

# How to Write a Protocol and Lab Report

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# The Report

- 4 pages A4 (available on Moodle)
- You must only complete the document with **PEN** (H and P phrases may be typed)
- 1<sup>st</sup> and 2<sup>nd</sup> pages: Must be completed **BEFORE** you begin the experiment: mechanism, quantities and H and P phrases
- 3<sup>rd</sup> page : Introduction, experimental procedure **AND** observations
- 4<sup>th</sup> page : Results, analysis and conclusion; any comments by assistants

**Balanced Equation:** Can be written ( $C_2H_5O$ ) or drawn, but you must also show the mechanism of reaction.

**Name the reaction mechanism**

**DO NOT FORGET YOUR NAME**

Organic Chemistry for Materials Scientists (MSE 211)

- Lab Course -

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**EPFL**

Signature(s):

Date:

Assistants:

Name, surname:

**Synthesis and Purification of** \_\_\_\_\_

**Reaction Equation + Mechanism** (including electron arrows)

**Name of the Reaction Mechanism:**

## Chemical products

**Molar mass:** you can search on the internet or calculate them

**Moles, masses, volumes :** calculate the amount required

**Security symbols:** corrosive, toxic, dangerous for the environment, etc.

**Phrases H and P:** search the compound, find an SDS (FDS in French) from a supplier. Write ALL OF THEM.

This site lists all the phrases: [https://www.oc-praktikum.de/nop/en/articles/pdf/HPPhrases\\_en.pdf](https://www.oc-praktikum.de/nop/en/articles/pdf/HPPhrases_en.pdf)

### Quantities and Safety

| Compound Name | Molar Mass<br>(g.mol <sup>-1</sup> ) | Mass/Volume,<br>(g, mL) | Hazardous<br>Symbol | Phrases H/P (n°) |
|---------------|--------------------------------------|-------------------------|---------------------|------------------|
|               |                                      |                         |                     |                  |
|               |                                      |                         |                     |                  |
|               |                                      |                         |                     |                  |
|               |                                      |                         |                     |                  |
|               |                                      |                         |                     |                  |
|               |                                      |                         |                     |                  |
| Phrases H:    |                                      |                         | Phrases P:          |                  |
|               |                                      |                         |                     |                  |

Introduction: Purpose of the experiment, mention the type of experiment perhaps

# Do not just copy the protocol !!

## What to find:

- Which glassware is used for a reaction (main container, volume)
- Which reagents are used
- In which order the reagents are added
- At what is the temperature of the reaction
- Stirring?
- The reaction time (not necessarily the one indicated in the protocol)
- How does one end the reaction (quenching)?
- Describe the filtration steps and wash / extractions needed (workup) (if applicable)
- Purification of the final product
- Performance of the reaction (color change, gas generated?)
- Appearance and color of the product

## Introduction

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### Experimental Procedure and Observations

[illegible]

- Complete the results table
- Analysis
  - Melting point
  - NMR (list signals, coupling constants, and assign peaks)
  - Mass Spectrometry (identify fragments)
  - UV-Vis (lambda max, extinction coefficient)
  - Answer Questions from the protocol here.
- Conclude how the experiment went (purity of product, yield, major issues during synthesis, type of reaction)

## Results

| <u>Theor. Mass</u> (g) | Actual Mass (g) | Yield (%) | Melting Point (°C) |
|------------------------|-----------------|-----------|--------------------|
|                        |                 |           |                    |

**Analysis** (Melting Point, NMR, Mass Spectrometry, UV/vis spectrum)

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## Conclusion

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Don't forget your names!

Balanced Equation, with the mechanism!

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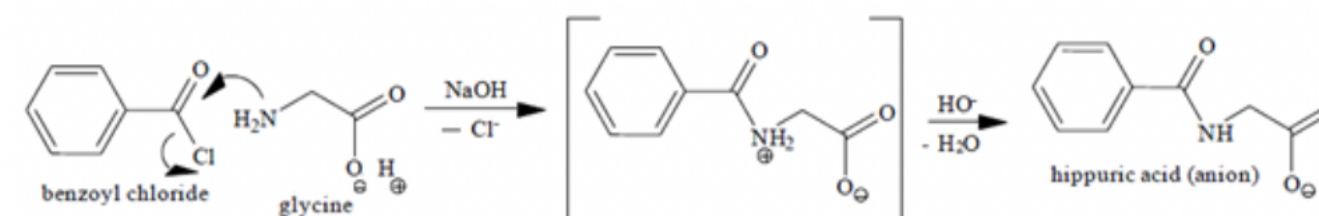
EPFL

|                                               |
|-----------------------------------------------|
| Date:                                         |
| Assistants: (names of assistants in your lab) |
| Name, surname: DUPONT, Jean-Pierre            |
| Name, surname: MARTIN, Olivier                |
| N°: (reaction number)                         |

Signature(s):

## Synthesis and Purification of hippuric acid

Reaction Equation + Mechanism (including electron arrows)



Strong base (NaOH) picks up the proton from the  $\text{NH}_2^+$ , water is released.

Name of the Reaction Mechanism: Addition-elimination, amidation

## Masses, volumes, and mole quantities

Remember to look up the SDS!

### Quantities and Safety

| Compound Name            | Molar Mass (g.mol <sup>-1</sup> ) | Mass/Volume, (g, mL)  | Hazardous Symbol    | Phrases H/P (n°)                                                                                                             |
|--------------------------|-----------------------------------|-----------------------|---------------------|------------------------------------------------------------------------------------------------------------------------------|
| <u>Benzoyl chloride</u>  | 140.57                            | 33mmol, 4.6 g, 3.8 mL | Toxic and corrosive | H: 302, 312, 314, 317, 331 P: 280, 301 + 312 + 330, 301 + 330 + 331, 303 + 361 + 353, 304 + 340 + 311, 305 + 351 + 338 + 310 |
| <u>Glycine</u>           | 75.07                             | 33mmol, 2.5g          |                     | NONE                                                                                                                         |
| <u>Sodium hydroxide</u>  | 40.0                              | Excess, 25 mL (10%)   | Corrosive           | H: 290, 314; P: 260, 280, 301 + 330 + 331, 303 + 361 + 353, 305 + 351 + 338                                                  |
| <u>Hydrochloric acid</u> | 36.5                              | Drops (2M)            | Corrosive           | H: 290, 314, 315, 319, 335                                                                                                   |

#### Phrases H:

290: May be corrosive to metals.  
 302 + 312: harmful if swallowed or in contact with skin.  
 314: Causes severe skin burns and eye damage.  
 315: Causes skin irritation.  
 317: May cause an allergic skin reaction.  
 319: Causes serious eye irritation.  
 331: Toxic if inhaled.  
 335: May cause respiratory irritation.

#### Phrases P:

260: Do not breathe dust or mist.  
 280: Wear protective gloves/ protective clothing/ eye protection/ face protection.  
 301 + 312 + 330: IF SWALLOWED: Call a POISON CENTER/doctor if you feel unwell. Rinse mouth.  
 301 + 330 + 331: IF SWALLOWED: Rinse mouth. Do NOT induce vomiting.  
 303 + 361 + 353: IF ON SKIN (or hair): Take off immediately all contaminated clothing. Rinse skin with water.  
 304 + 340 + 311: IF INHALED: Remove person to fresh air and keep comfortable for breathing. Call a POISON CENTER/doctor.  
 305 + 351 + 338 + 310: IF IN EYES: Rinse cautiously with water for several minutes. Remove contact lenses, if present and easy to do. Continue rinsing. Immediately call a POISON CENTER/doctor.  
 305 + 351 + 338: IF IN EYES: Rinse cautiously with water for several minutes. Remove contact lenses, if present and easy to do. Continue rinsing.

Always write in the passive, and past tense.

You are describing what has been done in the general sense.

Mention how you analyzed products (TLC, Melting point, etc)

## Introduction

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The purpose of this laboratory experiment is to synthesize hippuric acid by an amidation reaction. This reaction takes place between benzoyl chloride and glycine in alkaline medium. The product precipitates from the reaction mixture and is then purified by recrystallization from water. Analysis of the melting point allows determination of the relative purity of hippuric acid.

## Experimental Procedure and Observations

In a three necked round bottom flask equipped with a thermometer and condenser, glycine (2.5 g, 33 mmol) was dissolved portion wise in 10% NaOH (25 mL) under vigorous stirring. After complete dissolution of glycine, benzoyl chloride (3.8 mL, 33 mmol) was added dropwise via a dropping funnel. The progress of the reaction was followed by temperature evolution. **After complete addition, the temperature increased rapidly to 45°C and then decreased slowly to room temperature.** Pieces of ice were added to quench the reaction. Then, the pH of the solution was adjusted to pH = 1-2 (pH paper test) by addition of hydrochloric acid (2 M). **A white precipitate formed** that was filtered on a Büchner funnel and finally washed with cold water. The crude product (7.4 g, R = 116 %) was purified by recrystallization from water (100mL). After filtration on a Büchner funnel and drying in an oven (100°C), the melting range of the as-obtained colourless crystals (4.3 g, R = 82 %) was determined (MP = 186-188 °C).

## Results, Analysis (and questions), and conclusions

Remember to ask questions,  
We are here to help!

### Results

| <u>Theor. Mass</u> (g) | Actual Mass (g) | Yield (%) | Melting Point (°C) |
|------------------------|-----------------|-----------|--------------------|
| 5.9 g                  | 4.3 g           | 73 %      | 186-188 °C         |

### Analysis (Melting Point, NMR, Mass Spectrometry, UV/vis spectrum)

The melting point of the final product was determined by measuring three samples. The melting range was 186-188 °C (literature: 187-188 C°), thus the target compound being of sufficient purity. The NMR showed peaks at XX and YY ppm, which corresponded well to the expected ranges for these protons.

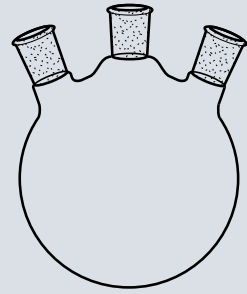
### Conclusion

Hippuric acid was successfully synthesized by an amidation reaction of benzoyl chloride with glycine. The pure target was obtained by recrystallization in good yield of 73%. The decent purity was confirmed by melting point measurement (186-188 °C) and <sup>1</sup>H-NMR analysis.

# Verrerie/Glassware

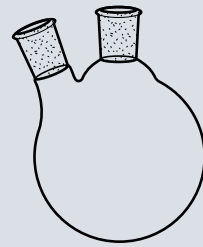
## Round bottom flasks

Ballons à fond rond



Three neck round bottom flask

Tricol



Two neck round bottom flask

Bicol



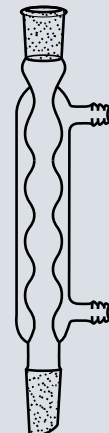
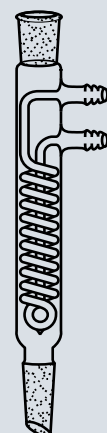
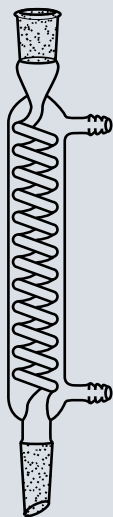
Single neck round bottom flask

Ballon



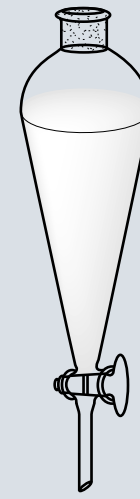
## Condensers

Réfrigérant



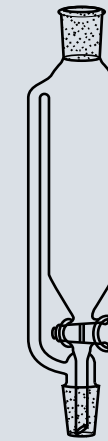
Drysin

## Funnels



Separating funnel

Ampoule à décanter



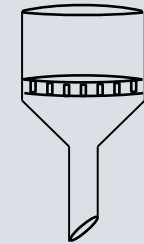
Dropping funnel

Ampoule à addition isobare



Funnel

Entonnoir



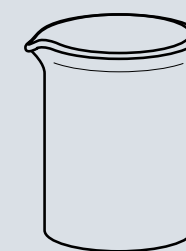
Büchner funnel

Büchner



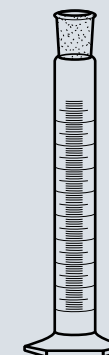
Frit

Fritté



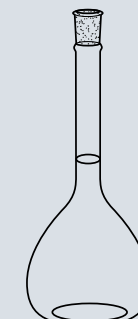
Beaker

Bécher



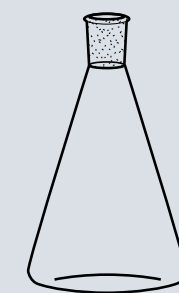
Graduated column

Eprouvette ou colonne graduée



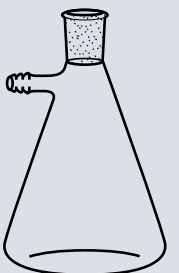
Volumetric flask

Fiole jaugée



Erlenmeyer

Erlenmeyer



Filtering flask

Fiole à vide

- Have your lab coat (or have arranged with your class rep to receive it when you arrive the first day)
- Arrive PROMPTLY at 8:00 to the lab, with the prelab completed
- Wear long pants and close-toed shoes
- Be aware of the hazards you are dealing with each day
- There are lockers, but you must bring your own lock if you want to use them