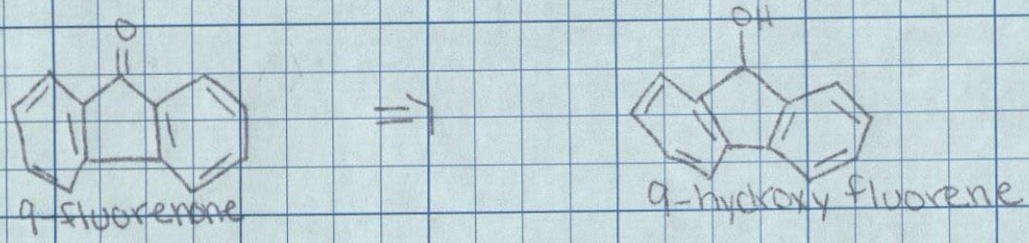
Name: Erin Thibodeau Date: ~~1-30-18~~ 1-23-18 63/65 03

Experiment: $NaBH_4$ Reduction of 9-Fluorenone 97%

Objective:

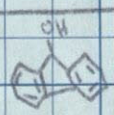
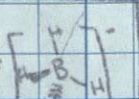
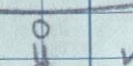
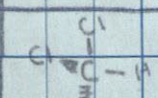
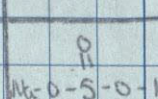
In this lab a metal hydride, sodium borohydride, was used as a reducing agent. The sodium borohydride was used to reduce the ketone 9-fluorenone in order to make an alcohol product, 9-fluorenol. ET How will the product be characterized?

Chemical Equations:



9-fluorenone ⇒ 9-hydroxy fluorene

Physical Properties:

Compound name	Structure	CAS#	MWt	State & Color	BP or MP	RI (Liquids only)
9-Fluorenone	 ✓	486-25-9	180.21 g/mol	solid, yellow	BP = 342°C MP = 83.5°C ✓	~~1.6369~~ ET 1-23
9-hydroxy fluorene	 ✓	1689-64-1	182.22 g/mol	solid, white	~~BP=~~ ET MP = 153-154°C ✓	~~1.681~~ ET 1-23
Sodium Borohydride	 ✓	16940-66-2	37.84 g/mol	solid, grayish white	MP = 400°C	~~1.542~~ ET 1-23
Acetone	 ✓	67-64-1	58.08 g/mol	liquid, clear	BP = 56.2°C MP = -95.35°C	1.3586
Dichloromethane	 ✓	75-09-2	84.93 g/mol	liq. d, clear	BP = 39.75°C MP = -96.7°C	1.4244
Hexane	 ✓	~~110~~ ET 110-54-3	86.18 g/mol	liquid, clear	BP = 68°C MP = -95°C	1.3749
1M HCl	 ✓	7647-01-0	36.46 g/mol	liquid, clear	BP = 110°C MP = -74°C	1.254
Anhydrous Na_2SO_4	 ✓	7757-82-6	142.06 g/mol	solid, white	BP = 1100°C MP = 888°C	~~1.468~~ ET 1-23

Important: *Place card under blue copy.* Erin Thibodeau 1-23-18

Name: Erin Thibodeau Date: 1-23-18

Experiment: $NaBH_4$ Reduction of 9-Fluorenone

Compound name	Structure	CAS#	MWt.	BP or MP	St Co
Silica Gel (SiO_2)	O=Si=O	7631-86-9	60.09 g/mol	MP = 1610°C	Soli wh

Compound name	Hazards
9-Fluorenone	irritant to skin and eyes, hazardus if ingested
9-hydroxy fluorene	irritant to eyes and skin. Can be irrita or digestive tract if inhaled or inges
Sodium Borohydride	corrosive and extreme irritant to c cause burning, sneezing, and coughi
Acetone	Slight irritant to skin, permea eyes, and if ingested or inhaled
Dichloromethane	Very hazardous in case of eye conta as irritant. Irritant and permeat carcinogenic effects
Hexane	Permeator in case of skin contact, inge Slight irritant to skin and eyes
HCl	Extream irritant to skin and eyes c or inhalation. Corrosive to eyes. Corros of skin.
Anhydrous Na_2SO_4	Irritant to eyes. Slight irritant in co contact, ingestion, or inhalation
Silica gel	Slight irritant in case of skin or eye contac inhalation

References:

www.sciencelab.com/msds/

fscimage.fishersci.com/msds/26347.htm

Void

1/2

Erin Thibodeau

1-23-18

Important: Place card under blue copy.

me: Erin Thibodeau Date: 1-24-18

periment: $NaBH_4$ Reduction of 9-Fluorenone

rocedure:

0.050g of 9-Fluorenone ~~was~~ were weighed in a tared 5mL conical vial. A magnetic spin vane ~~and~~ was added to the vial and an air condenser was attached. ~~1 mL of~~ The vial was placed in a aluminum block on a stir plate and 1 mL of 100% ethanol was added to dissolve. Once the solid was dissolved 3 drops of $NaBH_4$ ~~was~~ were added. While waiting 10 minutes ~~the TLC plate and~~ a TLC plate was prepared and chamber ~~was~~ were prepared. Once the solution became ~~clear~~ clear about 1.5mL 1M HCl was added. ~~and it became clear~~ The pH was tested and found to still be basic so more HCl was added until ~~the solution was~~ the solution was acidic. Then 1mL of methylene chloride was added and a small amount of solid remained. The solution was stirred on the stir plate to dissolve the solid. Once dissolved the top layer became acidic so more HCl was added. Then the bottom layer was removed with a pasture pipet and put into a clean dry beaker. This process was repeated twice with .5 mL of methylene chloride. Once removed, a spatula full of anhydrous sodium sulfate was added to the beaker to dry the extract. It was then left to stir for about 5 min. A 100 mL beaker was weighed and found to be 48.182 g. Once dried the solution was poured into the weighed beaker. The sodium sulfate was rinsed twice with methylene chloride. The product was dried with a hair dryer until it reached a constant mass of 48.202g. This value results in product weighing ~~0.020g~~ 0.025g. The product was tested for melting point in a melting point apparatus. The apparatus ~~the~~ range was found to be from ~~.~~ The results of the TLC are attached below.

TLC STRIP

rtant: Place card under blue copy.

Erin Thibodeau 1-24-18

1/24/18 OBoler

Name: Erin Tribodeau Date: 1-24-18

Experiment: NaBH4 Reduction of 9-Fluorenone

Observations:

The solid 9-Fluorenone dissolved in the ethanol fairly quickly. The yellow solution did not turn clear in the 10 minutes described in the procedure, a large scoop of solid NaBH4 had to be added to quicken the process. Much more HCl than 1.5 mL was required to turn the solution acidic. Adding the HCl made lots of bubbles, and formed a white precipitate. Adding methylene chloride immediately dissolved the white solid.

Great Observations!

Results:

Product	Yield exp.	Yield theor	% Yield	MP exp	MP lit	MP %Error
9-fluorenol	0.020g	0.051g	40.%	148°C-159°C	153°C-154°C	0%

Sample	RF value
9-fluorenone	0.611
9-fluorenol	0.462
reaction mixture	0.432

RF calculations:

pure 9-fluorenone = 4.84cm

pure 9-fluorenol = 3.66cm

reaction mix = 3.42cm

dye front = 7.92cm

$R_{f_1} = \frac{4.84}{7.92} = 0.611$

$R_{f_2} = \frac{3.66}{7.92} = 0.462$

$R_{f_3} = \frac{3.42}{7.92} = 0.432$

Theoretical Yield calculation:

$$0.050g \text{ 9-fluorenone} \times \frac{182.22 \text{ g/mol 9-fluorenol}}{180.21 \text{ g/mol 9-fluorenone}}$$

$= 0.050557768279$

$= 0.051g$ 9-fluorenol

Percent Yield calculation:

$$\frac{0.020g}{0.051g} \times 100 = 39.558775\overline{11}\%$$

Melting Point & Percent Error calculation:

$(148+159) \div 2 = 153.5$

$(153+154) \div 2 = 153.5$

$\%e = \left(\frac{153.5}{153.5}\right) \times 100 = 0.0\%$

VOID

VOID

Important: Place card under blue copy.

Erin Tribodeau 1-24-18