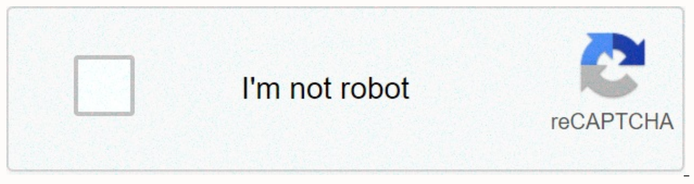

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Preparation of benzoin from benzaldehyde lab report

Introduction : Benzoin consists of hydroxyl ketone attached to two phenyl groups. It is prepared by the condensation of benzaldehyde, which is the simplest aromatic aldehyde, consisting of a benzene ring with a formidable group. Benzaldehyde is the main ingredient of bitter almond oil and has an almond odour. It can also be synthesized by chlorination and oxidation of toluene, and synthetic benzaldehyde is often used to taste almond extract. Benzaldehyde is converted by benzoin condensation into benzoin, which includes steel such as cyanide or tiamine chloride, and two aromatic aldehydes. The condensation product of benzoine is acyloin, which is a compound with hydroxy ketone. Symmetrical or asymmetric acyloin is produced, depending on whether both aldehyde are the same or not. The use of cyanide as a catalytic was first introduced in 1832 by Friedrich Wohler and Justus von Liebig and was used for the condensation of benzoine for more than 100 years. Then, in 1958, the ability to use tiamine ilide as a base catalyzer was introduced by Ronald Breslow. When using tiamine chloride as a catalyst, the steelophile attacks aldehyde, with a steelophilic supplement. Then carbonyl protonates water and deprotonates carbon. This leaves carbon negatively charged and attacks the next benzaldehyde. Then transfer the proton and secrete the tiamine catalyzer gives benzoin. In the case of cyanide as a catalyst, after the steel supplement, oxygen is deprotoned and cyanide reformed as carbonyl, with the nitrogen atom being negatively charged. Carbonyl then becomes steelophile, and attacks the next benzaldehyde. Tiamine pyrophosphate or vitamin B1 is an important vitamin that participates in many biological reactions. It acts as a coenzim, which are necessary to convert enzymes into its active form, and helps to enfixe the substrate to the enzyme. Thiamine also acts as basic catalysers when ilide thiazole ring is formed. Ilid is a coupling with a positive charge and a negative charge side by side, which stabilizes with resonance. Tiamine chloride is often used as a basic catalyst In the synthesis of complex organic molecules it is usually that it has at least a dozen individual transformations, with the product of one reaction then used as the starting material for the next reaction. You will have the opportunity to do multistep synthesis, starting with cheap, easily available benzaldehyde. The sequence you will try is first convert benzaldehyde into benzoin using vitamin, tiamine, as a catalytic. In the second step, benzoin is oxidised to benzyl using an oxidising agent. The third step is the condensed reaction of benzyl with dibenzil ketone (1,3-diphenyl-2-propaneon) for the production of tetraphenyllcicloctentadinon. The second step is to reduce benzyl to dihydrongouszoin by reducing sodium An additional fourth step is the possible conversion of tetraphenylcyclopenent-dienone into an alternative naphthalene via the Diels-Alder reaction (followed by decarbonilation) using microwaves as an energy source. One of the problems that becomes apparent is that the yield of the entire finished product will be limited by the lowest profitable individual reaction. Therefore, any reaction in sequence must be a high profitable reaction. Secondly, the total final return of the product is the product of each individual percentage return. If each step is a yield of 90% and is 10 steps, the total final return of the product is (0,90)10 or 35 %. For a reaction in 20 steps, the total return would be only 12 %. In a two-step reaction, if one step had a yield of 50 %, the maximum total return is 50 %. It is not necessary or desirable to use all your material at every turn. This reaction is a classic, conversion of two aldehyde molecules into alpha-hydroxy ketone. The reaction is known as benzoin condensation (condensation, because two molecules condensed into a single molecule). This reaction, which requires catalysing if it is often performed with cyanide ion. We're going to use tiamin as a catalyzer. It is sensitive to heat and can degrade if heated too much. Instead of this reaction at elevated temperatures for a few hours, we will allow the reaction to continue closer to room temperature for 24 hours or more. Benzaldehyde is easily oxidised to benzoic acid, which may interfere with the desired reaction by using freshly distilled benzaldehyde. The concentration of reaktivants and the temperature of the solution are crucial to

obtain a good return and procedures should be carefully followed. Too much water will force benzaldehyde from a solution that prevents an effective reaction. Too young water prevents tiamine chloride from dissolving. Some bases react with tiamine chloride to produce tiamine, which is an active catalysed. Procedure: Place 1.5 ml of 5M NaOH (CAUTION: extremely caustic) in a 10 ml Erlenmeyer flask and cool in an ice bath. Dissolve 0,80 g of ethylamine chloride (MW=337) in 50 ml of Erlenmeyer flasks in 2,5 ml of water. Add 7.5 ml of 95% ethanol to the tiamin and cool the solution for a few minutes in an ice bath. While keeping both flasks in an ice bath, add 1,5 ml of previously chilled 5M sodium hydroxide to the dropwise (3-5 minutes) in the thiamine solution by oscillation so that the solution remains below room temperature. Remove 50 ml of the flask from the ice bath, add 5,0 ml of benzaldehyde (d=1,044 g/ml) at the same time, rotating with the flask, mixing the benzaldehyde with a yellow, tuby, base layer. The solution becomes milky and then cleaned*. Seal the bottle with parafilm and place it in a drawer until the next laboratory period. *If the mixture does not go into the solution (e.g. if two layers are obvious), place the flask in a warm water bath at approximately 50 oC until the solution is cleaned or for a maximum of 10 minutes. you use hot water from oohs on the front of the laboratory. The mixture must become homogeneous in the water bath, but must not remain homogeneous when cooled. Next laboratory period: Filter the crystals, wash them without mother alcohol with 10-15 ml of cold 2:1 water mixture and 95% ethanol, and the air dries for strength for 15 minutes. Weigh your raw yield, break down all solid particles and recrystallize from a hot 95% ethanol (8 ml per gram). You don't have to filter the hot solution. After cooling, the recrystallised benzoin should be filtered, washed with at least a cold 2:1 water mixture and 95% ethanol and the air dried for 15 minutes or left until the next laboratory period. Get mp recrystallized benzoin (lit mp as 133 and 137 oC for d,l-benzoin, most students will see mp 133 oC). Once you are satisfied that you have the product you want, you can remove the first filtrate by neutralising the diluted HCl, and then rinsing the water layer down the drain with plenty of water. The second filtrate (from restalling) can be flushed into the drain with water. CAUTION: Concentrated nitric acid is extremely caustic and will burn exposed skin. Work in a hoodie! Place 2,0 g* of benzoine (weighed to the nearest tenths g) in 125 ml of erlenmeyer flask and carefully add 7 ml of concentrated chokic acid. Heat the mixture in a steam bath with an occasional slow turn for 30 minutes or until brownish-red nitrogen oxide gases develop. The fumes are toxic and toxic, so rest assured that the smoke-based protective shield is pulled down. Use tap water to cool the flask and contents carefully (the plug should be covered with a plastic seal or cork), then pour into 35 ml of cold water and rotate to harden the upsdded product. Collect the yellow solid using suing filtration and wash twice with 5 ml of cold water to remove some of the nitric acids present. Press the crystals to remove more water by placing another part of the filter paper over the crystals and pushing with a container or cork; the sousk should be supported and sat flat on the desktop. This raw product can be recrystallized from 95% ethanol while still slightly soaked (4 ml/g). Dissolve it in hot ethanol, add water to a drop to reach the point of the cloud, and allow it to crystallize slowly. Filter, dry, record the yield and take the MP. * Do not use all the benzoine you have synthesized. If you do not have more than 2.0 g, save 100 mg of benzoin for mp, IR, and to surrender and use the rest for this next step, change the procedure on the scale. If your instructor requests, to compare rf values, start tlc re-deposited product with known samples of benzoine and benzyl. Once you are satisfied that you have the product you want, you can dispose of the filtrate by first neutralising it with sodium carbonate, diluted with water and flushed down the drain. Ethanol from relapsing goes into a non-halogen waste container. is a relatively relentless compound that will dimerize even at low temperature. However, the corresponding tetraphenyl compounds are quite stable. Procedure: Place 0.7 g* of benzyl, 0.7 g of dibenzil ketone and 5 ml of absolute ethanol in 50 ml rbf. Attach the reflux capacitor and heat the mixture in a steam bath, water bath or sand bath until the solids have dissolved. It is critical to prevent water (moisture) from coming into contact with the reactioners. Raise the temperature to ensure slow reflux and add the 0,1 g potassium hydroxide (CAUSTIC) solution to 1 ml of absolute ethanol (this solution may be ready) to drop through the top of the capacitor. The reaction is very fast and there will be a purple color. After adding the base, allow the mixture to be re-dissased for 15 minutes while regularly lubrication of the flask. Cool the reaction flask to room temperature, then in an ice bath. Filter with the buchner suitcase, wash twice with 5 ml of cold 95% ethanol and dry the air for another hour. When the crystals are dried (which may last until the next laboratory period), weigh, record the yield and percentage return. Part of the purple product can be recited with a mixture of 1:1 95% ethanol and toluene (12 ml/0.5 g). Record mp; 219-220 oC). Once you are satisfied that you have the product you want, you can neutralize the filtrate with diluted HCl water and rinse down the drain. Recrystallization of the solvent should be placed in a container for non-halogen waste. *Do not use all the benzyl you have synthesized. If you don't have more than 0.7 g, save 100 mg of benzyl for mp, IR, and to surrender and use the rest for this next step, change the procedure for scale or get additional benzyl from your instructor. There are many different reductions in hydrides that convert carbonyl compounds into alcohols. One of the least reactive active substances is sodium borohydride. Although it reduces aldehides and ketone, it is fairly stable in single and alcoholic solutions. More reactive hydride-reducing agents can reduce other functional groups such as carboxylic acids, ethers, epoxides and nitrile. Such hydrides react violently with hydrogen gas-releasing water and should be handled with extreme care. You will reduce diketon, benzyl, by using sodium borohydride. In this reaction we can form three stereoizomers hydrobenzoin, RR, SS and RS, which is a meso isomer. Mezo is thesis is predominant. The stoichiomtry of a characteristic borohydride reaction is: \[\ce{4 R_2C=O + NaBH_4} \rightarrow \ce{(R_2CHO)_4B}^-\ce{Na+}\] hydrolysis of the borate ester \[\ce{(R_2CHO)_4B}^-\ce{Na+} + \ce{H_2O} \rightarrow \ce{4 R_2CHOH}\] Your start joint is diketon so you need 1 mmol borohydride for 2 mmol ketone . Procedure: Using 25 ml or 50 ml Erlenmeyer flask, dissolve 0,50 g of benzyl (weighs to the nearest hundredth of a gram) in 5 ml of warm 95% ethanol. Cool solution in a water bath that will produce a fine suspension of benzyl particles. Add 0,10 g of sodium borohydride (weight to the nearest hundredth g), which warms the solution and dissolves the suspended benzyl. As the reduction reaction continues over the next few minutes, the yellow colour of the benzyl will disappear. After a total of 10 minutes, add 5 ml of water, heat to boiling on a steam bath, filter or cateno, if the solution is not clear. After cooling the solution, dilute to the site of the saturated with as much as 10 ml of water and place the solution on the crystallisation page. In the discussion mention that three stereoisomers are possible and suggest why the mezo isomer prevails (lit mp 136-7 oC). Once you are satisfied that you have the product you want, you can discard the filtrate by diluting with water, neutralising excess, non-corrosion with acetic acid and rinsing into the drain. Place 39 mg tetraphenyllciclobatedienone (0,10 mmol), 3 drops of dymethyliacetylenecarboxylate (excess) and 0,3 ml of triethylene glycol in a 10 ml container. Mix the ingredients with a swing, cover the oven with a thin glass for an hour or a suitable microwave film (the thick watch often breaks in the microwave) and place it in the microwave. Set the oven at power level=6 5 minutes. After 5 minutes of radiation, the container will be hot. Leave to cool for a minute. The reaction mixture must be a golden colour that cools down to colourless crystals. It might take me a few hours to see the crystals. If necessary, allow the material to crystallize until the next class. Collect the crystals in a micro Hirsch's mint and wash with a few drops of cold 95% ethanol. Recistalize using 95% ethanol. Record mp product; 255-257oC. Contributions and distribution