Preliminary test and solubility classification of organic compounds

Preliminary Test and Solubility Classification of Organic Compound Keene Louise Topacio, Christopher Jay Robidillo Abstract The experiment focuses on how to classify organic compounds by its functional groups. It is done by preliminary test and with the solubility test. Preliminary test used two known compounds also the unknowns. Physical state, color, odor, and ignition test were noted. The known compounds are inorganic and organic compounds. A comparison is made from theobservationin the unknown sample, by which we had concluded that there were two organic compounds and one inorganic among the unknowns.

In order to have a strong background about the solubility test, we first classify organic compounds of known functional group using the said test. Butyl bromide, ethanol sucrose, butyraldehyde, ethanoic acid, methyl amine, nitrophenol, diethyl ether, benzoic acid and propanone were used. It is found out that they are class X, Sn, S, N, Sa, Sb, S, N, A1 and Sn, respectively. We use water, ether, HCl, NaOH, NaHCO3, H2SO4, litmus paper, and phenolphthalein in classifying each compound based on their solubility and color change. It follows a certain scheme on testing the compound.

Each test will link to another test until we ended up with the class of that compound. We finally determine the class of the three unknowns. It is found out to be an Sb - amine -, S -salt -, and an N -aromatic compound-. Keywords: solubility, classification, organic compounds, solubility test, preliminary test Introduction One important part of experimental organic chemistry is to be able to analyze and identify an unknown organic compound from its functional groups. There are several steps in order to acquire this and there is no definite process. But there are systematic ways in different experimental organic chemistry books.

For this experiment it will emphasize the preliminary examination and solubility characteristics of some known organic compounds and unknown samples. Preliminary test is basically noting informations with lesser effort compared to the other tests the physical state, color, odor, and its ignition properties. Physical state of a substance can make a distinction among organic compounds for the reason that at room temperature most of organic compounds are in its liquid state. This is due to the intermolecular forces of attraction in the compound. The color is also informative because most pure organic compounds are white or colorless.

Some discolorations of brown color are effect of oxidation reaction. The odor of many organic compounds, especially the ones that have lower molar mass are highly distinctive. Also, functional groups have its' own different smell particularly alcohols, ketones, esters, aliphatic and aromatic hydrocarbons. Caution must be observed while smelling the unknown sample because large amounts of organic vapors should never be inhaled because of toxicity. The ignition test involves a procedure in which a drop or two of a liquid or about 50-100 mg of a solid is heated gently on a crucible with a bunsen burner flame.

Whether a solid melts at low temperature or only upon heating more strongly is then noted. The flammability and the nature of any flame from the sample are also recorded. A yellow, sooty flame is indicative of an aromatic or a highly unsaturated aliphatic compound; a yellow but non-sooty flame is characteristic of aliphatic hydrocarbons. The oxygen content of a substance makes its flame more colorless or blue; high oxygen content lowers or prevents flammability, as does halogen content. The unmistakable and unpleasant odor of sulfur dioxide indicates the presence of sulfur in the compound.

If a white, nonvolatile residue is left after ignition, a drop of water is added and the resulting aqueous solution is tested with litmus or pHydrion paper; a metallic salt is indicated if the solution is alkaline. Solubility of an organic compound in different solvents can present useful but not efficient information about the presence or absence of certain functional groups. It is because the basic principle in solubility is " like dissolves like". In order to have a more distinct result a systematic approach can be considered and it is by solubility classification. First is to test the solubility of the unknown in water.

Several structural features of the unknown can be deduced if it is water-soluble. It must be of low molar mass and will usually contain no more than four to five carbon atoms, unless it is polyfunctional. Also, it must contain a polar group that will form a hydrogen bond with water, such as the hydroxy group of an alcohol or a carboxylic acid, the amino functionality of an amine, or the carbonyl group of aldehydes or ketones. Esters, amides, and nitriles dissolve to a lesser extent, and acid. chlorides or anhydrides react with water rather than simply dissolving in it.

Test in ether for water soluble compounds are also observed this is to identify further the functional groups in ther unknown. On the other hand, alkanes, alkenes, alkynes, and alkyl halides are water-insoluble. Supplementary test such as solubility in hydrochloric acid, then sodium hydroxide, sodium bicarbonate, and finally tested in sulfuric acid must be done accordingly. Solubility in one or more of these acids and bases is defined in terms of the compound being more soluble in base or acid than in water and reflects the presence of an acidic or basic functional group in the water-insoluble unknown compound.

Methodology The experiment was divided into two parts and the group was given three unknowns. The first part was for the preliminary test. Data were recorded according to the physical state, color, and odor of the compound. Small amount of the test compounds, copper nitrate, acetic acid also the unknowns were placed in separate porcelain crucible. It was then heated and covered. Observations were noted. If residues were sighted it was tested for acidity or basicity using a drop of distilled water and a litmus paper. Also, a drop of 15% HCl was added to determine if there was formation of gas.

Lastly, flame test using a nichrome wire was also observed in order to determine if metals were present. For the second part of the experiment, solubility class test. 1mL of the solvents; water, ether, HCl, NaOH, NaHCO3, and H2SO4, were placed on separate micro test tubes. A drop or pinch of the test compounds; butyl bromide, ethanol, sucrose, butyraldeyde, ethanoic acid, methylamine, nitrophenol, diethyl ether, benzoic acid, propanone, and the unknowns, were added to the solvent. It was then mixed and observed if it dissolved or not, miscible or not, or if there are discoloration present.

The solubility flow chart was used in order to classify the compounds. Results and Discussion Preliminary test determines the physical state, color, odor, and the combustion reaction of the compound during ignition. It is shown in Table 1. That copper nitrate which is blue liquid has an odorless smell while it produces a green flame when it is ignited. On the other hand, an organic compound, acetic acid, is liquid and is colorless. It has a sour smell and it produces no residue when it is ignited. Withrespectwith our group samples, all are in liquid state while differs in odor.

A1 has a very foul odor, while A2 possess an alcohol like then A3 have a strong odor. A1 and A3 does not have a residue after it was heated it only evaporated and evolution of smelly (unpleasant) gas was also observed. In the case of A2 there were close to black residue and after ignition, a red flame was observed. Table 1 Preliminary Examination Data X| Cu(NO3)2| C2H4O2| A1| A2| A3| A| Liquid| Liquid| Liquid| Liquid| Liquid| B| Blue| Colorless| Colorless| Colorless| Colorless| C| odorless| sour| foul| Alcohol like| Strong| D| Green flame| orange| No residue| red| orange| X - compounds, A - physical state, B - color, C - odor, D - ignition test| Copper nitrate and acetic acid are both in liquid phase. Normally, an organic compound such as acetic acid is a colorless liquid. Copper nitrate, on the other hand, is in aqueous form. Although they are in the same phase, it doesn't mean that they have the same volatility and solubility. Upon ignition, their volatility differences are noticed. The presence of ash is observed in copper nitrate while on acetic acid there is no present. It is an indication that there was solid present in copper nitrate solution.

Since a solid is not volatile, we can easily identify the presence of solid upon ignition. Thus, it is an indication that the physical state of copper nitrate is actually solid. Finally, copper nitrate has color while acetic acid has none. It is because of the presence of metal in the aqueous copper nitrate. While for our unknowns, it was all in liquid form which has an ammonia-like smell A1, alcohol like smell A2 and strong smell A3. By that, the one will know that A1 is already an amine. When it is ignited, it produces no ash and no soot. It is an indication that there is no metal present.

Also, we found out that it was an organic compound because the observation was similar with acetic acid. By all obtained observation, it is possible that A1 is an amine. For A2, it was observed that it contains metal, for the reason that when it ignited the flame was red with that we could say that it is a salt. Unknown A3, was found out to be an organic compound because the observation was similar with acetic acid. We can't determine the compound with just preliminary test due to the insufficient data. Table 2. 1 Solubility Test Data Y| C4H9Br| C2H5OH| C12H22O11| C4H8O| C2H4O2|

A| | | | | | B| | | | | | C| | | | | | D| | | | | | E| | | | | | F| | | | | | G| -| | | -| -| | | | H| -| colorless| -| -| colorless| I| X| Sn| S| N| Sa| Y - Compounds, A - Water, B - Ether, C - HCl, D - NaOH, E- NaHCO3, F- H2SO4, G - Litmus Paper, H - Phenolphthalein, I - Solubility Class| Table 2. 2 Solubility Test Data (Known) It is found out in Table 2. 1 and 2. 2 that ethanol, sucrose, nitrophenol, ethanoic acid, methyl amine, diethyl ether and propanone are soluble in water while butyl bromide, butyraldehyde, and benzoic acid are not. (Table 2. can identify the intermolecular forces of attraction and polarity from the structure and the functional group) Since water is a polar compound (possesses hydrogen bond) it is a poor solvent for saturated hydrocarbons. As we all know, saturated hydrocarbons are not polar because they only posses Van der Waals intermolecular force. Due to long chains (or high molecular weights) do not affect by polarity greatly; unsaturated hydrocarbons such as aromatic hydrocarbons have similar solubility with saturated hydrocarbons. The introduction of halogen atoms does not alter the polarity appreciably.

It does no increase the molecular weight, and for this reason, the water solubility falls off. On the other hand, salts are extremely polar, the ones encountered in this work generally being water soluble. Y| CH5N| C6H5NO3| C4H10O| C6H5COOH| C3H6O| A| | | | | | B| | | | | | C| | | | | | D| | | | | | E| | | | | | F| | | | | | G| | | -| -| | | -| | H| pink| colorless| -| -| colorless| I| Sb| S| N| A1| Sn| Y - Compounds, A - Water, B - Ether, C - HCl, D - NaOH, E- NaHCO3, F- H2SO4, G - Litmus Paper, H - Phenolphthalein, I - Solubility Class|

As might expected, acids and amines generally are more soluble than neutral compounds. The amines probably owe their abnormally high solubility to their tendency to form hydrogen-bonded complexes with water molecules. This theory is also linked with the fact that the solubility of amines diminishes as the basicity decreases. It also explains the observation that many tertiary amines are more soluble in cold than hot water. Apparently at lower temperatures, the solubility of the hydrate is involved whereas at higher temperatures the hydrate is unstable and the solubility measures are that of the free amine.

Ethers, esters, ketones, aldehydes, alcohols, nitriles, amines, acids and amines may be considered together with respect to water solubility. Due to their similarity in structure with water, their solubility is high. For that, the solute-solvent interaction is strong because of their compatibility made possible by their common polarity. Because of the polar nature of water; compounds owe their solubility in it almost entirely to the polar groups which they may contain. The functional groups stated above are hydrophilic. As the hydrocarbon percentage increases, the hydrophobic part also increases.

As a result, the solubility in water of these compounds containing functional groups, decreases as the hydrocarbon chain is getting longer or the ring is present. The tendency of certain oxygen-containing compounds to form hydrates also contributes to water solubility. The stability of this hydrate is therefore, a factor in determining water and ether solubility. These compounds have great solubility in water because of their capability to form hydrates. It is the reason why diethyl ether and propanone are soluble in water.

On the other hand, although generally, aldehydes are soluble in water, butyraldehyde is not. It is due to long chain of the butyl hydrocarbon. For the same reason as for long chains of amines, aromatic alcohols and aromatic carboxylic acid do not exhibit solubility in water. Greater number carbon chains due to ring decreases the effect of polarity. For propanone, it doesn't just dissolve in water. It undergo reaction which is a nucleophilic addition reaction. As a consequence, it yields a diol. As for the compounds that are water soluble, we can still further classify them through their solubility in ether.

It is obtained that ethanol, ethanoic acid, methyl amine, propanone are soluble while insoluble compound are sucrose and nitrophenol. Since dipole bond is present on ether groups, it can differentiate short chain ethers, alcohols, carboxylic acid, ketone and amines from carbohydrates. Ethers cannot dissolve carbohydrates, such as sucrose. Thus, it can be classified as class S. Table 2. 3 Structure and Functional Groups of Known Compounds| Compound| Structure| Functional group| Butyl bromide, C4H9Br| | Alkyl halides| Ethanol, C2H5OH| | alcohols| Sucrose, C12H22O11| | Carbohyd-rates|

Butyraldehyde| | Aldehydes| Ethanoic acid| | Carboxylic acid| Methyl amine| | Amines| Nitrophenol| | Alcohols| Diethyl ether| | Ethers| Benzoic acid| | Carboxylic acid| Propanone| | Ketone| The water soluble organic compounds that are also soluble in ether are monofunctional group compounds. We can classify their functional group base on their acidity and basicity and it is done with the use of litmus paper. When the blue litmus paper is dropped in the test compounds, only at ethanoic acid the litmus paper changes its color to red while at ethanol, methyl amine, diethyl ether and propanone.

On the other hand, when red litmus paper is dropped on the test compounds, only methyl amine turned the R. L. P to blue while it remained at red for ethanol, diethyl ether and propanone. By that, we concluded that, ethanoic acid is an acid, methyl amine is a base while ethanol, diethyl ether and propanone are neutral. In the other realm, water-insoluble compounds such as butyl bromide, butyraldehyde, nitrophenol and benzoic acid are further tested with 5% HCl. It is obtained that none of these compounds are soluble in the dilute HCl.

The explanation for this is that the acid can only dissolve are basic amines. Aliphatic amines (of any class) forms salts (a polar compound) when it is mixed with the dilute acid because of its basicity. 5% hydrochloric acid is also polar so they are soluble. Those compounds that dissolve in 5% HCl will probably fall under class B, which are amines. Compounds that are insoluble in 5% HCl (all compounds) are then tested with 5% NaOH. It is observed that only benzoic acid is the only soluble compound. Compounds dissolve in aqueous base solutions because they form sodium salts hat are soluble in aqueous medium. Probably, it is the only compound which is sufficiently acidic to form salt with the dilute base. Upon comparison, carboxylic acid is the functional group which is the most acidic among all organic functional group aside from sulfonic and sulfinic acids. To prove it, we can compare the acid constant of the functional group of each compound tested. The soluble benzoic acid can still be classified as either a strong organic acid and as a weak organic acid. Upon mixing, it is found that it is a strong organic acid because it dissolves with the reagent.

Thus, benzoic acid will fall under class A1 The compounds insoluble with 5% NaOH which are the butyraldehyde and butyl bromide and diethyl ether are further categorized. Upon mixing those compounds with the concentrated sulfuric acid, only butyraldehyde dissolves. So, butyraldehyde is on class X, while butyl bromide and diethyl ether are on class N. As we all know, butyraldehyde contains a functional group with oxygen (aldehyde), so it can undergo sulfonation with the concentrated acid. On the other hand, butyl bromide is saturated and has no functional group with O atom so it can’t undergo sulfonation.

Thus it is not soluble. Table 3. 1 Solubility Test Data (Unknown) unknown| A | B | C | D | E | F | 1| | | | | | | 2| | | | | | | 3| | | | | | | A - Water, B - Ether, C - HCl, D - NaOH, E- NaHCO3, F- H2SO4 For solubility test of the 3 unknowns of the group, it is on Table 3. 1 and 3. 2, we initially confirm its solubility in water. It is found that A1 and A2 are soluble. From that, we can anticipate that our sample can be a monofunctional group compound with short chain or a carbohydrate or salts. So, the next steps for the water-soluble compounds were to check its solubility with ether.

When we mixed ether and our samples were immiscible dissolves. From that, we may say that one is a salt. From the smell of the unknown A1 we already know that it will fall under amine that has a short chain so we stll tested it with litmus paper because amine are basic. It is observed that when red L. P. is exposed in the sample, it turned to blue, whereas the blue L. P. have no color change when it is exposed. It is an indication that our sample is basic. Thus, our assumptions are correct that it is a short-carbon chain, monofunctional amine and it falls under class SB. Table 3. Solubility Test Data (Unknown) unknown| 1| 2| 3| a| | | -| | | | b| Pink| -| colorless| c| Sb| S| X| Possible compounds| Monofunctional alcohols, aldehydes, ketones, amides, esters, aromatic amines, nitriles, and amides with five or fewer carbons. | Salts of organic acids; amine hydrochlorides, amino acids, polyfunctional compounds with hydrophilic functional groups, carbohydrates, polyhydroxy compounds, polybasic acids, etc. | Saturated hydrocarbons, haloalkanes, arylhalides, other deactivated aromatic compounds, diaryl ethers| a - Litmus Paper, b - Phenolphthalein, c - Solubility Class|

For A3, being immiscible to water it was tested with HCl, NaOH, $NaHCO_3$, and H_2SO_4 for its acidity or basicity. Unfortunately, it failed the entire tests so sum it up to a class X compound. Conclusion The objectives of the experiment were obtained. We were able to examine compounds by using the preliminary test. We were able to recognize the solubility class of each known compound given to us with the aid of solubility test. Finally, we were also able to classify organic compounds based on their solubility in some other compounds and on their acidity and basicity.

The analysis and identification of organic compound for this particular experiment consists of two parts which are probably adopted from common schemes. This systematic approach enables us to have a precise analysis without spending much reagents, thus it is useful and efficient tool of identifying organic compounds. Both preliminary test and solubility test are considered. We concluded that preliminary test and solubility test are effective ways of classifying organic compounds. In preliminary test, we had seen the different properties of different compound by its physical state, color, odor and ignition test.

We had compared the result of our unknown sample with the known ones, and upon comparison, we concluded that our sample is an organic compound which contains no metal. We had differentiated an organic compound from a non organic compound. An organic compound may be in a form of a solid or liquid, may be colored or not depending on the degree of conjugation, produces odor and has no ash during ignition because of the absence of metal. For our unknown sample, the significant observation is that its fishy, ammonia-like smell. Since it is an organic compound, we can say that it is an amine.

By the study of solubility data it has been found possible to lay down certain generalizations which often enable one to predict the solubility behavior of a compound merely by inspection of its structural formula. In the solubility test, we found out the different class of the different organic compound. It is obtained that water soluble compounds are compounds which have a short-carbon chain compounds with monofunctional group and are carbohydrates. For ether, it is capable dissolving short-carbon chain compounds due to their common polarity.

It is a way of differentiating carbohydrates from that. For those ether-soluble, their classifications are identified based on their acidity or basicity since they are monofunctional. Acidic groups are carboxylic acids, basic groups are amines while neutral groups are alcohols, aldehydes, ketones, and some carboxylic acid derivatives except the acid halides and anhydrides. For those insoluble compounds, a test for solubility with 5% HCl is made to identify strong bases such as amines that have longer chains or aromatic ring. The dilute acid can only form salt with strong bases, to dissolve.

On the other hand, 5% NaOH is then used to check for the presence of acidic organic compounds. For acids, they can form salt with strong base such as NaOH, thus making it soluble. The 5% NaHCO3, which is a weak base is used to identify the degree of acidity of those obtained acidic organic compounds. Strong acidic acids are soluble with 5% NaHCO3 while weak organic acids are not. Lastly, neutral compounds with long chains or ring remained. The concentrated sulfuric acid is used in identifying functional group that has O and organic compounds that are unsaturated.

Those compounds undergo sulfonation, making it soluble with the reagent. Solubility plays a major role in the identification and characterization of an organic compound. Through certain chemical tests, one can identify an organic compound. These concepts regarding the solubility of organic compounds are primarily applied in identifying the nature and properties of carbon-containing compounds as wells as predicting their behavior and reaction mechanism when allowed to be reacted with specific solvents during different chemical processes. References Baluyut John Y. G. , De Castro Kathlia A. Organic Chemistry Laboratory for Chemical Engineering Students Part 2, 2004 Klein, David (2012). Organic Chemistry. Danvers. John Wiley & Sons, Inc. , Gilbert, John. Experimental Organic Chemistry: A miniscale and Microscale Approach 5th Ed. Australia, Brooks/Cole Cengage Learning. http://www2. volstate. edu/chem/2020/Labs/classification. pdf http://www. sharjah. ac. ae/English/Academics/CentralLabs/asl/cl/Pages/IdentificationofOrganicCompounds. aspx http://www. wellesley. edu/Chemistry/chem211lab/Orgo_Lab_Manual/Appendix/ClassificationTests/index. html http://wwwchem. uwimona. edu. jm/lab_manuals/c10expt25. html