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Borohydride reduction of a ketone lab report

Hydrogen-based reducing agents LiAlH₄ (Lithium Aluminium Hydride) and NaBH₄ (Sodium Borohydride) react with ketones and aldehydes to produce a 1st or 2nd Alcohol product. Both reagents were discovered by Schlesinger in the 1940s and are usually used in organic synthesis. NaBH₄ is a milder reducing agent of LiAlH₄ and can be used in protic solvents, such as ethanol. On the contrary, LiAlH₄ must always be used in aprotic solvents, such as tetrahydrofuran, and under extremely strict anhydrous conditions (free moisture). LiAlH₄ also requires a separate acid work up where reduction with NaBH₄ does not. For more information on these reagents see sections Ege 14.4 and 21.3 B.Schlesinger, H.I.; Brown, H.C. et. al. J. Am. Chem. Soc. 1952, 75, 186. Note the stoichiometry of the reaction. Each NaBH₄ molecule can reduce up to 4 carbonyl groups as it provides 4 hydrides per NaBH₄ molecule. Keep this in mind, try to draw the balanced equation for this reaction. Tip: One H on alcohol comes from an hydride and the other comes from the acid proton on CH₃CH₂OH (ethanol). Used techniques Beilstein test melting point recrystallization extraction and washing agent drying gravity & vacuum filtration thin layer chromatography and infrared spectroscopy. In part 1 it reduces 9-fluorenone using the procedure described below. Part 2 reduces an unknown ketone also using the method below. Your goal is to determine if the unknown ketone can be reduced by NaBH₄ to form an alcohol, compare the two reactions (part 1 and part 2), and to determine the identity of the unknown ketone. 9-fluorenone (part 1) Unknown Chetone (part 2) Ethanol NaBH₄ In a dram vial, dissolve 0.1 g of 9-fluorenone (part 1) or unknown aromatic ketone (part 2) in 1 mL of 95% ethanol, and cool the ice solution (most ketones will produce a fine suspension). Add to this solution or suspension 20 mg sodium borohydride (a great excess). If you have a suspension the suspended ketone will melt. The reaction mixture should warm up. After 15 minutes, add 1 mL of water, heat the solution to the boiling point, dilute the hot solution with hot water (1-2 mL) to the saturation point indicated by the cloud.* Collect the crystalline precipitates generated during the cooling of the mixture at room temperature using vacuum filtration. Recover the reduction product. With the guidance of your GSI determine an appropriate solvent for re-crystallization. Note Three of unknown aromatic ketones should produce liquid products after reduction. If your unknown product is a liquid the mixture will not become cloudy after addition of 2 mL of water. In this case, a microscale extraction is carried out to isolate the product. Add 4-5 mL of diethyl ether to the mixture and mix to allow the product to migrate into the organic layer. Transfer the ether layer into another dram vial and wash with an equal volume of brine (saturated solution of NaCl). Pipet out the layer of ether and wipe it overmagnesium sulphate. remove magnesium sulphate by gravity filtration and evaporate organic solvent by applying a nitrogen gas flow. determine the purity of the products in part 1 and 2, as well as the success of each reaction using tlc. collect the infrared spectrum of each product and the unknown ketone starting material. characterizing the starting materials and products of part 1 and 2 per m.p. uses beilstein test for halogens to exclude product ketone 1 (the only unknown with halogens) from table 2-1 below. use reaxsys or sdb to search for the ir spectra literature for the unknown ketone and its alcoholic product corresponding to comparison with the spectrum you get. Write your answer at the end of your notebook pages for this experiment: describe in your words what happened to your ketone as NaBH₄ is added. Why would a suspended solid dissolve during this passage? Why is 95 % ethanol used? Why not ethanol anhydrous? compares the behavior of unknown ketone with 9-fluorenone in the reduction reaction. How do the two reactions differ? how can structural differences in unknown ketone cause it to react differently from 9-fluorenone? How do you compare the infrared spectrum of 9-fluorenone with its alcoholic product? how does the spectrum of your unknown alcohol product compare with that of the unknown ketone starting material? table 2-1 1 4'-Chloroacetophenone mp 20°C bp 232°C 2 4'-Methoxyacetophenone mp 36-38°C 3 9-Fluorenone mp 82-85°C 4 benzoin mp5 Benzophenone mp 48-49°C 6 Propiophene bp 218°C 7 Methyl 2-naphthyl ketone mp 53-55°C 8 4'-Hydroxypropiophenone mp 147.5-148.5°C Thank you for the interesting in our services. We are a non-profit group running this website to share documents. We need your help to maintain this site. To keep our site running, we need your help to cover our server cost (about \$400/m.) a small donation will help us a lot. 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