term *solubility* refers to a quantitative maximum amount of substance that can dissolve in a given volume of solvent at a specific temperature. The ability of a substance to dissolve in a particular solvent depends on the identity of both the solvent and the substance; the general rule is "likes dissolve likes." Water is a polar covalent molecule and, as a solvent, can dissolve similar molecules (polar covalent). The polarity of water is responsible for its remarkable solvent properties and explains why ionic compounds (i.e., NaCl) and polar covalent compounds (i.e., sucrose, ammonia) are soluble, whereas nonpolar molecules (i.e., organic, gasoline, oils) are not. Terms such as soluble, slightly soluble, insoluble, and solubility are used to describe the ability of a substance to dissolve in a solvent. Intuitively, we associate the term "dissolving" with solids and liquids; however, a liquid may also be soluble in another liquid. For example, when 25.0 ml of ether is added to 25.0 ml of water, the resulting total volume is <u>not</u> 50.0 ml, in fact, it is slightly less (approx. 48.5 ml). This is the result of solubility; ether and water are slightly soluble in one another and consequently, the volumes are not additive. The solubility of one liquid in another is difficult to determine and is usually not readily observed upon mixing. For this reason, liquids are often characterized using *their ability to mix with other liquids* rather than their *solubility* in other liquids. The degree of mixing between two liquids is described using the terms *miscible* and *immiscible*. Two liquids are miscible (soluble) if a uniform solution results after mixing (i.e., water and ammonia). Two liquids are immiscible (insoluble) if two distinct layers form after mixing (i.e., oil and water). Water is miscible in polar liquids and immiscible in nonpolar (organic) liquids.

Part A:

Add 1.0 ml of each of the following reagents to four separate, clean, and dried test tubes: chloroform, ammonia, methanol, water. Add 1.0 ml of acetone to each test tube and mix. Observe the results and characterize the liquids as miscible (**M**) or immiscible (**I**). Record your results in the table below under the corresponding reagents. Clean and dry three test tubes and add 1.0 ml of each of the following reagents to separate test tubes: ammonia, methanol, water. Add 1.0 ml of chloroform to each test tube and mix. Record your results in the table below under the corresponding reagents. Clean and dry two test tubes and add 1.0 ml of each of the following reagents to separate test tubes: methanol, water. Add 1.0 ml of ammonia to each test tube and mix. Record your results in the table below under the corresponding reagents. Clean and dry one test tube and add 1.0 ml of methanol and 1.0 ml of water to the test tube and mix. Record your results in the table below under the corresponding reagents.

Reagents	Chloroform	Ammonia	Methanol	Water
Acetone				
Chloroform	----------			
Ammonia	----------	----------		
Methanol	----------	----------	----------	

Identify the organic liquids:

Identify the inorganic liquids:

What liquids are polar?

What liquids appear to be non-polar?

List the liquid-pairs that are miscible:

List the liquid-pairs that are immiscible:

Are immiscible liquids soluble in one another? Briefly explain.

Are miscible liquids soluble in one another? Briefly explain.

Experiment # 7

Name _____

Molecular Geometry

Reference: Chapter 3

Objective: To illustrate the geometric structures of simple molecules and to demonstrate the relationship between bonding and molecular geometry.

Materials: Molecular model kits.

Introduction:

In recent years, advancements in research and technology have provided precise information on molecular geometry, i.e., bond distances, angles, and energies. Structural theory has advanced far beyond the simple electron dot representation and now rests securely on the foundations of quantum and wave mechanics. Although problems involving only simple molecules can be solved through rigorous mathematical calculations, approximations such as Valence Bond Theory (VBT) and Molecular Orbital Theory (MOT) are very successful in providing results that agree favorably with experimental measurements. This exercise will utilize Valence Bond Theory to illustrate the geometry of a variety of simple molecules. This will be accomplished through the use of model kits that show the correct angles formed between atoms in the molecule. The first covalent bond formed between any two atoms is always a sigma-bond (σ-bond). This type of bond has electrons distributed symmetrically about the bond axis and is used to define the bond axis. Additional bonds (double or triple) formed between the same two atoms will be pi-bonds (π-bonds). These bonds are perpendicular to the defined σ-bond and do not influence geometry. It is the σ-bonds, and any lone pairs of electrons occupying sigma hybrid orbitals, that determine molecular geometry.

Model kits are a necessary and integral part of the study of molecular geometry. They are tools that allow students to transcend the inherent difficulties that arise from visualizing a three dimensional structure on a two dimensional piece of paper. A complete understanding of the capabilities and limitations of model kits is essential in their successful use as a visual aid. Open your model kits and carefully read the instructions. Inventory the various pieces contained in your kit and pay special attention to the color codes used to designate specific atoms. It is important that you take the time now to familiarize yourself with all the components contained in your kit. This will allow you to concentrate on the structure or concept currently under study and prevent wasted time (and confusion) that may result from constantly referring back to the instructions. A table is included at the end of the lab for reference.

Methane (CH_4)

Construct a model of methane using your model kit. Locate a tetrahedral center (a carbon atom) and attach four rods (bonds) in each hole of the atom. Attach 4 balls (hydrogen atoms) of the same color to each rod extending from your structure.

Sketch the model using the solid/dashed wedge convention and name the geometry.

Write the structural formula.

Write the condensed structural formula.

What are the H–C–H bond angles?_____
How many σ-bonds are on the central carbon?_____
Identify the two planes present in this molecule. Do the planes divide the molecule into equal halves?_____
How many atoms, including carbon, are in the same plane?_____

Ammonia (NH_3)

Construct a model of ammonia using your model kit.

Sketch the model, including the lone pair of electrons, and name the geometry.

Write the structural formula.

Write the condensed structural formula.

What is the H–N–H bond angle?_____
How many σ-bonds are on the central nitrogen?_____
How many lone pairs are on the central nitrogen?_____
Are any atoms in ammonia in the same plane?_____
Suggest a reason why the H–N–H bond angles in ammonia differ from the H–C–H bond angles observed in methane.

Water (H_2O)

Construct a model of water using your model kit.

Sketch the model, including lone pairs of electrons, and name the geometry.

Write the structural formula.

Write the condensed structural formula.

What is the H–O–H bond angle? _____
How many σ-bonds are on the central oxygen? _____
How many lone pairs are on the central oxygen? _____
How many atoms, including oxygen, are in the same plane? _____
Why is the H–O–H bond angle slightly smaller than the bond angles observed in ammonia and methane?

What conclusions can be made on how lone pairs affect molecular geometry?

Sulfur Hexafluoride (SF_6)

Construct a model of sulfur hexafluoride using your model kit.

Sketch the model and name the geometry.

Write the structural formula.

Write the condensed structural formula.

What are the F–S–F bond angles? _____
How many σ-bonds are on the central sulfur? _____
How many lone pairs are on the central sulfur? _____
How many atoms, including sulfur, are in the same plane? _____

Carbon Dioxide (CO_2)

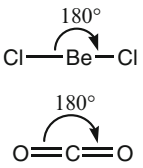
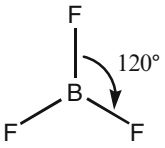
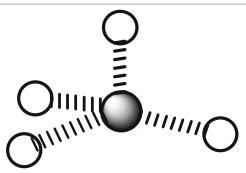
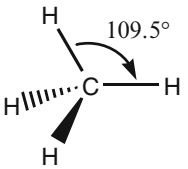
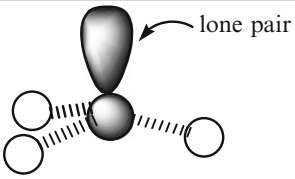
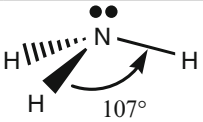
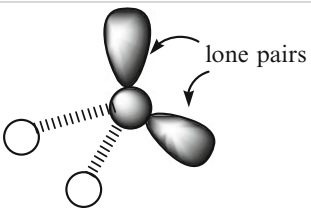
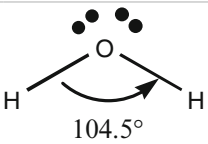
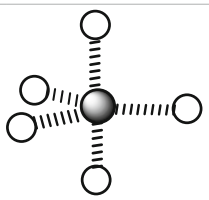
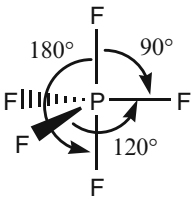
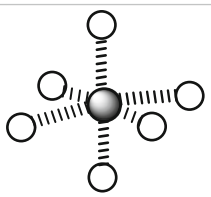
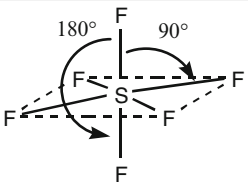
Construct a model of carbon dioxide using your model kit.

Sketch the model and name the geometry.

Write the structural formula.

Write the condensed structural formula.

What is the O–C–O bond angle? _____
How many σ-bonds are on the central carbon? _____
How many π-bonds are on the central carbon? _____
How many lone pairs are on the central carbon? _____
How many atoms are in the same plane? _____

Lone pairs	Bonded atoms[a]	Geometry	Molecular Shape	Examples
0	2	Linear		
0	3	Trigonal Planar		
0	4	Tetrahedral		
1	3	Trigonal Pyramidal		
2	2	Angular (bent)		
0	5	Trigonal Bipyramidal		
0	6	Octahedral		

aTechnically this should read # of σ-bonds. A single bond contains 1 σ-bond and 0 π-bonds, double bonds contain 1 σ- and 1 π-bond, triple bonds contain 1 σ- and 2 π -bonds. Therefore, when counting bonded atoms to determine molecular geometry, count single bonds as 1, double bonds as 1, and triple bonds as 1. See CO_2 example above.

Experiment # 8

Name _____

Organic Chemistry and Functional Groups

Reference: Chapter 4

Objective: Students will gain experience in the basic recognition of functional groups present in a variety of organic molecules.

Materials: Molecular model kits.

Introduction:

Organic chemistry is the study of the properties, structure, and function of compounds containing carbon. Although the defining element in organic molecules is carbon, it is common practice to define them by the obligate presence of both carbon and hydrogen. This does not mean that organic molecules contain only carbon and hydrogen; elements such as nitrogen, oxygen, sulfur, phosphorous, and chlorine may also be present. The study of organic chemistry does not involve the individual study of the vast number of organic compounds. Instead, organic compounds are divided into broad classes based on the presence of a *functional group*. Functional groups are atoms, groups of atoms, or common structural features used to classify organic molecules. In general, functional groups will react in a unique, predictive manner and this chemical behavior is similar in all compounds containing a specific group. It is possible, and quite common indeed, to have more than one functional group on a single molecule. In these cases, the molecule will exhibit chemical and physical properties of all groups present. The study of functional groups is the most effective and efficient approach to the study of organic chemistry. A wide range of functional groups can be found on various types of controlled substances.

Part A:

Use model kits to construct the following molecules using their chemical formulas. Rotate each structure in space and observe the geometry from different perspectives. Compare the structures of different molecules and determine some factors that influence geometry, i.e., number and types of bonds present, bond orientation on central atom, number of atoms in the molecule, etc. Draw the structures of each molecule.

Alkanes:

Name	Formula	Structure
Methane	CH_4	
Ethane	C_2H_6	
Propane	C_3H_8	
Butane	C_4H_{10}	

Alkenes:

Name	Formula	Structure
Ethene	C_2H_4	
Propene	C_3H_6	
1-Butene	C_4H_8	
2-Butene	C_4H_8	

1-Butene and 2-Butene are positional isomers because they have the same molecular formula and differ only in the location of the double bond.

Alkynes:		
Name	Formula	Structure
Acetylene	C_2H_2	
(Ethyne)		
Propyne	C_3H_4	
1-Butyne	C_4H_6	
2-Butyne	C_4H_6	

Note: acetylene does not contain a double bond despite its "ene" ending.

Alcohols:		
Name	Formula	Structure
Ethanol	C_2H_5OH	
Propanol	C_3H_7OH	
Isopropanol	C_3H_7OH	
(Isopropyl alcohol)		
1-Butanol	C_4H_9OH	
2-Butanol	C_4H_9OH	

Aldehydes:		
Name	Formula	Structure
Formaldehyde	HCHO	
(Methanal)		
Acetaldehyde	CH_3CHO	
(Ethanal)		
Propanal	CH_3CH_2CHO	
Butanal	$CH_3CH_2CH_2CHO$	

Note: The location of the aldehyde functional group will always be carbon # 1.

Carboxylic Acids:		
Name	Formula	Structure
Formic Acid	HCOOH	
Acetic Acid	CH_3COOH	
(Ethanoic Acid)		
Propanoic Acid	CH_3CH_2COOH	
Butanoic Acid	$CH_3CH_2CH_2COOH$	

Others:		
Name	Formula	Structure
Chloroform	$CHCl_3$	
Benzene	C_6H_6	
Cyclohexane	C_6H_{12}	

Draw structures for the following:
1-bromo-2,2-dichloropentane

2-methyl-3-heptene

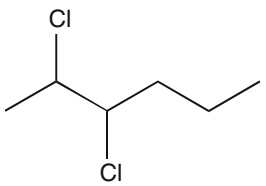
1-chloro-1-pentanol

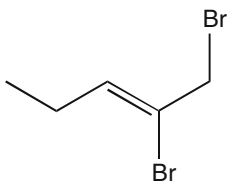
3-isopropyloctane

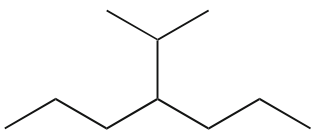
3-methylbutanal

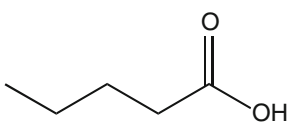
4-chloropentanoic acid

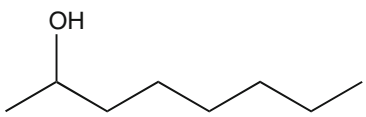
Name the following:

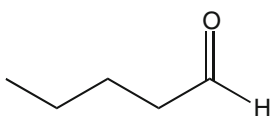






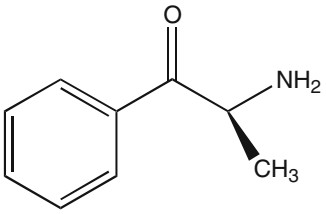




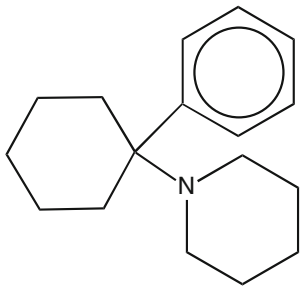


Part B:

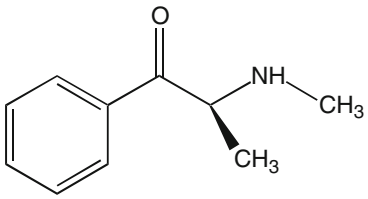
Circle and name all functional groups present in each of the following compounds. A table of functional groups is provided on the last page of the lab.



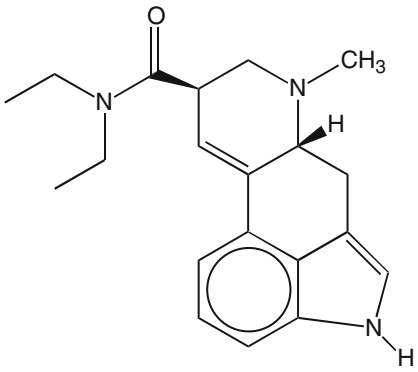
Cathinone



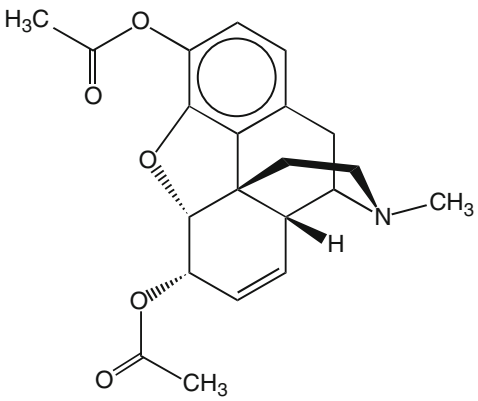
PCP



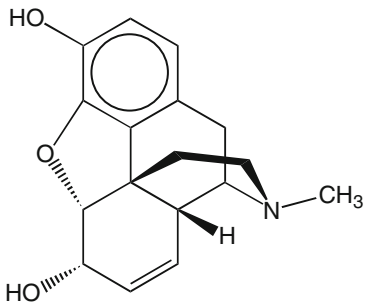
Methcathinone



LSD



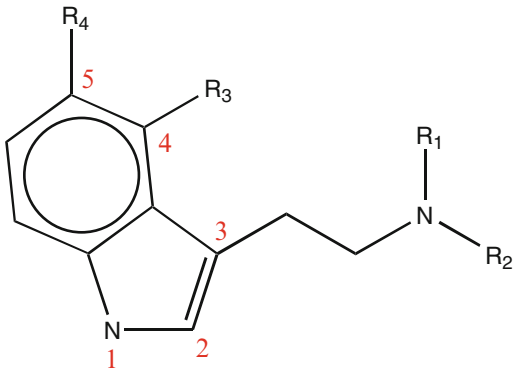
Heroine



Morphine

Part C:

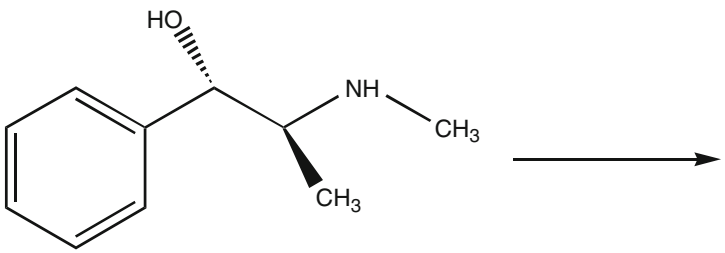
The structure below is tryptamine when all R-groups are hydrogen (H). Substitute the groups indicated in the table below for each corresponding "R" and name the resulting compounds (reference Chap. 15).



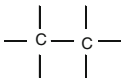
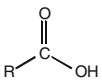
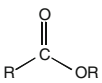
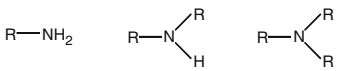
R_1	R_2	R_3	R_4	Name
H	H	H	H	Tryptamine
CH_3	CH_3	H	H	
CH_3	CH_3	H	OH	
CH_3	CH_3	OH	H	
CH_3	CH_3	PO_4^{3-}	H	
C_2H_5	C_2H_5	H	H	
CH_3	CH_3	H	OCH_3	

Part D:

Use your model kit to construct pseudoephedrine as shown below. Replace the "OH" functional group with "H" and draw the resulting structure. Name the new compound (reference Chap. 13).



Pseudoephedrine

Class	Functional Group	IUPAC ending
Alkane		"-ane"
Alkene		"-ene"
Alkyne		"-yne"
Alcohol		"-ol"
Ketone		"-one"
Aldehyde		"-al"
Carboxylic Acid		"-oic acid"
Ester		
Nitro Compounds		
Amines	 1° 2° 3°	"-amine"

Experiment # 9

Name _____

Microcrystallography

Reference: Chapter 8

Objective: Students will gain experience in the forensic identification of elements using microcrystalline technique.

Materials: Solid form and 5% solutions of the following: NH_4Cl, NaCl, LiCl, ammonium molybdate (AMM-hydrate or anhydrous), and NaOH.
5% solutions of the following: $Mg(C_2H_3O_2)_2$, $Mg(NO_3)_2$, $Na_2HPO_4$and $KMnO_4$(or alternatively, $NaMnO_4$).
Acids: 5M HCl and 5M $HClO_4$ (perchloric acid).
Others: KCl (solid), $CaCO_3$ (sat. solution), and MgO (sat. solution), Microscope, microscope slides, droppers.

Solutions: Prepare 5% (m/v) solutions by dissolving 5 g of solid in 100 ml total volume. Alternatively, methanol may be used as solvent, however water forms slightly more stable crystals. $KMnO_4$ should be prepared fresh. If $NaMnO_4$ is used instead of $KMnO_4$, you will need to add a few drops of H_2SO_4 (5% v/v) to solution to increase solubility.

Part A:

Technique 1: Mount a slide on the microscope, place a few crystals of NaCl on the slide using a small spatula, and focus the solid in the microscope. Add a drop of Na_2HPO_4 solution directly on the solid and observe crystal formation using the microscope without mixing. Record your results below. Repeat with other solids and solutions listed below.

Solid	Solution	Crystals (Yes/No)	Sketch Crystals (if yes)
NaCl	Na_2HPO_4		
NaOH	$Mg(NO_3)_2$		
KCl	$HClO_4$		
AMM	HCl		
LiCl	$KMnO_4$		

Conclusion:

Part B:

Technique 2: Mount a slide on the microscope, place a drop of Na_2HPO_4 solution on the slide, and focus the drop in the microscope. Add a few crystals of NH_4Cl to the drop and, without mixing, observe crystal formation using the microscope. Record your results below. Repeat with other solutions and solids listed below.

Solids	Solutions	Crystals (Yes/No)	Sketch Crystals (if yes)
NH_4Cl	Na_2HPO_4		
NaOH	$Mg(NO_3)_2$		
KCl	$HClO_4$		
AMM	HCl		
LiCl	$KMnO_4$		

Conclusion:

Part C: Micro Test Technique

Technique 3: Mount a slide on the microscope, place a drop of solution-I on the slide and focus the drop in the microscope. Add a drop of solution-II to first drop and, without mixing, observe crystal formation using the microscope. Record your results below. Repeat with other solutions listed below.

Solutions-I	Solutions-II	Crystals (Yes/No)	Sketch Crystals (if yes)
NH_4Cl	Na_2HPO_4		
$CaCO_3$	$Mg(C_2H_3O_2)_2$		
MgO	$HClO_4$		
$Mg(NO_3)_2$	HCl		
MgO	$KMnO_4$		

Conclusion:

Part D: Macro Test Technique

Technique 4: Mount a slide on the microscope, place a drop of solution-I on the slide, and focus the drop in the microscope. Add a drop of solution-II to first drop and, without mixing, observe crystal formation using the microscope. Record your results below. Repeat with other solutions listed below.

Solution I	Solution II	Crystals (Yes/No)	Sketch Crystals (if yes)
NaOH	$HClO_4$		
NaCl	$HClO_4$		
AMM	Na_2HPO_4		
KMnO4	Na_2HPO_4		
LiCl	Na_2HPO_4		

Conclusion:

Experiment # 10

Name _____

Chemical Extraction

Reference: Chapter 9
Objective: Students will gain practical experience using forensic extraction techniques to isolate solids from mixtures.
Materials: $NaHCO_3$ (sodium bicarbonate, saturated solution), 0.1 M HCl, starch, aspirin, acetaminophen, H_2SO_4 (concentrated), formaldehyde, 1% iodine solution acidified with glacial acetic acid, chloroform, hexane, vortex, spot plates, 5 ml test-tubes, droppers, and centrifuge.

Prepare a sample mixture containing 100 parts starch:1 part aspirin: 1 part acetaminophen, provide each group with a 1 g sample of the mixture.

Part I: Extraction:

1) Place about 0.25 g of sample in a 5 ml test tube labeled "A."
2) Add 1.0 ml (approx. 20 drops) of 0.1 M HCl.
3) Vortex the mixture and centrifuge.
4) Remove the top liquid using a dropper and place in a clean 5 ml test tube labeled "B." Be careful not to disturb the solid pellet at the bottom of test tube "A".
5) Save test tube "A" for Part-II.
6) Add a few drops of $NaHCO_3$ (sat.) to test tube "B" and vortex the mixture.
7) Add 1.0 ml of hexane and vortex the mixture.
8) Carefully transfer the organic layer using a dropper to a clean test tube labeled "C." Save test tube "C" for Part-II. The organic layer is hexane and should be the top layer, but check using solubility in DI water.
9) Add 1.0 ml chloroform to test tube "B" and vortex the mixture.
10) Carefully transfer the organic layer using a dropper to a clean test tube labeled "D." Save test tube "D" for Part-II. The organic layer is chloroform ($CHCl_3$) and should be the bottom layer, but check using solubility in DI water.

Part II: Screening Tests:

Perform the screening tests below on the samples contained in test tubes A (solid pellet), B (aqueous layer), C (organic layer, hexane), and D (organic layer, chloroform). The best results are obtained when the tests are run side-by-side as opposed to in sequence (reference Chap. 7). Starch test is positive when blue color appears with iodine. Concentrated sulfuric acid and formaldehyde produce red color with aspirin.

Test 1: Reference

Place a small sample (1/2 pea size) of the original mixture of starch, aspirin, and acetaminophen mixture (100:1:1) into each of two clean, separate wells of a spot plate. These wells will be your reference, so do not empty or clean after the tests are performed.

Starch Test:

Add one drop of 1% iodine solution to the first well containing the sample and record the color change: _____. Do not empty or clean the well.

Aspirin Test:

Add one drop of formaldehyde and three drops of concentrated sulfuric acid to the second well containing the sample and record the color change: _____. Do not empty or clean the well.

Test 2:
Place a small amount of solid from test tube "A" into each of two clean, separate wells of the spot plate. Repeat the tests above and record your results. Compare the intensity of the colors produced to the colors in the reference.

Result of starch test:_____

Result of aspirin test:_____

Test 3:
Place 3 three drops of solution in test tube "B" into each of two clean, separate wells of the spot plate. Evaporate the solvent and repeat the tests above. Record your results below and compare the relative intensity of the colors produced to the colors in the reference.

Result of starch test:_____

Result of aspirin test:_____

Test 4:
Place 3 three drops of solution in test tube "C" into each of two clean, separate wells of the spot plate. Evaporate the solvent and repeat the tests above. Record your results below and compare the relative intensity of the colors produced to the colors in the reference.

Result of starch test:_____

Result of aspirin test:_____

Test 5:
Place 3 three drops of solution in test tube "D" into each of two clean, separate wells of the spot plate. Evaporate the solvent and repeat the tests above. Record your results below and compare the relative intensity of the colors produced to the colors in the reference.

Result of starch test::_____

Result of aspirin test:_____

Summarize your conclusions based on the test results:

Experiment # 11

Name _____

Chromatography

Reference: Chapter 10

Objective: Students gain practical experience using paper chromatography to separate the components of a variety of mixtures.

Materials: Red, blue, green, brown, and black felt tip pens, chromatographic paper, 1-butanol, 1-propanol, saran wrap.

Introduction:

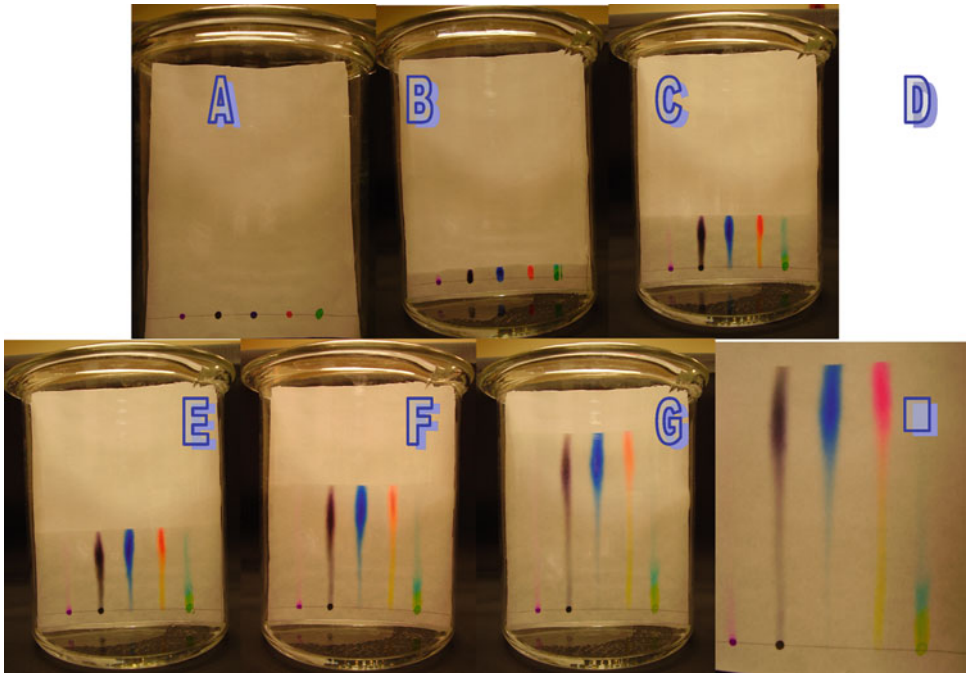
Chromatography is a technique that utilizes a stationary phase and a mobile phase to separate the components of a mixture. The separation process is based on the fact that a given component (molecule) will exhibit a higher affinity for one of the phases and will either move in the mobile phase or remain in place on the stationary phase. Affinity is influenced by several factors, but size and polarity often play key roles. In this experiment, paper chromatography will be used to separate the components of ink commonly found in felt-tipped pens. The mixture (ink) will be spotted on the chromatographic paper (stationary phase) and placed in a developing chamber containing the mobile phase (solvent mixture). The mobile phase will migrate up the paper and contact each of the ink mixtures. The components in the ink that are soluble in the mobile phase will move up the stationary phase (paper) at different rates depending on size and polarity. The components that are not soluble will remain in place on the stationary phase. There are many types of chromatographic techniques and several factors must be considered when choosing a particular method. Thin-layer chromatography (TLC), for example, is simple, quick, and relatively inexpensive; however, it provides only qualitative results. High-performance liquid chromatography (HPLC) is more complex and requires sophisticated instrumentation, but it provides extremely accurate quantitative results. Other forms of chromatography include column chromatography, liquid chromatography (LC), and gas chromatography (GC).

Procedure:

1. Place 15.0 mL of 1-propanol, 15.0 mL of 1-butanol, and 15.0 mL of deionized water in a clean, dry 600 ml beaker. Cover the top of the beaker tightly with saran wrap. A rubber band may be used to secure the plastic wrapping. Lightly swirl the beaker to mix the contents. This will be used as the developing chamber.
2. Obtain a 10 cm × 15 cm piece of chromatographic paper. *Handle the chromatographic paper using the edges at all times*. Touching the paper with bare hands may deposit oils and dirt that will affect your results.
3. Using a ruler and a lead pencil, draw a straight line across the entire 10 cm width, 1.5 cm up from the bottom edge of the paper. *Do not use a pen to draw this line*.
4. Mark five small dots on the line with a pencil at 1.5 cm intervals.
5. Spot each dot with a different colored pen and allow the spots to dry.

6. Remove the saran wrap cover on the developing chamber and carefully place the chromatographic paper in the beaker with the edge nearest the samples placed downward in the solvent (handle the paper using the edges only). Important: Be sure that the samples are NOT immersed in the solvent. Replace the saran wrap cover.

7. The developing solvent will begin to migrate up the paper. Allow the system to stand undisturbed until the solvent has run close to the top of the paper, or for a period of 60 min. Do not allow the solvent to reach the top of the paper.



8. Remove the paper from the developing chamber and draw a line across the solvent front using a pencil. Allow the paper to dry.

9. Answer the following questions:

(a) What colors have properties similar to the solvent?

(b) What colors have properties that are different from the solvent?

(c) What pens contain components that are similar?

(d) How many components are contained in the red ink?

(e) How many components are contained in the blue ink?

(f) How many components are contained in the black ink?

(g) What components appear to be common in the pens tested?

(h) Do you believe your results would be different if ink pens from another manufacturer were used? Explain.

Experiment # 12

Name _____

GCMS Interpretation

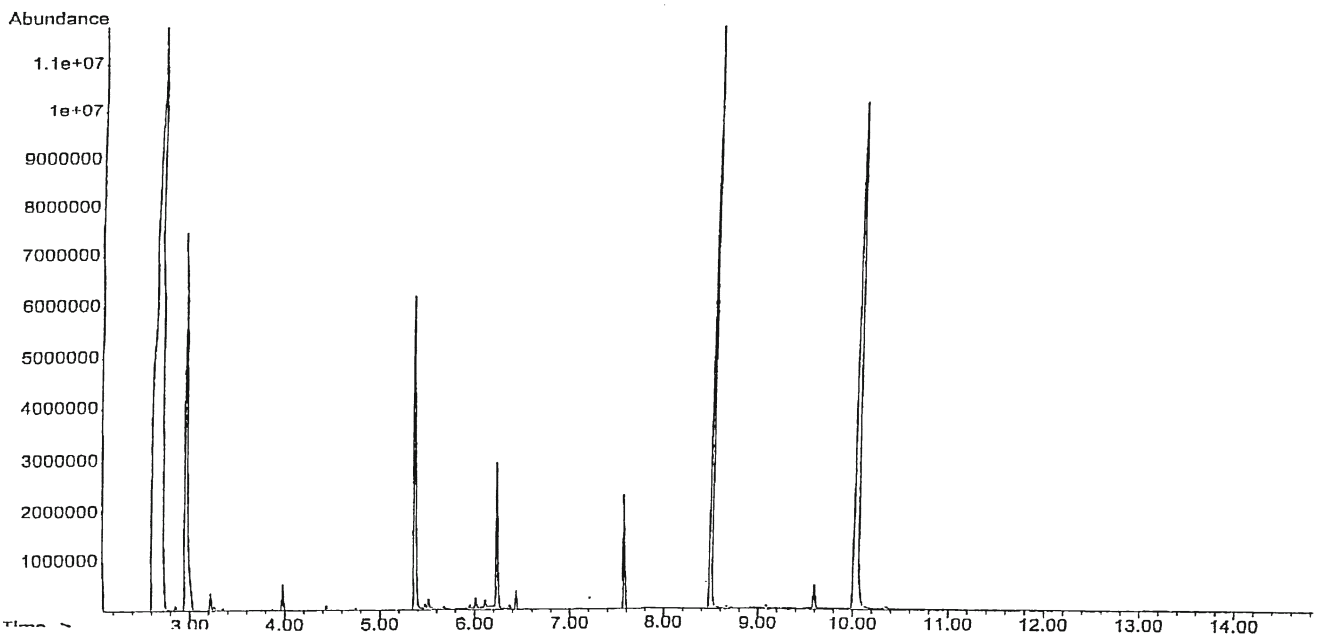
Reference: Chapter 10
Objective: Students will gain experience in the interpretation of data provided by GCMS analysis.

Introduction:

Gas chromatography (GC) can be combined with mass spectrometry (MS) to provide scientists with an extremely valuable technique used to identify the components present in a gaseous mixture (GCMS). A sample of the mixture under analysis is injected into the gas chromatograph where it is immediately vaporized in the injection port. A carrier gas (mobile phase, usually N_2 or He) passes the vaporized sample through a column contained in the GC (stationary phase). The individual components in the gaseous mixture interact with the column to accomplish separation. In gas chromatography, the separation process is usually size dependent; smaller molecules interact less with the column and arrive at the detector faster than larger ones. The *time required for a particular component to travel from the injection port to the detector* is called the *retention time*. Retention times are basically the time each component spends in the column and are specific to that component. The detector records each component eluting off the column as a peak on a *chromatogram* at specific retention times. Although a GC does not routinely provide quantitative data, it does give important information about the components in a gaseous mixture. Since each component is recorded as a peak on the chromatogram, the number of components present in the mixture is easily determined by simply counting the number of observable peaks. Most modern GCs are automated and contain software that calculates the area under each peak. These calculations are used to determine the relative amounts of each component. For example, if the area under one peak is twice the area under another, that component is present in twice the quantity. The exact amounts are not known, but the relative concentrations are. Retention times can be used to determine the relative size of each component present. Peaks observed at low retention times represent smaller molecules, while those with greater times represent larger ones. Once again, the actual size of each component is not known, but the relative sizes are easily determined. The identity of each component is determined through mass spectrometry. The separated components from GC are passed into a mass spectrometer coupled directly to the GC. In the mass spectrometer, each component is bombarded with a beam of high-energy electrons that fragment the molecule into positively charged ions. The fragmented ions are recorded on a *mass spectrum* by mass (specifically charge/mass). The *base peak* is the most intense (highest) and represents the *base ion*; usually the most stable ion formed (the one formed the easiest). The base peak is assigned 100% and the intensities of the other peaks are recorded as percentages of the base peak. Tables identifying ions of specific mass are used to reassemble the molecule from the ion fragments. The base peak and the molecular ion peak often play key roles in the identification process. The *molecular ion* is formed when a single electron is ejected from the molecule by the imposed high-energy beam. This peak commonly represents the mass of the molecule under investigation. In some cases, the molecular ion may be the base peak; however, this is not a requirement and should not be considered a standard.

Part A:

Interpret the gas chromatogram below and answer the following questions.



1. Identify each component in the mixture using retention times. How many different components are present? (Hint: peaks below Abundance 1000000 are not considered components).

2. Identify the peak representing the component of smallest mass.

3. Identify the peak representing the component of greatest mass.

4. Predict the peak representing the molecule present in greatest abundance.

5. Predict the peak representing the molecule present in least abundance.

Part B:

Identify each of the following using the MS data provided. You will need to reference additional information from your text.

Name	Molecular Ion (m/z)	Base Ion (m/z)	Other Prominent Ions
1)	135	044	91, 92, 65, 120, 134, 77
2)	303	082	182, 83, 77, 94, 105
3)	369	327	268, 204, 310, 315
4)	237	180	182, 209, 152, 138
5)	243	200	91, 242, 243, 186

Part C:
List the mass (m/z) of the major ion fragments formed from each the following. Draw the structure of each fragment.

1) Methamphetamine:

2) MDMA:

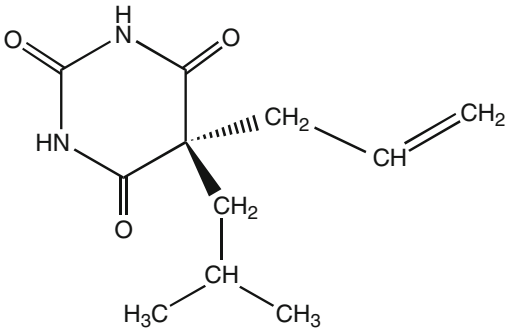
3) Psilocin/psilocybin:

4) THC:

5) Pseudoephedrine:

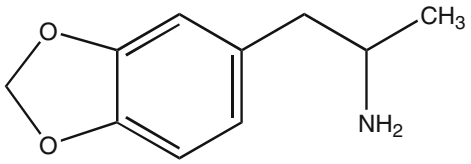
Part D:

Draw the ion fragments represented in the MS data provided for each of the following molecules.



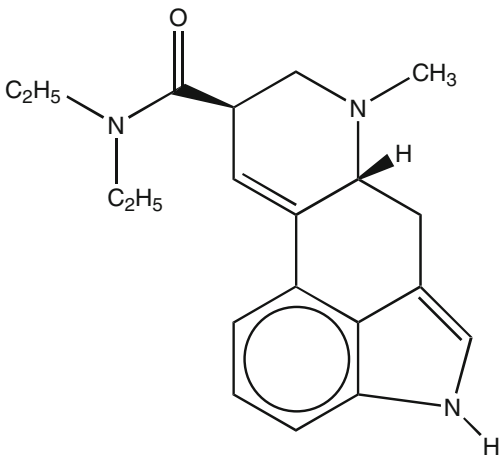
Butalbital

Molecular weight = 244
Base peak = 168
Prominant peaks:
97, 124, 141, 153, 167, 181



MDA

Molecular weight = 179
Base peak = 44
Prominant peaks:
51, 77, 81, 135, 136



Molecular weight = 323
Base peak = 221
Prominant peaks:
72, 167, 181, 196, 207

Experiment # 13

Name _____

IR Spectroscopy

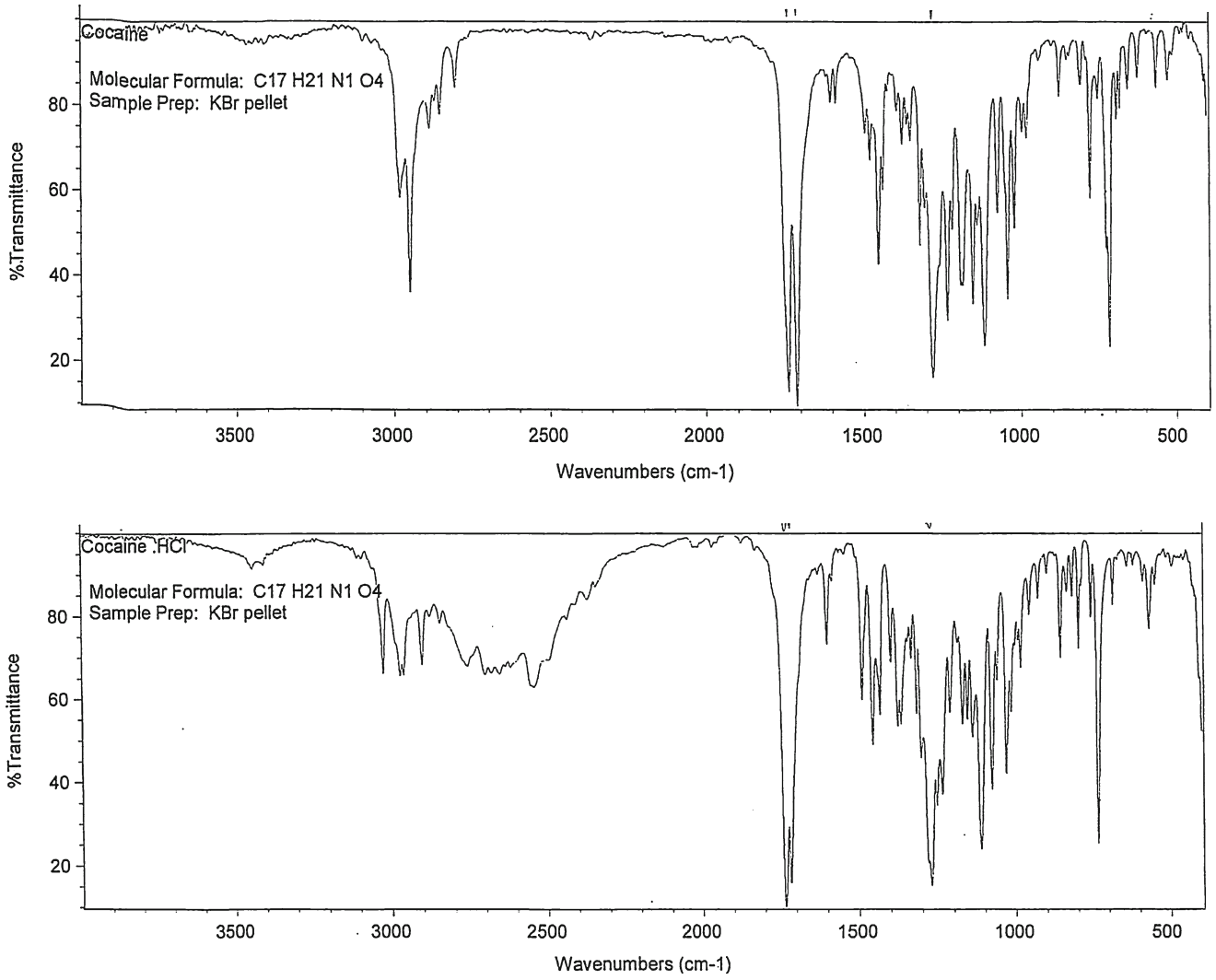
Reference: Chapter 11
Objective: The student will gain experience in the interpretation of infrared spectra.

Introduction:

Spectroscopy is the study of the interaction of atoms and molecules with electromagnetic radiation. These interactions often involve the emission or absorption of discrete amounts of energy, which is detected using analytical instrumentation. When energy, in the form of infrared radiation (IR), is absorbed by a particular substance, it produces measurable affects that are detected using an *infrared spectrometer*. Infrared radiation is a low energy form of radiation that is best described as "heat." When we feel the warmth of the sun, we are actually responding to infrared radiation. The heat emitted from the sun is absorbed by our bodies and stimulates molecules in our skin to vibrate. We interpret this vibrational energy as "warmth." A similar activity is observed in molecules. When infrared radiation is absorbed by a molecule, the chemical bonds convert the energy into molecular vibrations. IR spectroscopy measures the frequency of the radiation absorbed by a particular bond and records this as a band on an *infrared spectrum*. Tables containing bond vibrational frequencies are used to identify the bond from the absorption bands observed on the spectrum. The location of absorption bands on spectra are commonly represented in units of *wavenumbers* or *wavelengths*. The *wavenumber unit* (cm^{-1}, reciprocal centimeters) is used most often because it is directly proportional to vibrational energy and most modern spectrometers are linear in the wavenumber scale. Identification of a molecule based solely on an infrared spectrum is rare because IR spectroscopy is used to *identify specific bonds in a molecule*. For example, a carbonyl group (carbon–oxygen double bond) is easily identified by a characteristic absorption band present on the spectrum; however, ketones, aldehydes, carboxylic acids, and esters all contain a carbonyl group. Therefore, absolute confirmation of identity will require information from other analytical methods. Nonetheless, IR spectroscopy is often an important source of supporting evidence in the identification process.

Part A:

The IR spectrum of cocaine base and cocaine HCl are provided. Compare the two spectra and identify absorption bands common to both and those distinct to each spectrum (uncommon bands). Identify each band using wavenumber region and record your observations in the table below.



Wavenumbers (cm^{-1})

Common Bands

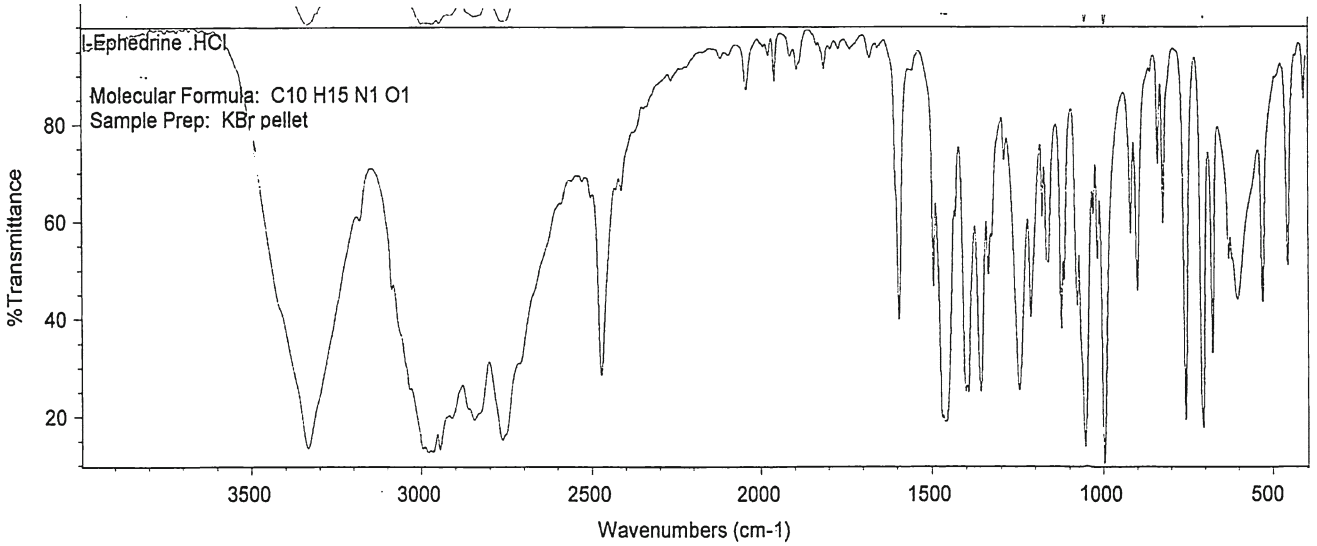
Distinct Bands

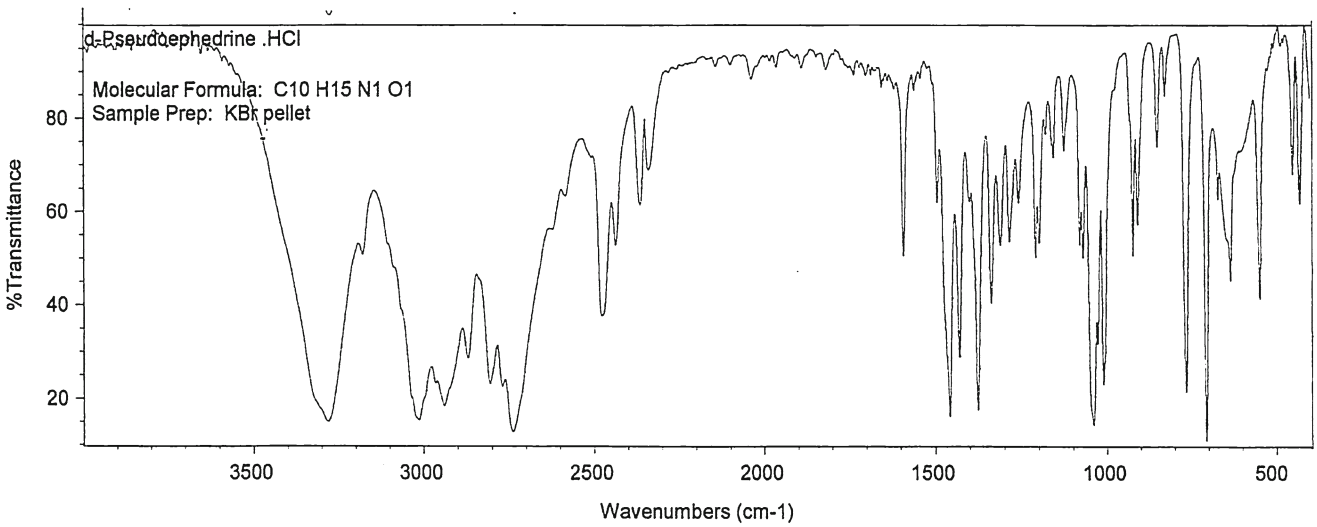
Cocaine HCl

Cocaine Base

Part B:

The IR spectrum of ephedrine and pseudoephedrine are provided. Compare the two spectra and identify absorption bands common to both and those distinct to each spectrum (uncommon bands). Identify each band using wavenumber region and record your observations in the table below.





Wavenumbers (cm^{-1})

Common Bands

Distinct Bands

Ephedrine

Pseudoephedrine

Experiment # 14

Name _____

Examination of Marijuana (moot)

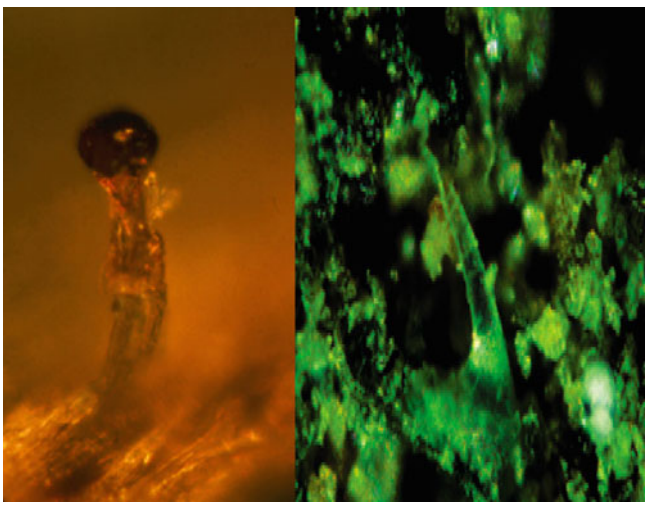
Reference: Chapter 12

Objectives: The student will gain experience in the forensic identification of marijuana plants. A report of findings will also be written and presented.

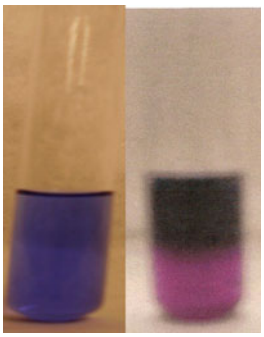
A plant material case submission contains following information/evidence.

Visual Inspection:

Microscopic Investigation:



Chemical Investigation: Duquenois-Levine test results



Questions:

1. Describe the physical characteristics of the leaves based on visual inspection.

2. Describe the physical characteristics of the seeds based on visual inspection.

3. Describe your observations from microscopic investigation. What types of hairs are visible?

4. The Duquenois-Levine test was performed as part of the chemical investigation.
   (a) What reagents are found in the top layer of the Duquenois-Levine test?

   (b) What reagents are found in the bottom layer of the Duquenois-Levine test?

   (c) What is the result of the Duquenois-Levine test?

5. What conclusions can be made based on your results in this investigation?

Experiment # 15

Name _____

Examination of Controlled Substances: Primary and Secondary Amines (moot)

Reference: Chapter 13

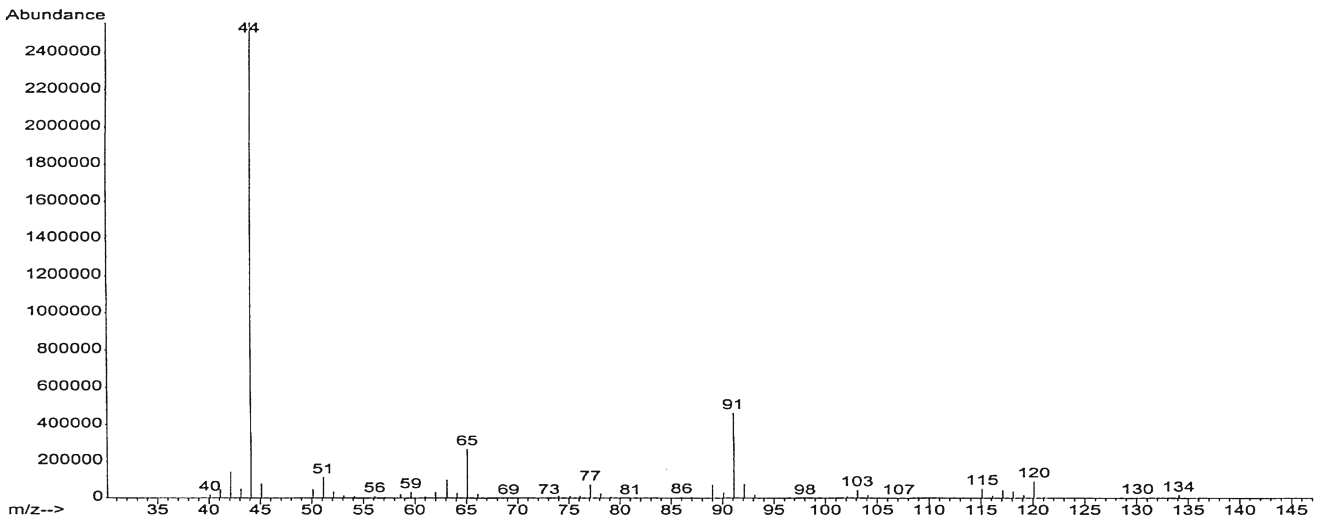
Objective: Students will gain experience in the forensic identification of controlled substances classified as primary and secondary amines using case data.

You are provided forensic data for three separate examination scenarios. Carefully study the information and predict the controlled substance(s) in each profile. Write a report of your findings.

Part A:

White Crystals:

Mass Spectrum:



Questions:

1. Name the color-screening tests that you would perform in this case.

2. Predict the results of the screening tests in question 1.

3. Describe the method you would use as a confirmatory examination.

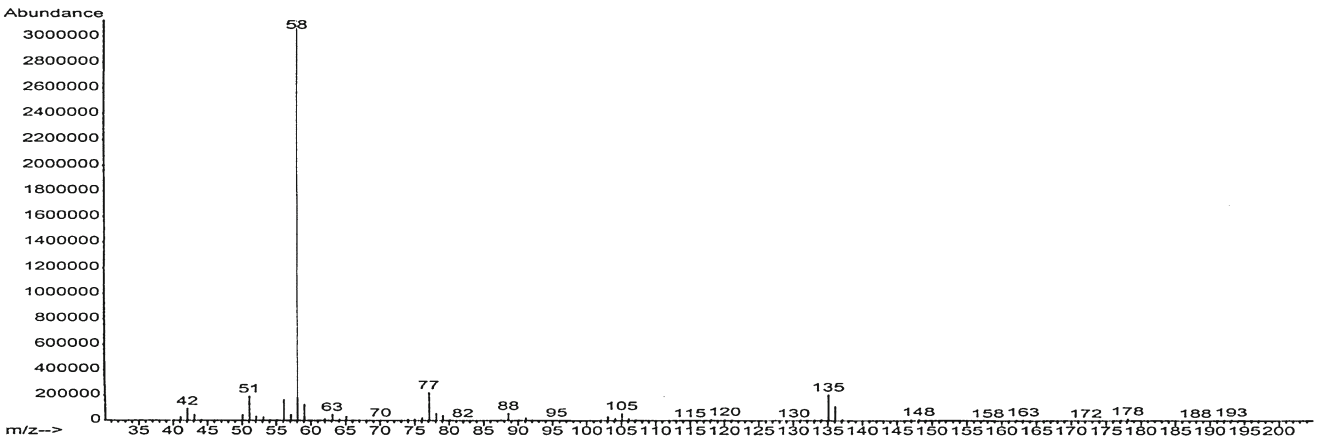
4. What type of extraction would be performed to prepare a sample for GCMS analysis?

5. Identify the controlled substance and provide supporting data for your conclusion.

Part B:

Red Tablets

Mass Spectrum



Questions:

1. Name the color-screening tests that you would perform in this case.

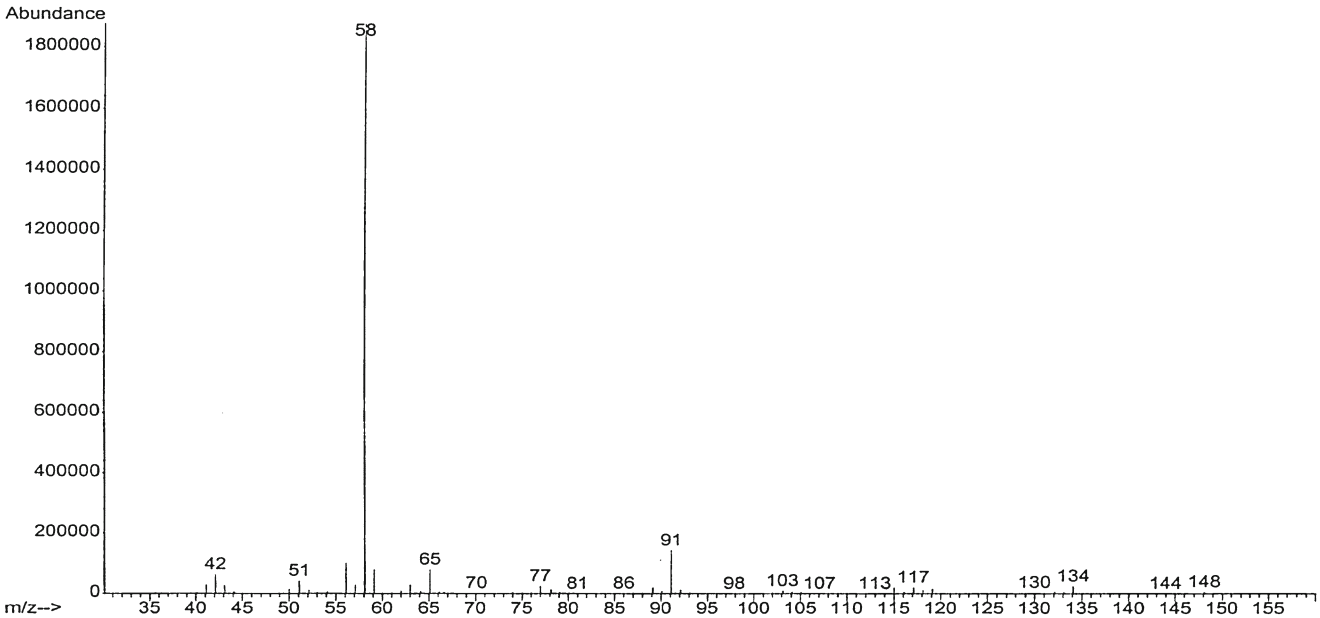
2. Predict the results of the screening tests in question 1.

3. Describe the confirmatory method that you would use to identify this substance.

4. Identify the controlled substance and provide supporting data for your conclusion.

Part C:
Light Brown Powder

Mass Spectrum



Questions:

1. What color-screening tests would you perform in this case?

2. Predict the results of the screening tests in question 1.

3. Describe the confirmatory method that you would use to identify this substance.

4. What type of extraction would you perform to prepare a sample for GCMS analysis?

5. Identify the controlled substance and provide supporting data for your conclusion.

Experiment # 16

Name _____

Examination of Controlled Substances: Tertiary Amines and Opiates (moot)

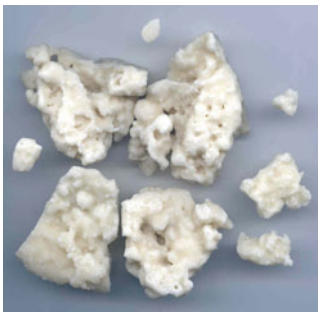
Reference: Chapter 14

Objective: Students will gain experience in the forensic identification of the tertiary amine class of controlled substances using case data provided.

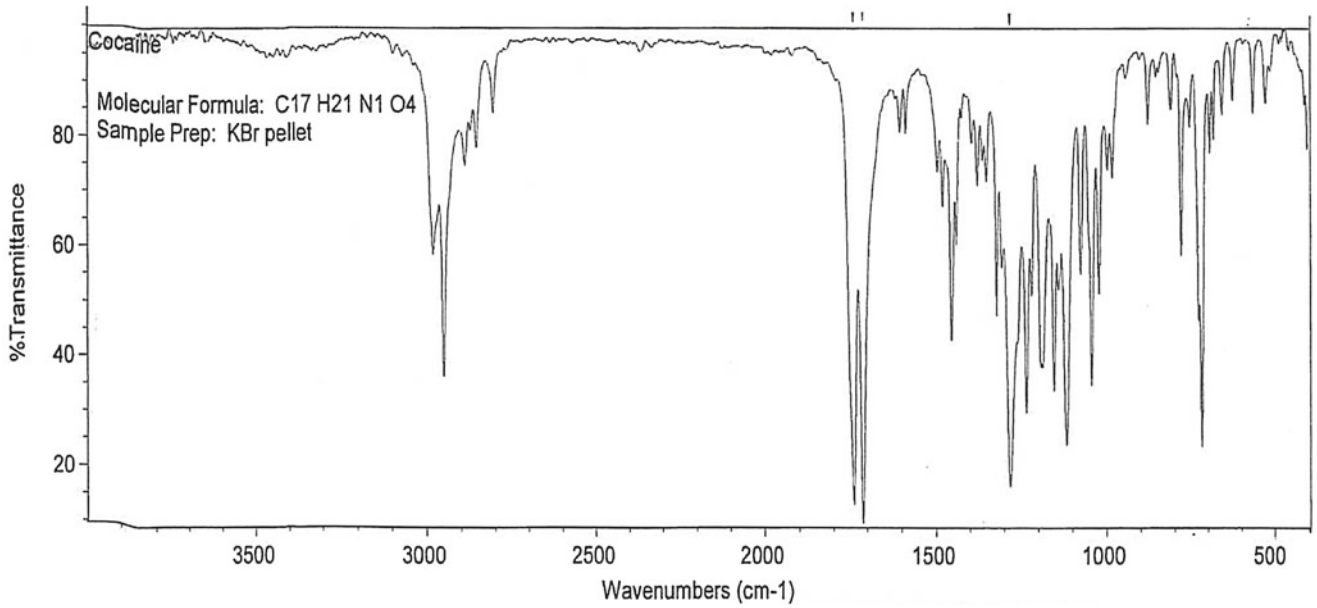
You are provided forensic data for three separate examination scenarios. Carefully study the information and predict the controlled substance(s) in each profile. Write a report of your findings.

Part A:

Chunks of Tan/White Substance



IR Spectrum



Questions:

1. What color-screening tests would you perform in this case?

2. Predict the results of the screening tests in question 1.

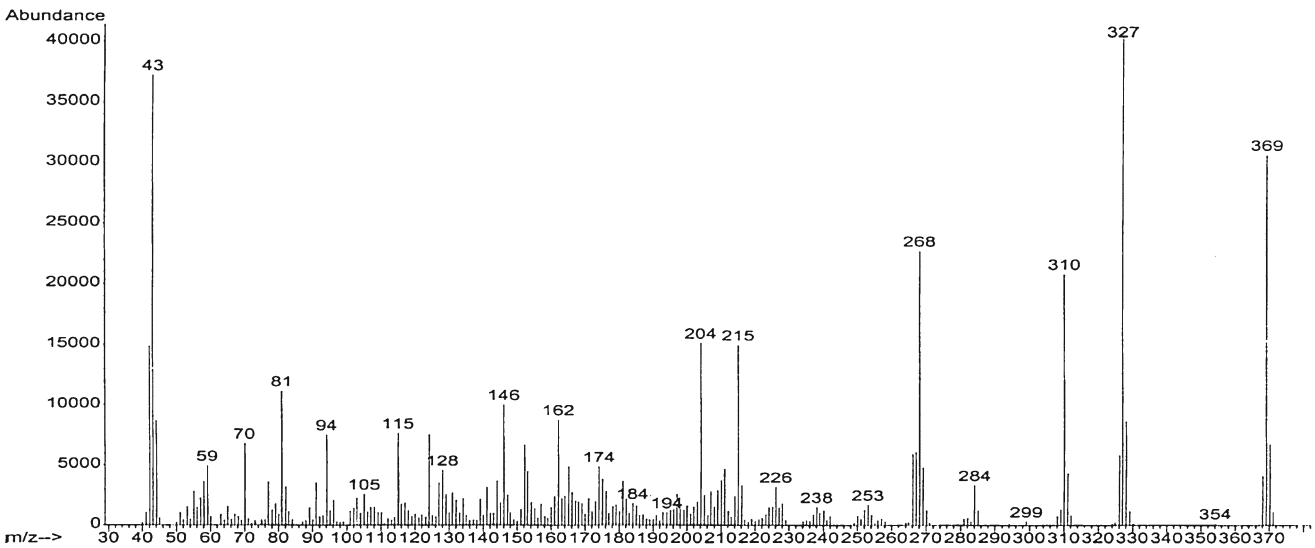
3. Describe the confirmatory method that you would use to identify this substance.

4. Identify the controlled substance and provide supporting data for your conclusion.

Part B:

Black Chunk

Mass Spectrum



Questions:

1. What color-screening tests would you perform in this case?

2. Predict the results of the screening tests in question 1.

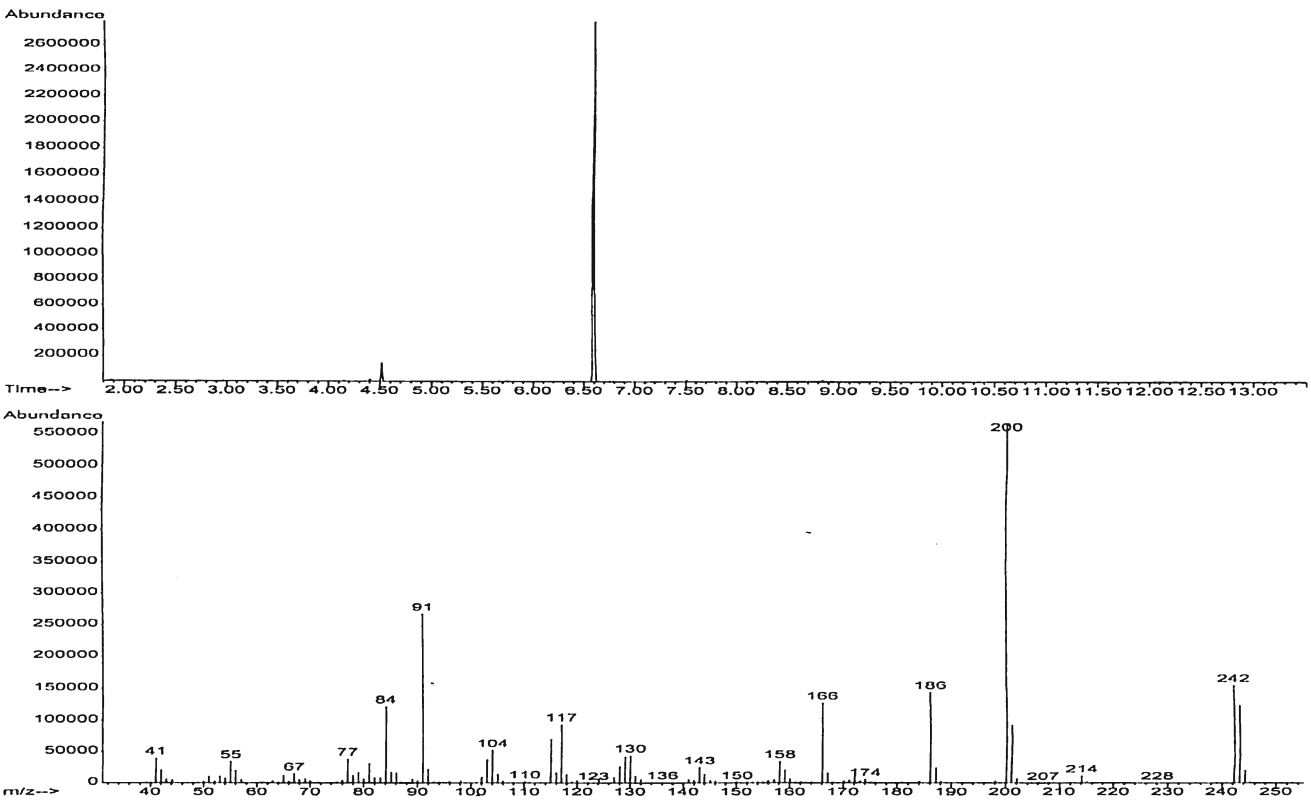
3. Describe the confirmatory method that you would use to identify this substance.

4. Identify the controlled substance and provide supporting data for your conclusion.

Part C:

Stained Cigarette and Yellow Oil

GCMS spectra



Questions:

1. What color-screening tests would you perform in this case?

2. Predict the results of the screening tests in question 1.

3. Describe the confirmatory method that you would use to identify this substance.

4. Identify the controlled substance and provide supporting data for your conclusion.

Experiment # 17

Name _____

Examination of Controlled Substances: Tryptamines (moot)

Reference: Chapter 15

Objective: Students will gain experience in the forensic identification of the tryptamine class of controlled substances.

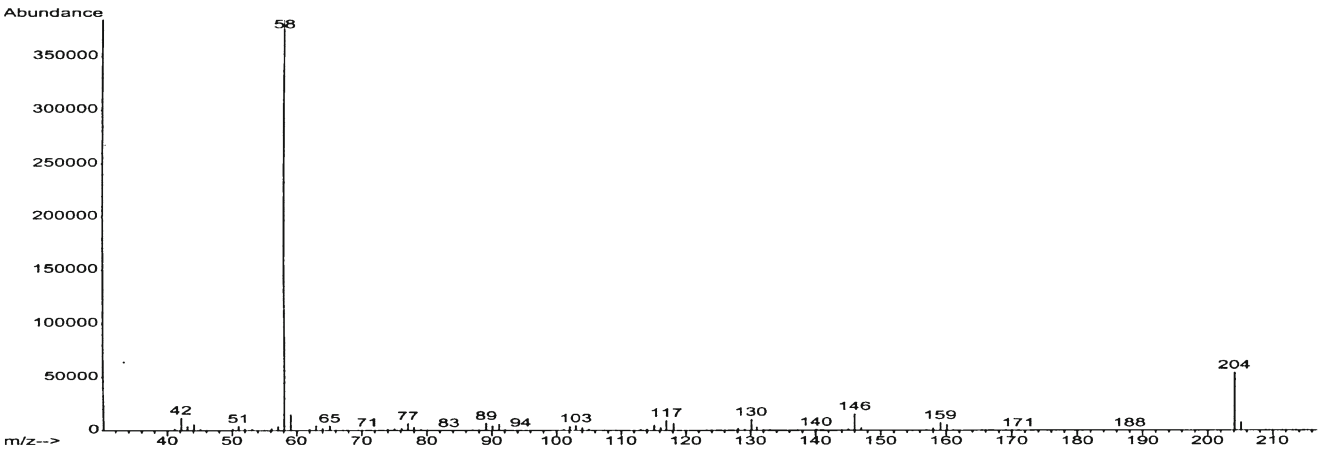
You are provided forensic data for three separate examination scenarios. Carefully study the information and predict the controlled substance(s) in each profile. Write a report of your findings.

Part A:

Mushrooms

Results of Thin-Layer Chromatography (TLC)

Mass Spectrum:



Questions:

1. What color-screening tests would you perform in this case?

2. Predict the results of the screening tests in question 1.

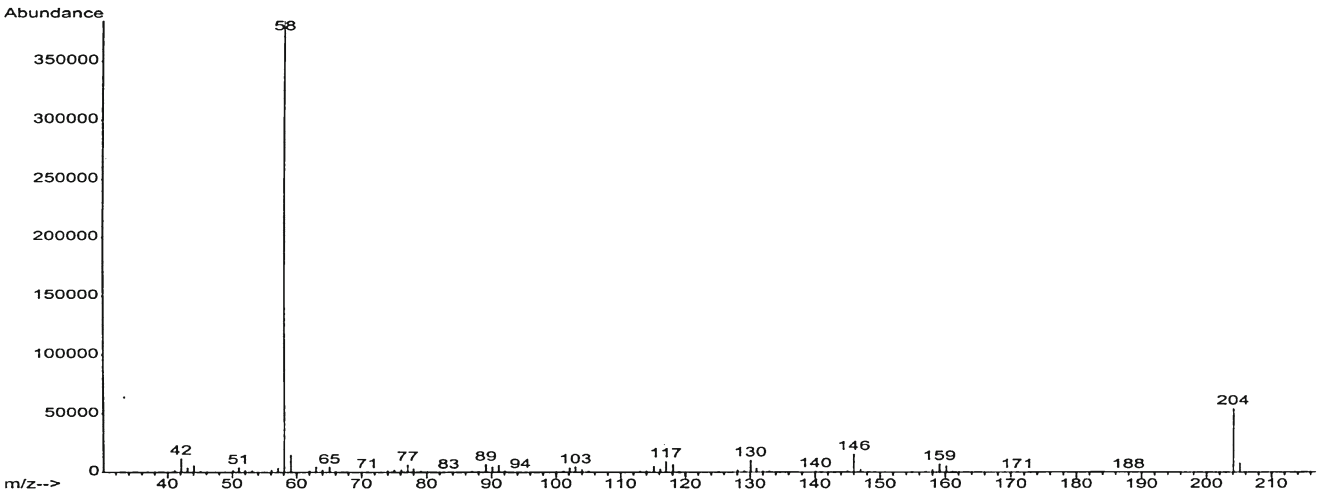
3. Describe the confirmatory method that you would use to identify this substance.

4. Identify the controlled substance and provide supporting data for your conclusion.

Part B:

Plant Material

Mass Spectrum:



Questions:

1. What color-screening tests would you perform in this case?

2. Predict the results of the screening tests in question 1.

3. Describe the confirmatory method that you would use to identify this substance.

4. Identify the controlled substance and provide supporting data for your conclusion.

Experiment # 18

Name _____

Examination of Anabolic Steroids (moot)

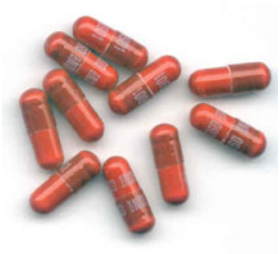
Reference: Chapter 16

Objective: Students will be exposed to the forensic identification of anabolic steroids.

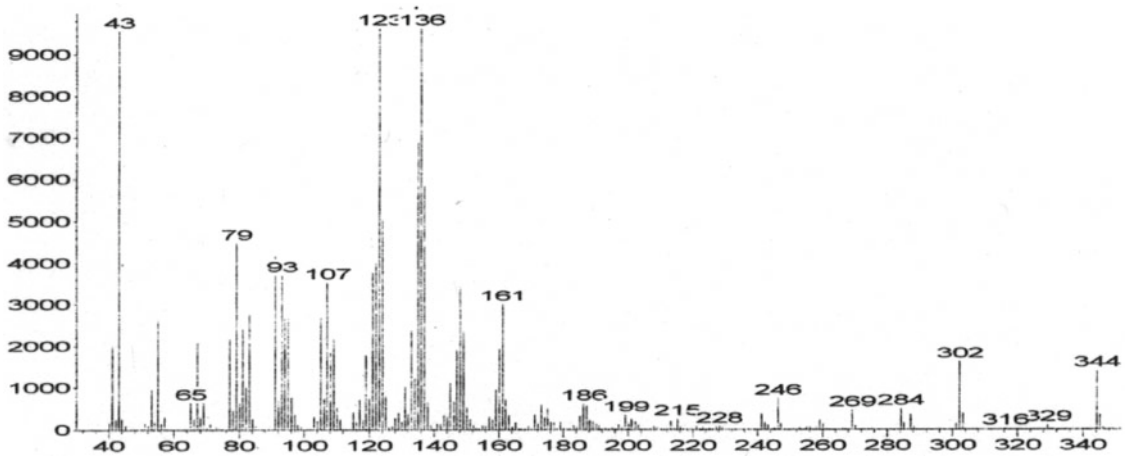
You are provided forensic data for three separate examination scenarios. Carefully study the information and predict the controlled substance(s) in each profile. Write a report of your findings.

Part A:

Red Capsules



Mass Spectrum



Questions:

1. What color-screening tests would you perform in this case?

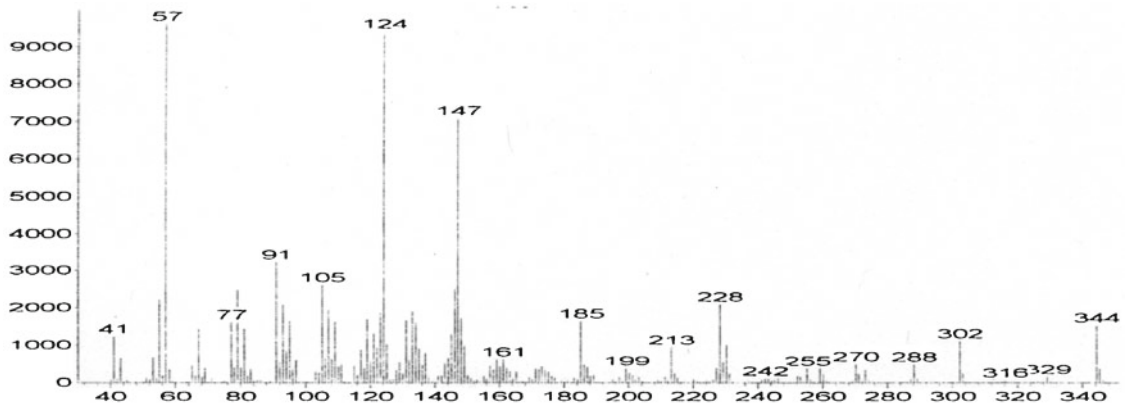
2. Predict the results of the screening tests in question 1.

3. Describe the confirmatory method that you would use to identify this substance.

4. Identify the controlled substance and provide supporting data for your conclusion.

Part B:
Injection Vial

Mass Spectrum:



Questions:
1. What color-screening tests would you perform in this case?

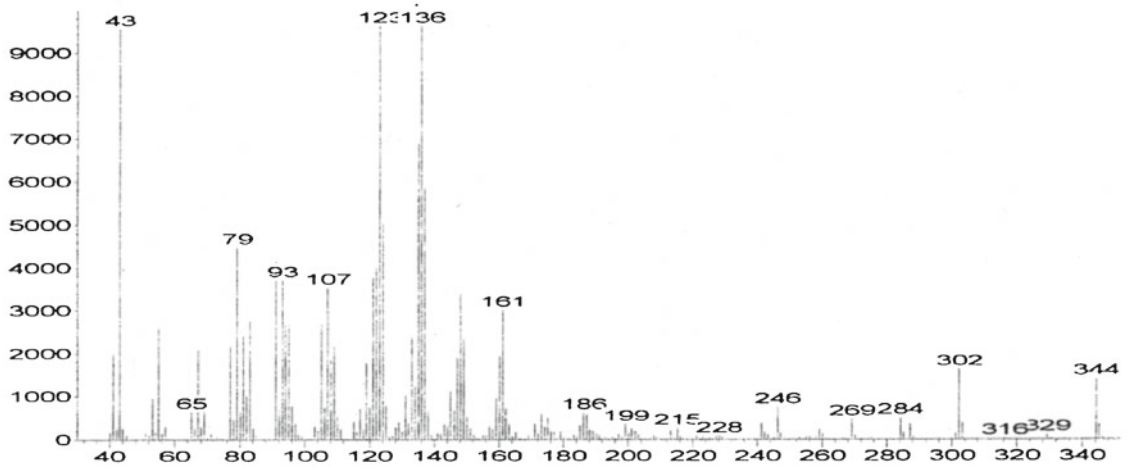
2. Predict the results of the screening tests in question 1.

3. Describe the confirmatory method that you would use to identify this substance.

4. Identify the controlled substance and provide supporting data for your conclusion.

Part C:
Tablets

Mass Spectrum:



Questions:

1. What color-screening tests would you perform in this case?

2. Predict the results of the screening tests in question 1.

3. Describe the confirmatory method that you would use to identify this substance.

4. Identify the controlled substance and provide supporting data for your conclusion.

Experiment # 19

Name _____

Examination of Miscellaneous Controlled Substances (moot)

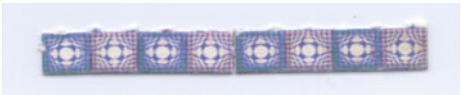
Reference: Chapter 17

Objective: Students will gain experience in the forensic identification of various functional groups present in different controlled substances.

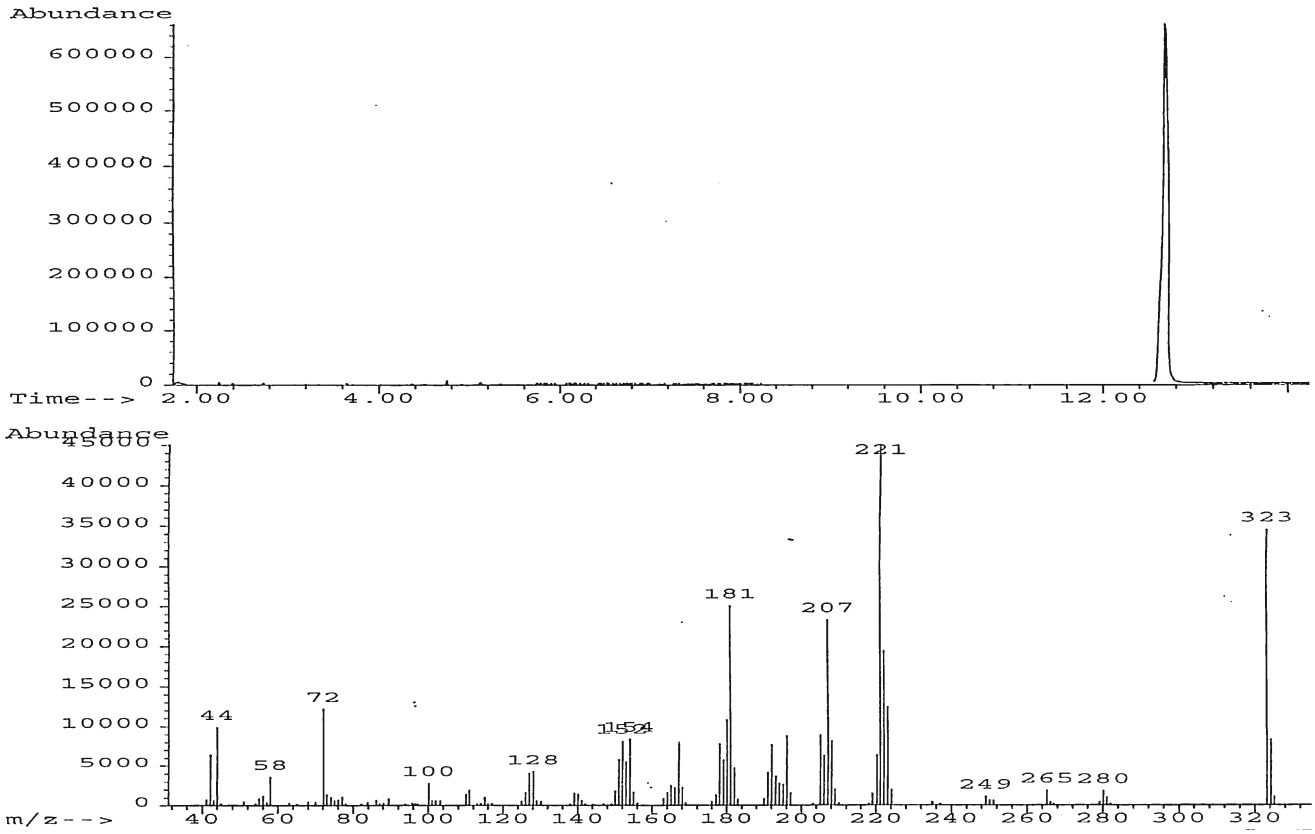
You are provided forensic data for three separate examination scenarios. Carefully study the information and predict the controlled substance(s) in each profile. Write a report of your findings.

Part A:

Paper Tabs



GSMS Spectra



Questions:

1. What color-screening tests would you perform in this case?

2. Predict the results of the screening tests in question 1.

3. Describe the confirmatory method that you would use to identify this substance.

4. Identify the controlled substance and provide supporting data for your conclusion.

Part B:

Capsules

Questions:

1. What color-screening tests would you perform in this case?

2. Predict the results of the screening tests in question 1.

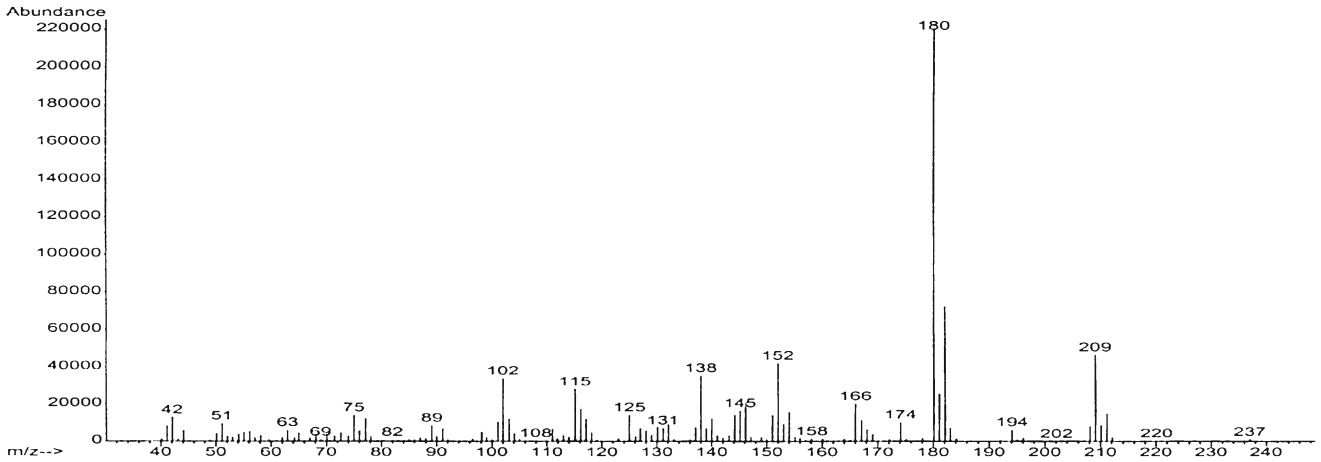
3. Describe the confirmatory method that you would use to identify this substance.

4. Identify the controlled substance and provide supporting data for your conclusion.

Part C:

Tablets

Mass Spectrum:



Questions:

1. What color-screening tests would you perform in this case?

2. Predict the results of the screening tests in question 1.

3. Describe the confirmatory method that you would use to identify this substance.

4. Identify the controlled substance and provide supporting data for your conclusion.

Experiment # 20

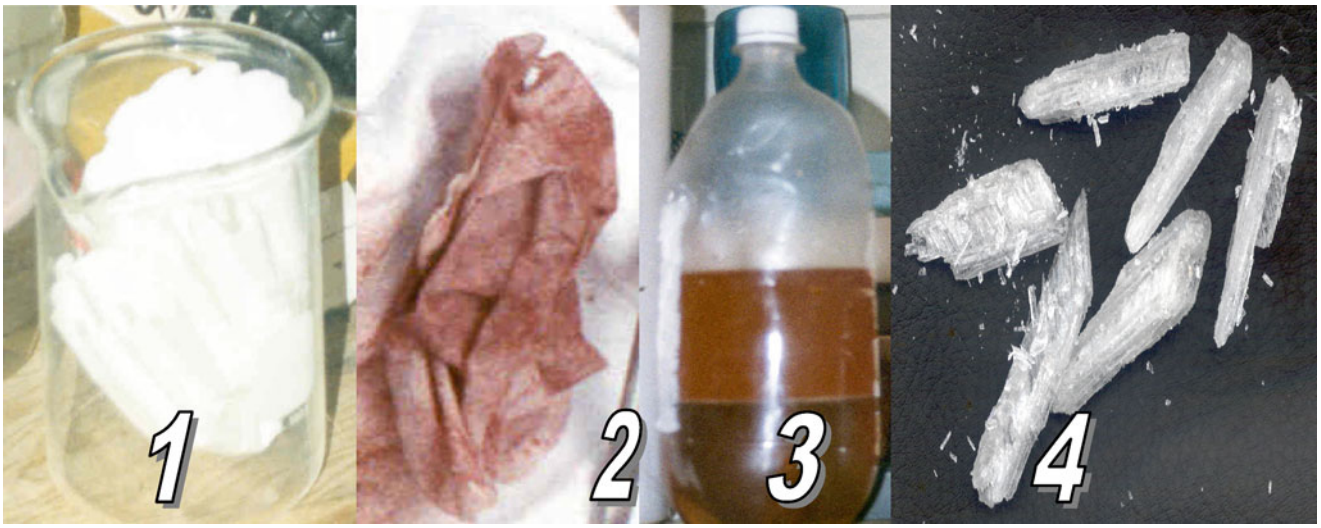
Name _____

Clandestine Manufacturing of Methamphetamine (moot)

Reference: Chapters 18, 19, and 20

Objectives: Students will gain experience in the forensic identification of case evidence collected at clandestine lab sites. This will be extended to include the association of distinctive evidence to specific synthetic steps used in the illegal production of methamphetamine.

Carefully study the photographs below and answer the following questions based on your observations. You will need to refer to your text.



1. Identify the method used in the production of methamphetamine. Justify your answer.

2. What chemicals are required for the method in question 1?

3. List the synthetic steps used in the production of methamphetamine using this method.

4. What synthetic step is indicated by the evidence shown in photograph 1?

5. List the chemical(s) required to prove this step.

6. Briefly outline the procedure you would use in the forensic examination of the evidence shown in photograph 1.

7. Summarize your conclusion on this piece of evidence.

8. What synthetic step is indicated by the evidence shown in photograph 2?

9. List the chemical(s) required to prove this step.

10. Briefly outline the procedure you would use in the forensic examination of the evidence shown in photograph 2.

11. Summarize your conclusion on this piece of evidence.

12. What synthetic step is indicated by the evidence shown in photograph 3?

13. List the chemical(s) required to prove this step.

14. Briefly outline the procedure you would use in the forensic examination of the evidence shown in photograph 3.

15. Summarize your conclusion on this piece of evidence.

16. What synthetic step is indicated by the evidence shown in photograph 4?

17. List the chemical(s) required to prove this step.

18. Briefly outline the procedure you would use in the forensic examination of the evidence shown in photograph 4.

19. Summarize your conclusion on this piece of evidence.

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