

Organic chemistry ii laboratory manual assignment



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Cool the content of the flask with cold water, and then pour into ice water (120 ml) and stir vigorously to assist the hydrolysis of unreacted acetic anhydride.

6. 7. 8. 9. After 30 min, the oil which first separates will gradually solidify. *

Collect the crude product using Hirsch funnel. Record the melting point.

Dissolve the crude product in minimal amount of hot methanol. Once the crude product is dissolved, let it cool down and pour water to rationalize it.

10. Filter out the rationalized product. 11. Record the melting point. (the literature value is 1. In this experiment, a α -C)-glucoside is obtained.

If we use c) Question (Mice)20/McCann to react with powdered C)-glucose, - C)-glucoside will be resulted. Explain. ?? END?? Page 2 of 8 Expect. 2.

CICS-I anhydride (Dills-alder reaction) The Dills-Alder reaction is an important, synthetically useful reaction in organic chemistry. It is named after Otto Dills and his student Kurt Alder who were awarded the Nobel Prize in chemistry in 1950 in recognition of the importance of their discovery. The Dills-Alder reaction is the syllabication of a 1,3-diene system (a diene) with a dienophile that usually bears an electrocyclic group (a dienophile) to produce a six-member ring.

The reaction is concerted (bond breaking and forming occur simultaneously) and therefore results in high stereoselectivity (products with predictable stereochemistry). In this experiment, you will perform a Dills-Alder reaction using 2,3-dimethyl-1,3-butadiene as the diene and maleic anhydride as the dienophile. These substrates react to form the Dills-Alder product, CICS?? I OZ, 6-tetrahydro-4,5-dihydro-2H-pyridin-2-one. After synthesizing your anhydride, you will need to purify it by rationalizations, and characterize it by obtaining a melting point and an IR spectrum Page 3 of 8 Procedure 1 .

Weigh 0.72 g (0.74 mol) of finely powdered malefic anhydride contained in a small conical flask. Add 0.65 g (0.0080 mol) of freshly distilled 2,3-timetable-1,3-dine (this will be provided by lab. Technician) to (addition should be performed in fume cupboard). Wrap the conical flask and tilt the flask to ensure malefic anhydride is immersed into the dine solution.

Reaction occurs in a few minutes (indicated by evolution of heat). Allow to stand until the mixture attains room temperature. Add ?? ml acetone to dissolve the mixture. Add ?? ml water to precipitate out the product. Wash with ml water for two times.

Wash with ml petroleum ether (b. P. 60-80 co) for two times. 10. Air dry the product upon filter paper. 11. Record the yield and its melting point. (the literature value is co) 12. Obtain an IR spectrum of your product and identify the characteristic signals of the compound. Question 1. Is the adduct of the reaction in end or ex. form? Explain. 2. If some of the product is hydrolysis to the corresponding carboxylic acid, how to identify the presence of the acid in the product? Page 4 of 8 Expect. 3. Photochemical Reaction and Acid-catalyzed Rearrangement: Preparation of Pensacola and Opinionatedly

Procedures: Part A: 1. Dissolve phenomenon (3 g) with 'so-proposal (20 ml) in a 50-ml Erlenmeyer flask by warming on the steam bath. Add more 'so-proposal (?? 10 ml) and acetic acid (half drop) to the flask; transfer the solution to a given-test tube; wash the Erlenmeyer flask with 'so-proposal and transfer to fill up the test tube, stopper the test tube with a well-rolled tight fitting cork ensuring there is no air bubble trapped in the tube. Expose the solution to direct UP light for 1 week to complete the reaction (hand in your reaction mixture to our Lab Technician for this UP activated reaction).

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Since Pensacola is sparingly soluble in ' so-proposal, it will crystallize out once is formed. Part S: 1 . Chill the flask and collect the crystals by a Hirsch funnel. 2. Dry the crystals and record the yield together with its m. P. (the literature value 3. Add Pensacola (1 g) (from part A), acetic acid (5 ml) and one small crystal of iodine ((about 0. 01 g) too pear-shaped flask fitted with a condenser. (In case your yield is less than 1 g, please add in the other reagent in correct proportion. Reflux the solution for 1-2 minutes until the crystals dissolve and reflux for further 5 ins.

Cool the solution, the pinochle separate as paste; filter the paste and wash it free from iodine by 95% ethanol (about 1-2 ml). Rationalize the crystals from a suitable solvent (made a suggestion and consult your demonstrator; hint: like dissolve like and record the amount of solvent used in your report)

Record the yield and m. P. Of the crystals. (the literature value is 1 . Write the feasible mechanism for the formation of Pensacola and Opinionatedly ??

END?? Page 5 of 8 Expect. 4. Michael Addition: Preparation of 1-Phenyl-3-phenylaminopyrrolidine-2, 5-iodine. Part A Preparation of Millennial acid (A) 1

Stir finely powdered malefic anhydride (g, 30 mol) and aniline (g, 32 mol) in glacial acetic acid (1 5 ml) in conical flask for 30 min. At room temperature.