Monostep (or Multistep) synthesis
Protocol N°

I- Introduction

- Scheme of the reaction (produced with ChemDraw in ACS style) with appropriate title. In case of multistep synthesis, the different steps are presented in the same scheme (as in the manuscript that you received at the beginning of the practical session)
- Brief explanation about the experiment and references from the literature. Only the type of reaction is cited here (nucleophilic substitution, elimination, cycloaddition, electrophilic addition, etc). The mechanism is discussed in another section of the report.
  Example: The electrophilic addition of double bonds to strong acids of type HX is a classical reaction in organic synthesis to generate halogenated compounds from olefins. It follows the Markovnikov's rule affording the more substituted halogenated isomer.

II- Mechanisms

Reaction mechanisms are represented with drawing softwares such as ChemDraw (ACS style). Reactivity arrows represent the moving of electrons (from nucleophile to electrophile). You should check that charges are balanced within species at each step of the mechanism. When a catalyst is used, it should be regenerated in the reaction mechanism. Full arrows (→) stand for normal reactivity and half arrows stand for radical transformations. The correct bond angles and geometries should be represented.

III- Experimental Part

III-1. Table of reactants.

Compound	FW (g/mol)	Mass (g)	Volume (mL)	n (mmol)	Equivalents (eq)	Density (g/mL)	Safety (symbols)
Starting material							
Reagent 1							
Reagent 2							
Catalyst							
Solvent							

III-2. Procedure

Example

To a solution of vanillin (50.33 mmol, 1 eq., 7.66 g) in acetic acid (60 mL) was added hydroxylamine hydrochloride (75.5 mmol, 1.5 eq., 5.25 g). The solution was stirred at reflux for 90 min. The reaction was monitored by TLC PET/AcOEt 1:1 R_f: 0.67. Then the reaction mixture was cooled to room temperature and diluted with Et_2O (300 mL). The solution was washed with water (300 mL) and brine (3x100 mL). The organic layer was dried with $MgSO_4$ and concentrated under reduced pressure. The crude product was purified by flash column chromatography (petroleum ether/AcOEt 3:1) to afford 4-hydroxy-3-methoxybenzonitrile as a slightly yellow solid (49.4 mmol, 7.37 g, 98 % yield).

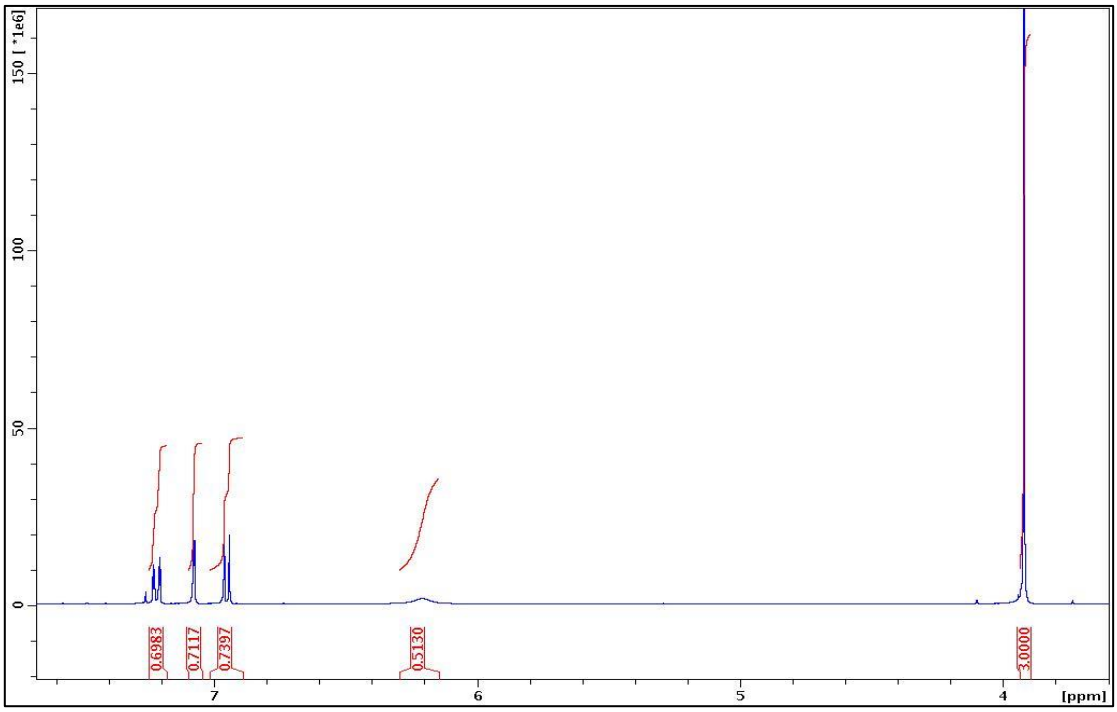
III-3. Characterization / purity

- Give the analytical method that was performed to assess purity and confirm structure
- Report the corresponding data

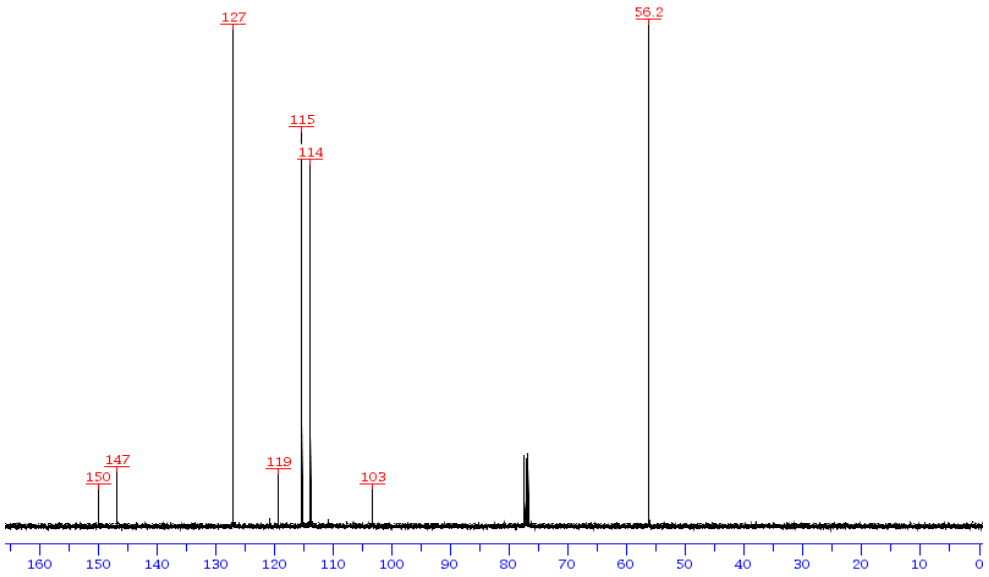
Example

^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.21 (dd, 1H, *J*= 8.2, 1.8 Hz, **H**3), 7.07 (d, 1H, J= 1.8 Hz, **H**1), 6.95 (d, 1H, J= 8.2 Hz, **H**2), 6.20 (s, 1H, **H**O), 3.92 (s, 3H, C**H**$_3$-O). **^{13}C-NMR (101 MHz, $CDCl_3$)** δ 150.0 (C_{ar}), 146.7 (C_{ar}), 127.0 (CH_{ar}), 119.3 (C), 115.3 (CH_{ar}), 113.8 (CH_{ar}), 103.3 (C_{ar}), 56.3 (CH_3). **Elemental Analysis** for $C_8H_7NO_2$ (%): C 64.42, H 4.73, N 9.39; found: C 64.03, N 9.26, H 4.59. **IR :** 3360, 2940, 2225, 1590, 1510, 1280, 1030, 795 cm^{-1}.

Copies of spectra can be included here or in annex.
^{1}H-NMR spectrum of **36**



^{13}C-NMR spectrum of **36**



IV- Discussion

This section contains:

- Discussion of the reaction procedure: Any problems encountered during the reaction development, in any of its parts must be discussed here. The procedure that you tried to overcome these problems should be explained.
- Comparison of the analytical data of the product with values reported in the literature (if available) and.

Example
The product did not distill at the expected temperature. Why?
The product did not crystallize. Why?
Purity is not satisfactory after all the manipulations. Why?

V- Conclusion

Summary of the experience. You have to recall to the reader what type of reaction was performed, the success or not of the experience (how much of the product you have obtained and with what purity), eventual major inconvenient encountered during the experience, any suggestion to give to overcome the problems.

VI- References and notes

VII- Annexes

Date and signature

Date of completion of the report and signature of the author should be included at the end of the document.