Sodium Borohydride Reduction of 9-fluorenone

**Required Pre-Lab Readings:** McMurry, Sect. 13.3

**Techniques you must be prepared to perform:** Microscale reaction,vacuum filtration, melting point.

**Introduction**

Reduction reaction in chemistry refers to the gain of electrons. In organic chemistry, reduction reactions usually happen through the gain of hydrogens, or the loss of oxygen atoms, or both. Carbonyl compounds such as aldehydes, ketones, carboxylic acid and acid derivatives can be reduced to alcohols when its carbonyl group gains a hydride (H-) and a proton.

There are two widely used reducing reagents which can be considered as hydride (H-) donors: sodium borohydride (NaBH4) and lithium aluminum hydride (LiAlH4). Lithium aluminum hydride is a powerful and broad spectrum reducing reagent and its reduction of carbonyl compounds must be carried out under anhydrous conditions using aprotic solvents such as ether. On the other hand, sodium borohydride is a milder and therefore safer reagent than lithium aluminum hydride. It can be used in solution in alcohols with the presence of base such as NaOCH3 or NaOH. NaBH4 is not stable under acidic conditions and decomposes slowly under neutral conditions.

**Procedure**

A stock reducing reagent solution has been freshly prepared. Every 1.0 mL of reducing solution consists of 20 mg sodium ethoxide and 40 mg sodium borohydride dissolved in ethanol.

Dissolve 0.25 g 9-fluorenone (\_\_\_\_mmol) in 1.5 mL of warm ethanol (high temperatures may decompose the reducing agent) in a 20 mL screw top scintillation vial. To this solution is added *drop-wise* 1.0 mL of the reducing reagent (record any observations: heat evolution, gas evolution, color change, etc.). Swirl the reaction vial periodically while the reaction continues for 10 - 20 min. Use TLC (2:1 hexane:ethyl acetate) to monitor your reaction. After the reaction is complete, the product is precipitated by the cautious addition of 2.5 mL of water. A color change is observed as the reaction proceeds from the yellow 9-fluorenone to the white 9-fluorenol. The reaction mixture is then acidified with 0.5 M HCI to pH 6 or below to decompose the excess sodium borohydride. The crude product is vacuum filtered, and washed with cold water to remove any residual inorganic salt formed by the excess of the sodium borohydride and hydrochloric acid. Allow the product to air dry until next week when its weight and mp can be obtained.

Sodium Borohydride Reduction of 9-fluorenone

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| **Name:** |  | **Section:** |  |

Overall Reaction: (chemical drawing software)

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| mass of 9-fluorenone: |  |  | theo mmol 9-fluorenol: |  |
|  |  |  |  |  |
| mmol of 9-fluorenone: |  |  | theo mass 9-fluorenol: |  |
|  |  |  |  |  |
| Total volume of  |  |  | mass recovered 9-fluorenol: |  |
| reducing solution: |  |  |  |  |
|  |  |  | mmol recovered 9-fluorenol: |  |
| mass of NaOCH2CH3: |  |  |  |  |
|  |  |  | % yield of 9-fluorenol: |  |
| mmol of NaOCH2CH3: |  |  |  |  |
|  |  |  | TLC |  |
| mass of NaBH4: |  |  |  | Product Rf: |  |
|  |  |  |  |  |  |
| mmol of NaBH4: |  |  |  | Solvent: |  |
|  |  |  |  |  |  |
|  | Observed melting point of 9-fluorenol: |  |
|  |  |  |  |  |  |
|  | Literature melting point of 9-fluorenol: |  |

Literature source for melting point:

Show complete calculations: (notebook)

**Post Lab Questions:**

1. Show the electron pushing mechanism for this reaction.

2. Complete the reduction reactions below. Use chemical drawing software for your answers and electronically paste them into the boxes.

|  |  |  |
| --- | --- | --- |
| 1) |  |  |
|  |  |  |
| 2) |  |  |
|  |  |  |
| 3) |  |  |
|  |  |  |
| 4) | Hint: reductive amination |  |