

Competing Nucleophiles via Tosylate Intermediate

Nucleophilic Substitution
Heating under Reflux
Extraction
Gas Chromatography
NMR Spectroscopy
2-Step Synthesis

Required Reading

New: Page 194, Exp. 19

Review: Technique 15 (Gas Chromatography)

Special Instructions

Sodium Hydride is highly volatile and must be stored in a tightly closed container. When handling sodium hydride, gloves should be worn and caution should be taken to prevent inhalation. The preparation of a pentyl tosylate reaction should be carried out in a well-ventilated room or in a fume hood because the formation of HCl(g) will be emitted during reaction.

Notes to the Instructor

A tosylate prepared from 2-pentanol and 3-pentanol can also be prepared with similar results, but a tertiary alcohol will not form a tosylate using these methods.

Suggested Waste Disposal

Dispose of all aqueous waste in a container labeled for aqueous waste, and all organic waste in a container labeled for organic non-halogenated waste. The organic waste from the nucleophilic substitution can be included in the waste labeled for experiment 18. Unreacted NaH should be placed in a separate waste container for neutralization.

Procedure

Preparation of a tosylate:

Add 1 mL hexane, 0.1g sodium hydride, and a stir bar to a 5-mL conical vial. Stir for 2 minutes, and then remove the conical vial from the magnetic stirrer. Once the mixture has settled, decant the hexane solution using a Pasteur pipette. Repeat this two more times with fresh 1 mL portions of hexane. A small amount of hexane residue with the washed sodium hydride is acceptable.

Add 2-mL of ether and slowly add 0.18g 1-pentanol to the conical vial, letting H₂ (g) evolve. Leaving the vial uncapped, let the mixture stir for 30 minutes until all the hydrogen gas has evolved off. While stirring, add 0.39g tosyl chloride in small increments to the mixture over 10 minutes. If the mixture stops stirring because too much ether evaporates, add a 1 mL portion of ether. Once all the tosyl chloride has been added, let the mixture stir uncapped for another 60 minutes, adding another 1 mL of ether if necessary.

Let 3 mL DI water, 3 mL saturated sodium bicarbonate solution, and 3 mL 2M sulfuric acid solution chill on ice. After the reaction has stirred for 60 minutes, remove the spin vane from the conical vial. Slowly add 1 mL chilled DI water, cap the vial and gently mix for 1 minute. Let the phases separate, and then remove the lower aqueous layer using a Pasteur pipette. Repeat the extraction twice more with fresh 1-mL portions of chilled DI water. In a similar manner, extract three times with 1 mL portions of chilled 2M sulfuric acid solution, then three times with 1 mL portions of chilled saturated sodium bicarbonate solution. Add sodium sulfate, cap the conical vial and let sit.

Competing Nucleophilic reaction:

To a clean dry 5-mL conical vial equipped with a spin vane, add 0.2 g of the prepared pentyl tosylate, 0.172 g sodium bromide, and 0.314 g sodium iodide. Dissolve in 4 mL methanol, cap the vial, and allow the solution to stir for 2 hours.

Perform Experiment 18 during this time.

After the reaction has stirred for 2 hours, transfer the solution to a 15-mL centrifuge tube, add 5 mL DI water, 1 mL ether, then cap the tube and gently mix for one minute. Using a Pasteur pipette draw off the lower aqueous layer, and isolate the ether layer.

Spectroscopy.

Submit your sample for Gas Chromatography, according to your instructor's directions.

Report

Attach the Gas Chromatogram to your report, label the major peaks and determine the relative areas of the relevant peaks. And do whatever else Charlie tells you. He's the man.