Qualitative test for organic compounds pdf

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Qualitative analysis of organic compounds Analysis and identification of unknown organic compounds is a very important aspect of experimental organic chemistry. There is no specific established procedure that can be applied as a whole to organic qualitative analysis. Different books have different approaches, but a systematic approach based on the diagram below will yield satisfactory results. High-quality tests, which require a significant number of (often dangerous) chemicals to be available in the laboratory for experimental use, often gradually move out of organic chemistry in favor of modern spectroscopic methods. In order to deduce the identity of the two unknowns, you will combine one qualitative test, which for the classification of halids, with modern analytical methods of infrared spectroscopy (IR) and mass spectrometry (MS). The development of quality tests can be found in this app and Hornback on page 291. General Scheme Analysis A. Preliminary Test Note Physical Characteristics: Solid, Liquid, Color and Smell. The yellow and red compounds are often very conjugated. Aminas often have a fishy smell, while esters usually have a pleasant fruity or floral smell. Acids have a sharp, biting smell. Odors may have illegal information about your unknown; it makes sense to sniff them with caution. Some compounds may have corrosive vapors or make you feel nauseous. B. Physical constants determine the boiling point or melting point. Distillation is recommended in the case of liquids. It serves the dual purpose of determining the boiling point as well as cleaning the liquid for subsequent tests. C. Solubility Tests Solubility Tests Solubility of the unknown in the following reagents provides very useful information. In general, about 1 ml of solvent is used with approximately 0.1 g or 0.2 ml (2-3 drops) of unknown compound. Help in analysing the results of your trial can be found in the solubility flowchart given in Fieser and Williamson on page 606. (Beware, this flowchart is much more complex than our lab requires, and can often make it more difficult to draw conclusions from your compatibility data..) D. Group classification tests After analyzing previous tests and IR spectrum compounds, if necessary, additional information can be obtained by watering carefully selected functional group classification tests. CHEMICAL Tests FOR IDENTIFIES ORGANIC FUNCTIONAL GROUPS Doc Brown's Chemistry Qualitative Methods of Analysis Revision Notes Doc Brown's Chemistry Revising Advanced Organic Chemical Identification Tests Part 2 Qualitative Tests to identify functional groups of organic molecules Following tests for the following organic groups are fully described, including explanations, methods, observations and equations, so described functional group tests for alkenes alkenes functional group hydroxy group - primary, secondary and tertiary alcohols ROH, phenols, carboxic group RCOOH, acil/acid amide group RCONH2, acel/acid chloride group RCOCl, aliphatic amin group R-NH2, aryl/aromatic amin, aldegid functional group RCHO, keton functional group RCOR halogenalcans/haloalcans (chloroalcan, bromoalcan, odoalcan) R-Cl, R-Br, RI, RCOOR esters and CHI3 iodoform test also - Part 11 qualifying test analysis for and identify organic functional the group notes Chemical Tests Index Part 1 Introduction Part 2 Qualitative Tests to identify organic molecules of functional groups of homologous groups of carbonates, ammonium ion and hydrogen ions (acids) Part 4 of Gaza, water and non-metallic elements Part 5 anion (negative and ions), including hydroxide (lye) APPENDIX 1. IDENTIFYING ELEMENTS from LINE SPECTRA (non-chemical testing method) some related revision references GCSE/IGCSE Revision to chemical tests to detect ions, gases and compounds Level quantitative analysis: acid-base, silver nitrate-chloride, EDTA Titration Level quantitative analysis: Redox Titration Full list of KEYWORDS for inorganic/organic identification techniques. test/reagent for: acid No qgt; H' - acid/akychloride RCOCl - alcohols - common ROH/prim RCH2OH/sec R2CHOH/tert R3COH) Aliphatic/aromatic carboxyline acids - alkaline - OH- - lye/alken-C-C'lt;/alkyne-CC- (saturated compared to unsaturated) - aluminium/aluminium ion Al3H2 - prim aliphatic amines R-NH2 - ammonia gas NH3 - ammonium ion NH4 - choppy aromatic Amin Bromine Br2 - Cs' cesium ion - calcium ion Ca2 with flame or hydroxic ppt. acid RCOOH - carboxyline acid (aliphatic) salt, for example, RCOO-Na' - chloride ion Cl- - chlorine gas Cl2 - Chomate (VI) ion CrO42- - copper (II) ion Cu2 flame or hydroxide ppt. 24DNPH (for aldehyde/ketone test) - RCOOR esters - test/fehling solution, flame test for metal ions, fluoride ion F- acid HZ - hydrogen bromide gas/hydrobromic acid HBr - hydrogen chloride gas/hydrochloric acid HCl - hydrogen iodide gas/hydrochloride Alkaline OH- hydroxia/alcohol/phenol (organic) CHI3 - iron (II) ion Fe2 - iron (III) ion Fe3 - ketone R2C'O - lead (II) ion Pb2 - lithium-ion ion li-ion lee (8)2 (aq) - ion magnesium Mg2 - metal carbonates nitrate or nitrate (V) nitrate (V) no3 - nitrite or nitrate (III) NO2- - nitrogen or nitrogen dioxide (IV) oxide NO2 - oxygen gas O2 - phenols C6H5OH, etc. iodide tests) - sugar (reduction) - sodium ion Naz - strontium-sulfate or sulfate (VI) SO42- or sulfate (IV) SO32- - So32- - So32- - So32- So32- - So32- So32- - So32- Sulphur dioxide gas SO2 - Tollen's reagent, unsaturated/saturated, water H2O and zinc ion (n2) - Use alphabetical test list above to identify the aon, cathes molecules etc. to find what you need! for your KS3-KS4 Science-GCSE-IGCSE-Chemistry and GCE-AS-A2-IB-USA classes 9-12 K12 advanced auxiliary chemistry courses, etc., and will help you identify unknown inorganic and organic compound-molecules for qualitative analysis. EMAIL REQUEST? Comment? No test? - Gas Preparations - WARNING signs/symbols-symbols marking TOP OF PAGE Analysis and identification of unknown organic compounds is a very important aspect of experimental organic chemistry. There is no specific established procedure that can generally be applied to organic qualitative analysis. Different books have different approaches, but a systematic approach based on the diagram below will yield good results. Students should, however, consult with the laboratory guide and the textbook of practical organic chemistry, A.I. Vogel (4th edition). Before setting out a general diagram, one or two points of practical significance should be noted. (a) The amount of substance for testing. For most tests, you should use about 0.1 grams of solid or 0.1 - 0.2 ml (2 - 3 drops) of liquid material (NOT MORE). (b) Reagents that can be performed as part of an organic analysis are on the shelves of reagents. Students are encouraged to develop general knowledge about the physical characteristics of common organic compounds. If you doubt the expected test result between a particular compound and the reagent, conduct a trial test with a known compound and compare it to an unknown one. (c) The amount of derivatives. Students have spent a lot of time and material in the past taking too much of the substance to prepare derivatives. In general, 0.5 - 1 g (or 0.5 - 1 ml) of the substance gives the most satisfactory results. If the practical book prescribes the use of large quantities (3 - 4 grams or more), the amount should be reduced to 1 g or 1 ml of unknown substance and the appropriate amount of reagents should be used. General analysis scheme A. Preliminary tests (a) Note the physical characteristics - solid, liquid, colored and odorous. (b) Conduct a ignition test (heat a small amount on a metal spatula) to determine whether the compound is althymatic or aromatic (i.e. a glowing flame - alyphatic; flame - aromatic). B. Physical constants determine the boiling point or melting point. Distillation is recommended in the case of liquids (see Annex 3). It serves the dual purpose of identifying B.P., as well as cleaning the liquid for subsequent tests. C. Analysis of elements present at the C10 level will tell you, but read the method. D. Solubility Tests Solubility Uncertainty in the following reagents provides very useful information. In general, about 3 ml of solvent is used with 0.1 g or 0.2 ml (2 - 3 drops) of the substance. The connection class can be listed from the following table: SOLUBILITY TABLE REAGENT AND TEST CLASS CLASS GROUP OF COMPOUNDS in cold or hot water. (If the unknown is soluble, do not perform the solubility tests below) Neutral, sour or basic. (Test with litmus test or universal indicator) Lower participants in the series. Neutral, for example, alcohol; Acid, for example, acids, phenols; Basic, such as amines soluble in dil. HCl Major most amines (except III amines only with aromatic groups soluble in dil. NaOH acid is the most acid, most phenols. Soluble in NaHCO3 Strongly acidic Most carboxylic acids. Insoluble in water, acid and alkaline neutral hydrocarbons, nitrohydrocarbons, alkyl or aril halides, esters and esters. Higher molecular weight of alcohols, aldehydes and ketones E. Group classification tests from previous tests can often deduce functional groups present in an unknown compound. Consult with i.r. spectrums if available. Individual tests are then carried out to identify and validate the functional groups present. NOTE: 1. Students are strongly advised not to conduct unnecessary tests because they are not only a waste of time, but also increase the likelihood of error. Thus, it is pointless to first test for alcohol or ketone in the basic composition containing nitrogen! Instead, tests for amines, etc. should be done at such a compound. The systematic approach in group classification tests cannot be overstated in order to avoid confusion and error. F. Literature Consultation Once the functional group has been identified, the reference is made to the tables in the book on organic analysis, to assess the possibilities and to prepare suitable solid derivatives. It should be noted that while two substances with the same functional group can sometimes have very similar b.p. or m.p., solid derivatives canusually be selected from literature, with m.p. differences of about 10 (or more) that distinguish the two possibilities. Example: COMPOUND B.P. DERIVATIVES (M.P.) 2.4-DNPH SEMICARBAONE Diethyl ketone 102 156 139 Methyl n-propyl ketone 102 144 112 G. Preparation derivatives Final feature prepared for suitable solid derivatives. The derivative should be carefully selected and its m.p. preferably be between 90 - - for ease of crystallization and m.p. definition. An attempt should be made to prepare one derivative. The derivative must be cleaned by redistlation, dried and m.p. determined. Derivatives should be submitted correctly labeled for evaluation along with the record. The results should be recorded systematically. The results must be recorded in the practical book at the time (not recorded afterwards). The record must be done by each test, regardless of whether THE NEGATIVE RESULT WAS OBTAINED. The results of trials, observations and conclusions should be given. At the end of the analysis, you should include a brief summary of the results with the title, b.p. or m.p., as well as the formula of the connection being analyzed. High-quality analysis of elements (reference only) In organic compounds, elements usually occurring together with carbon and hydrogen are oxygen, nitrogen, sulfur, chlorine, bromine and iodine. The detection of these elements depends on converting them into water-soluble ion compounds and applying specific tests. Lassaigne in sodium Fusion Test C, H, O, N, S, X NaX NaCN - Na2S NaCNS PROCEDURE Place a piece of pure sodium metal the size of peas in a thermouncular tube. Add a little compound (50 mg or 2 to 3 drops). When the charring begins, heat the bottom of the tube to dull the redness for about three minutes and finally dip the tube, at the same time hot, in a clean plate containing cold distilled water (6 ml) and immediately cover with clean wire gauze. A: The tube is destroyed, and any residual sodium metal reacts with water. Stir the mixture, boil for 1 to 2 minutes, on a tripod and filter hot through the fluted paper. The fusion filtrat, which should be clear and colorless, is used for SPECIFIC TESTS DESCRIBED BELOW: 1. To the part (2 ml) of the fusion filtrat is added 0.2 g of powdered sulphate crystals. Boil the mixture for half a minute, cool and acidify, adding diluted sulphuric acid dropwise. The formation of a bluish-green sediment (Prussian blue) or blue solution indicates that the original substance contains nitrogen. If precipitation does not appear, let stand for 15 minutes, filter out and inspect the filter paper. 2. SULPHUR (SULPHIDE) To cold synthesis filtrat (1 ml) add a few drops of cold, freshly cooked, diluted solution of sodium nitropruz. The latter can be prepared by adding a small solid crystal to 2 ml of water. The production of a rich purple color indicates that the original substance contains sulfur. This test is very sensitive. Only strong positive results are significant. 3. HALOGENS (HALIDES) Acidify part (1 ml) of 'fusion' phytrat with 2N acid, and if there is nitrogen and/or sulfur, boil for 1 - 2 minutes. The formation of heavy, white or yellow deposition of silver halide indicates a halogen. When obtaining a positive result: acidify the rest of the phytrast fusion diluted sulphuric acid, boil and cool. Add carbon tetrachloride (1 ml) and a few drops of freshly prepared chlorine water. Shake the mixture. (a) If the carbon tetrachloride layer remains colorless, it indicates chlorine. (b) If the carbon tetrachloride layer is brown , indicates bromine. c) If the layer of carbon tetrachloride is purple - indicates iodine. If nitrogen and/or sulfur are also present, the addition of silver nitrate to acidified fusion solutions will result in the deposition of silver cyanide and/or silver sulfide in addition to silver galids. Removal of hydrogen cyanide and/or hydrogen sulfide is carried out by boiling the solution merger. GROUP CLASSIFICATION TESTS Some functional group tests are listed below. Students should turn to a practical tutorial for detailed information as well as additional information, such as Vogel. Insatiety Tests 1. Cold diluted solution of potassium permanganate. 2. Bromine solution in carbon tetrachloride. Tests for compounds containing nitrogen 1. Amina (a) nitric acid. (b) Confirming tests. 2. Compounds that give amines or ammonia to acid or alkaline hydrolysis: Amides, replaced by amides, anilids, nitriles. 3. Compounds that give amines to shorten: Nitro, nitrozo, azo, hydraso, nitriles. Tests for compounds containing C, H and possibly oxygen 1. Na2CO3 carboxic acid or NaHCO3 solution will release carbon dioxide. 2. Phenols (a) sodium hydroxide solution (soluble). Insoluble in and no CO2 from NaHCO3 (except when electron attraction groups are present, such as 2.4-dinitrophenol). (b) Ferric chloride solution. (c) Bromine water. 3. Aldehydes and ketones (a) 2.4-dinitrophenylhydrazine (as Brady's reagent) for the C'O test. (b) Iodoform for CH3CO-. 4. Aldehydes only (decreased properties) (a) fehling solution. (b) Tollena reagent (AgNO3 ammonia solution). (c) Agent Jones. 5. Alcohol (a) Lucas reagent to distinguish between I, II and III alcohol. (b) Agent Jones. (c) Metallic sodium (use dry liquid and dry tube). 6. Sugar (a) Molisha test. 7. Oesters (a) Hydroxamic acid test. (b) Hydrolysis. Write about the identification of an unknown organic connection

Date..

A compound containing C, H (N, Hal, S) Physical characteristics.................. (solid, liquid, gas, colored, smell, etc.) Ignition test................................ Or alythic) Physical constant....................... (boiling point or melting point) Tests solubility (in tabular form) Group classification tests (in table form) Conclusion on observation testing from the above tests and and This connection is probably........................... (acid, phenol, aldehyde, etc.) Consultation literature (Opportunities) M.P. derivative (a) (b) (c) (c) Preparation derivative (cooking method) Observed m.p. derivative Lit. m.p. derivative Result Connection No................................. This is.. (give the formula) TESTS FOR FUNCTIONAL GROUPS I. UNSATURATED COMPOUNDS Two common types of unsaturated compounds - alkens and alkinas, characterized by carbon-carbon dual and triple bond, respectively, as a functional group. Two common quality tests for insatience are reactions of compounds with (a) bromine in carbon tetrachloride and (b) potassium permanganate. (a) 2% of the bromine in carbon tetrachloride dissolves 0.2 g (or 0.2 ml) of the compound in 2 ml of carbon tetrachloride or other suitable solvent and adds a solution of up to 2 ml 2 ml of bromine in carbon tetrachloride and shake. for example, the rapid disappearance of the bromine color to give a colorless solution is a positive test for insatiable. NOTE: The reagent is potentially dangerous. Keep it away from your skin and clothes; Protect your eyes and nose. (b) 2% of permanganate potassium dissolve 0.2 g (or 0.2 ml) of substance in 2 ml of water (acetone can also be used as solvent). Add a solution of potassium permanganate dropwise and observe the result. for example, for an empty definition, count the number of drops added to 2 ml of acetone before the color is retained. A significant difference in the number of drops needed in these two cases is a positive test for insatience. II. COMPOUNDS CONTAINING NITROGEN 1. Amina (a) The nitric acid reaction dissolves the amine (0.5 ml) in concentrated acid (2.0 ml) and water (3 ml) and cool the solution to 0 - 5 in the ice bath for 5 minutes. Add a cold solution (ice bath) of sodium nitrite (0.5 g) to the water (2.0 ml) from the drip, with a swirling test tube, still keeping the mixture in the ice bath. AMINE REACTION I aliphatic N2 has evolved. RNH2 - ROH and N2O - created aromatic diason salt. ArNH2 - HNO2 - ArN'N' Add a cold diasonium solution and swirl until cold solution 2-nafta (0.2 g) in 5% NaOH solution (2 ml). An orange-red dye is formed. In general, aliphatic and yellow oil nitrosamines are formed. II aromatic R2NH - HNO2 - R2N-NO - is not a visible reaction. - III aromatic Dianquilanylins give green solid compounds p-nitroso (if p-position is irrefutable). -I (b) Reaction with benzenesulphonyl chloride chloride reacts with primary and secondary, but not with tertiary amines, to give substitute sulfonamids. for example, (a) C6H5SO2Cl - H-NHR - NaOH - C6H5SO2NHR - NaCl - H2O (b) C6H5SO2Cl - C6H5SO2NR2 - NaCl - H2O Replaced sulfonamid, formed from primary amin, dissolves in alkaline environment, while the one produced from secondary amina is insoluble in lye. Place 0.5 ml (or 0.5 g) of compound, 15 - 10 ml 5% NaOH and 1 ml of benzensulphone chloride in a test tube, stop the tube and shake until the smell of sulphonic chloride disappears. The solution should be alkaline (if no reaction occurred, the substance is probably tertiary amine). If the sediment appears in the alkaline solution, dilute about 10 ml of water and shake; if the sediment is not dissolved, the secondary amin is indicated. If there is no precipitation, acidify it gently congo red with concentrated salt acid (added dropwise): the sediment indicates primary amin. 2. Amides R-CO-NH2 Simple primary amidi can be decomposed by boiling alkaline and thus developing ammonia. for example, CH3-CO-NH2 - NaOH - CH3-CO2- Na y NH3 Boil 0.5 g compound with 5 ml 10% sodium hydroxide solution and observe whether ammonia has evolved. III. COMPOUNDS CONTAINING C, H AND POSSIBLY OXYGEN 1. Carboxylic acids - a test with 5% aq. NAHCO3 R-CO2H - NaHCO3 - R-CO2- Na' CO2 - H2O Sodium hydrogen carbonate reacts with carboxycylic acids to give sodium acid salts and releases carbon dioxide. 2. Phenols soluble in NaOH and do not produce CO2 from NaHCO3 (a) bromine water phenols, usually highly react to electrophilic reagents and are easily bromine water bromine. For example, dissolve or suspend about 0.05 g of compound in 2 ml diluted salt acid and add dropwise water bromine until bromine color remains. White bromophenol sediment may form. Solid derivatives of bromofenol can be used to confirm the structure of phenol (wed preparation derivatives). (b) Ferric chloride test Most phenols react with iron chloride (III) to the formation of colored complexes. Colors vary - red, purple, blue or green - depending on various factors, such as phenolic compound used, solvent, concentration. Since some phenols do not give color, a negative test should not be taken as significant without additional information. Dissolve 0.05g of the compound in 2 ml water (or a mixture of water and ethanol if the compound is not water-soluble) and add the water solution of ferrikchloride dropwise. Observe any color changes that may occur. 3. Aldehydes and ketones (a) (as a Brady reagent) Test for a group of carbonyl (CLC) in aldehydes and ketones. 2.4-Dinitrophenylhydrazine gives economical soluble yellow or red 2.4-dinitrophenylhydrazons with aldehydes and ketones. Add 3 ml of the reagent into 2 drops of the compound in the test tube and shake. If the sediment is not formed immediately, heat and let stand for 5 - 10 minutes. Crystal sediment indicates the presence of a carbonyl compound. The bench reagent is very diluted and is designed only for qualitative tests and should not be used in the preparation of derivatives for identification purposes. (b) Iodoform test for CH3CO- Dissolution of 0.1 g (or 5 drops) of compound in 2 ml of water; If it is insoluble in water add enough dioxane to produce a homogeneous solution. Add 2 ml 5% naOH solution, then vimite potassium iodide - iodine dropwise with shaking until a certain dark color of iodine is preserved. Allow to stand for 2 - 3 minutes; If iodoform does not separate at room temperature, heat the test tube in a glass of water at 60. Add a few more drops of iodine reagent if the faint color of iodine disappears. Continue adding the reagent until the dark color is discharged after 2 minutes of heating into 60. Remove excess iodine by adding a few drops of diluted sodium hydroxide solution with shaking, dilute with equal amounts of water, and allow to stand for 10 minutes. The test is positive if the yellow sediment and the oroform is postponed. Filter yellow sediment, dry on filter paper pads and identify m.p. Iodoform melts at 120 (it can be repainting from methanol-water). The reaction is given by acetaldehyde and simple methyl ketones. Alcohol containing the CH3CHROH group will oxidize in reaction conditions, as well as give a positive test. 4. Only aldehydes (reduced properties). (a) Feling Aldehyde solution reduces Fehling solution to yellow or red copper oxide (I). Preparation of the reagent: Mix equal volumes of Fehling I solution (alkaline alkaline potassium tartrate) and Fehling II solution (copper sulfate solution). Add 2 drops (or 0.05 g) of the compound and 2 to 3 drops of reagent and heat in a boiling water bath for 3 to 4 minutes. The test is positive for alyphatic aldehydes, but is often indecisive for aromatic aldehydes, where Jones's reagent is often useful (see 5). (b) The tollenal reagent (Ammonite silver nitrate solution) aldehydes are easily oxidized by carboxylic acids and reduce the Tollen reagent to produce a silver mirror on the inside of a clean test tube. First clean the test tube with a little hot nitric acid (smoke cupboard) and rinse with distilled water. Preparation of the reagent: to 1 ml of silver nitrate solution add a few drops of sodium hydroxide. Then add the diluted ammonium hydroxide drop-by-drop until the sediment Add 2 to 3 drops of the compound in methanol to 2 to 3 ml of Tollen solution contained in a very clean test tube. If no reaction occurs in the cold, heat gently in the water bath. WARNING: After the test, pour the test tube contents into the sink and wash the test tube with diluted nitric acid. Any silver fulmit present, which is highly explosive when dry, will be destroyed. (see alcohol section). 5. Alcohol Tests for the hydroxyl group not only detect the presence of a group, but can also indicate whether it is primary, secondary or tertiary. (a) Jones reagent (CrO3-H2SO4 in H2O) This reagent distinguishes primary and secondary alcohol from tertiary alcohol; the test is based on a much greater resistance to tertiary alcohol oxidation than the other two types. Aldehydes also give a positive test. Place 1 ml of acetone in a test tube and dissolve one drop of liquid or about 10 mg of solid alcohol or aldehyde in it. Primary and secondary alcohol reacts within two seconds, as evidenced by the disappearance of the orange-colored reagent and the formation of green or blue-green sediment or emulsion. Tertiary alcohols do not react even after 3 minutes. (I) RCH2OH - RCHO --RCO2H (II) R2CHOH --R2C-O (III) R3COH - (b) Lucas's reagent nCl2 - conc. HCl This reagent converts the alcohols into the corresponding chlorides of alkyl. Zinc chloride (Lewis's acid) increases the reactivity of alcohols to acid. The test depends on the reaction rate of primary, secondary and tertiary alcohol with the reagent at room temperature. (I) RCH2OH - (II) R2CHOH --gt; R2CHCl and H2O (1 hour or maybe longer) (III) R3COH -'gt; R3CCl and H2O (immediately) Up to 1 ml of alcohol in a small test tube add 6 ml of Lucas reagent at room temperature. Cover the tube with a cork, shake and let stand. (i) Primary alcohols - aqueous phase remains clear (except allyl alcohol - drops after 7 minutes). (ii) Secondary alcohols are a very slow reaction (1 hour or maybe longer) when drops of alkyl chloride can be seen. (iii) Tertiary alcohols - a very quick reaction and drops of alkyl chloride formed almost immediately. 6. Sugar, Carbohydrate Test Molish Is a common test for carbohydrates. Dissolve 20 - 30 mg of the compound in 2 ml of water and add 0.5 ml of reagent (20% solution 2-nafta in ethanol). Pour 2 ml of concentrated sulfuric acid from the drip thoroughly down the side of the tube so that the acid forms a layer under the aqueous solution without mixing with it. Red coloring, moving on dark purple shapes on the interface. Conduct the second test on an empty solution. 7. Esters Hydroxamic R-CO-OR' Hydroxamic Test - H2N-OH --R-CO-NH-OH R'-OH Esters react with hydroxylamine in the presence of sodium hydroxide to form sodium salt corresponding hydroxamic acid. When acidification and addition of ferric chloride, a purple iron (III) hydroxamic acid complex is formed. It is always advisable to make sure that an unknown compound does not color with iron chloride (III) before conducting a hydroxamic acid test. Procedure for hydroxamic acid test (a) Ferric chloride test Dissolve a drop or a few small compound crystals in 1 ml 95% ethanol (corrected alcohol) and add 1 ml of hydrochloric acid M. Notice the color produced when added to the solution 1 drop of iron chloride (III). In a pronounced purple, blue, red or orange color, the hydroxamic acid test described below is NOT AN APP. (b) Mix 1 drop or several small crystals (about 0.05 g) of the compound with 1 ml of hydroxylamine 0.5 m in 95% ethanol and add 0.2 ml of sodium 6 M hydroxide. If the solution is cloudy, add 2 ml 95% ethanol. Observe the color produced when 1 drop 5% iron chloride solution (III) is added. If the resulting color is not retained, continue to add the reagent by a drop until the observed color permeates the entire solution. Usually only 1 drop of iron chloride solution (III) is required. Compare color to the color produced in the test (a). A positive test would be a distinct burgundy or magenta color compared to the yellow color observed in the testing of the original compound with iron chloride (III) solution in the presence of acid. It is often recommended to conduct a parallel test with, say, ethyl acetate to make sure that the conditions for this test are correct. PREPARATION OF DERIVATIVES OF ORGANIC COMPOUNDS Preliminary exam and group classification tests indicate a specific class (functional group) to which an unknown organic connection may belong. Further characterization and identification depends on the choice and preparation of a suitable solid derivative and the precise definition of its melting point (best, between 90 - 150). The following table lists some classes of organic compounds and a selection of derivatives that may be ready to characterize them. Check the melting points tables in Vogel which derivatives are most appropriate for the characteristics of your particular compound. CLASS OF COMPOSITE DERIVATIVES 1. ALCOHOLS 3.5-dinitrobenzoate 2. FENOL benzoate, acetate, bromo-derived 3. ALDEHIDES AND KETONES semi-scarbazone, 2.4-dinitrophenil-hydrazone, oxym 4. Anilid acids, amida, r-toluid. 5. AMINES benzoyl, acetyl and sulfonamed derivatives METHODS FOR WORK IOAKI (i) 3.5-Dinitrobenzoate chloride is usually partially hydrolyzed and should be prepared in a clean state, heating gently a mixture of 3.5-dinitrobenzoic acid (1 g) and pentachloride phosphorus (1.5 g) in a dry test tube until it sours (5 min.) of phosphorylchlorides removed by pressing a hard body with a spatula on a filtered packet of paper. Residual acid chloride is suitable for immediate use when cooking derivatives. Working under a smoke hood. Couples irritate the eyes and nose. Chloride 3.5 dinitrobenzolyl is mixed with alcohol (0.5 - 1 ml) in a poorly clogged dry test tube and heated on a steam bath for about 10 minutes. Wash a little carbonate solution, water and suck dry. Recrystallise from minimal hot ethanol or light oil. Cool slowly to avoid the formation of oil drops of your esther. FENOLS (i) benzoates (Sten-Bauman method). To phenol (0.5 g) is added 5% sodium hydroxide (10 ml) in well-clogged boiling tubes or a small conical flask. Benzoyl chloride (2 ml) is added in small quantities at a time, and the mixture is vigorously shaken with rare cooling under tap or in ice water. After 15 minutes the solid benzoate is separated: the solution should be alkaline at the end of the reaction; If not alkaline, or if the product is oily, add solid sodium hydroxide pellets and shake again. Collect the benzoate, rinse thoroughly with cold water and redistristize from alcohol or light oil. (ii) The acetates of many simple phenols are liquids; however, it is a suitable derivative for polyhydrols and replaced phenols. Phenol (0.5 g) dissolves in 10% of sodium hydroxide solution and an equal amount of crushed ice is added, followed by an acetic anhydride (2 ml). The mixture is vigorously shaken in the cork by a test tube until the acetate separates. The product is filtered and re-designed from alcohol. (iii) phenol bromine derivatives (0.3 g) are suspended in diluted hydrochlorin (10 ml) and bromine water added by drop until more decolourization occurs. Derivative bromo, which is sucked, filtered and redrawn from alcohol. ALDEHYDES AND KETONES (i) Semicarbazones Dissolve semicarbaside hydrochloride (1 g) and sodium acetate (1.5 g) in water (8 - 10 ml), add aldehyde or ketone (0.3 ml) and shake. Shake the mixture for a few minutes and then cool in the ice water. Filter from crystals, wash with a little cold water and recrystallise from methanol or ethanol. (ii) 2.4-dinitrophenylhydrazons suspend 0.25g 2.4-dinitrophenylggratazin in 5 ml methanol and gently add 0.5 ml of concentrated sulphuric acid. Warm filter and add a solution of 0.2 g of carbonyl compound in 1 ml of methanol. Recrystallise derived from methanol, ethanol or ethyl acetate. (iii) Oxymes hydroxylamine hydrochloride (0.5 g) dissolves in water (2 ml). Added 10% sodium hydroxide (2 ml) and carbonyl compounds (0.2 - 0.3 g), dissolved in alcohol (1 - 2 ml), the mixture is heated on a steam bath for 10 minutes, and then cooled in ice. Crystallization is induced by scratching the side of the test tube with a glass rod. Oxyms can crystallize from alcohol. ACIDS (i) Amides, anyylids and p-toluidida Acid (0.5 g) are refluxed with thionylchloride (2 - 3 ml) in a steam cupboard for about 30 minutes. The capacitor is removed and the excess of thionylchloride is distilled (b.p. 78). Thus, the acid chloride produced is treated with a concentrated solution of ammonia (5 ml) or aniline (0.5 - 1 ml) or p-toluydin (0.5 - 1 g), when the solid derivative is separated. He is going and recrystallised from alcohol adding decolourising charcoal if necessary. Alternatively, use PCl5 to form acid chloride. AMINES (i) Derivative acetyla (acetamide) reflux gently in a small dry flask under a dry capacitor of amin (1 g) with acetyrdic anhydride (3 ml) for 15 minutes. Cool the reaction mixture and pour 20 ml cold water. Boil to decompose excess vinegar anhydride. Cool and filter by suctioning an insoluble derivative. Recrystallise from ethanol. (ii) Benzoyl derivatives (benzamides) suspend 1 g of amin in 20 ml 5% of sodium aquier hydroxide into well-clogged flasks and add 2 ml of benzyl chloride (smoke hood!), about 0.5 ml at a time, with constant shaking. Shake vigorously for 5 to 10 minutes until the smell of benzoyl chloride disappears. Make sure the mixture stays alkaline. Filter from solid derivative, wash with a little cold water and recrystallise from ethanol. (iii) Benzolesulphonamide Up 1 g of amin in 20 ml 5% sodium hydroxide solution in well-clogged flasks add 1 ml benzenesulphone chloride (smoke hood!). Shake the mixture until the smell of sulfonide chloride disappears. Make sure the solution is alkaline. Acid if necessary to produce a beleaguered derivative. Concentrated salt acid added dropwise should be used. Filter the product, wash with a little cold water and suck dry. Recrystallise from ethanol. A return to chemistry, UWI-Mona, the main copyright home of © 2005 robert John Lancashire, all rights reserved. Created and maintained by Professor Robert Lancashire, Department of Chemistry, University of the West Indies, Mona Campus, Kingston 7, Jamaica. Established in March 1997. Links are verified and/or last changed on December 11, 2005. 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