Test Project

Chemical Laboratory Technology

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Introduction

The Knoevenagel condensation is an important chemical reaction responsible for forming carbon-carbon bonds between carbonylated substances and active methylene compounds. Due to its great versatility in producing alkenes, it is widely used in the synthesis of several compounds of biological relevance, including the formulation of drugs, natural products, and polymers.

Traditionally, these reactions depend on the addition of weak bases, such as amines, to act as catalysts. However, the compounds used in conventional methods frequently present considerable levels of toxicity. To solve this problem, the scientific community has begun seeking cleaner reaction conditions that are aligned with the principles of green chemistry and cause less environmental impact.

Within this sustainable perspective, the development of Knoevenagel condensations that completely dispense with the use of catalysts has gained prominence. This innovative approach is based on conducting the reaction using only renewable and eco-friendly solvents—such as water, green alcohols, and ionic liquids—ensuring the quick and efficient synthesis of compounds while drastically reducing the toxicity of the overall process.

Description of project and tasks

- Planning
- Reagent testing.
- Reaction and product testing.
- Data processing.
- Conclusion.

The duration of this module is 4 hours.

Instructions to the Competitor

Planning

In your planning, present:

1. The synthesis conditions: reagent amounts, reaction temperature, reaction time with a 15-minute range (±15 min), catalyst, solvent, glassware, and apparatus for the reaction (consult the glassware and apparatus at the workshop to choose). **Show this plan to the Experts before starting the experimental work.**
2. A list or table with all reagents that will be used during the task, their respective principal Globally Harmonized System (GHS) code, and their protective actions**. You must show this list to the Experts before starting the experimental work.**

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Reagent testing

Measure the density and refractive index of the benzaldehyde. Compare the results with the specifications table and request a new aliquot if deemed necessary. Record all values in your report.

Table 1- Benzaldehyde parameters

PARAMETER	MAXIMUM	MINIMUM
Density (g/cm³)	1,025	1,060
Refractive index (20°C)	1,54200	1,56640

Obtain the FTIR spectrum of malononitrile - identified with your country's code located in the refrigerator - and compare it with the malononitrile standard spectrum at "Malononitrile WS2026" library available in the equipment's software. Export a "SpectrumStacked" report and submit it along with your report.

Create a library at the FTIR software and name it "BZDML Standard – XX", were XX is your country's code. Use the benzylidenemalononitrile standard located in the desiccator identified with your country's code to record an FTIR spectrum in the library you created – save the file with the name "BZDML Standard – XX", were XX is your country code.

Reaction and product testing

Plan your reaction to produce 15 grams of benzylidenemalononitrile. You may work under conditions different from those initially planned; however, in this case, you must retain the original plan in your report and create a second one, designating it as a revised plan. **Inform the Experts** so they are aware of the new reaction conditions you will be using.

The table below shows the tolerance you must follow for the reaction parameters:

Table 2 - Reaction parameter tolerances

PARAMETER	TOLERANCE
Time	±15 minutes
Temperature	± 5 minutes
Reagent and solvent amount	± 0,01 g

Attention: call the Experts when measuring the reagents and the reaction solvent.

Attention: call the Experts to inform the moment you start the stopwatch of the reaction time.

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Data processing

The calculations for,

1. Planning
2. Reagent testing
3. Task questions

must be **identified and numbered using exactly these headings** and may be submitted either handwritten or in an Excel spreadsheet. For handwritten work, use the standardized sheets provided by the Experts. For Excel spreadsheets, use a separate tab for each of the three (3) headings and submit the .xlsx file within the allotted exam time.

Conclusion

All text (measurement records, discussion, planning justifications, conclusions, answers to the task questions, etc.) must be submitted within the task duration in a text file saved in PDF format. It can be submitted in your native language. **Any other format will not be considered in the assessment.**

Task questions

1. Calculate the reaction yield and if you don't get it, comment on your strategy for next time achieve the 15 g of product.
2. Compare the FTIR spectrum of the synthesized benzylidenemalononitrile with the spectrum you added to the library (standard) and discuss how the product can be identified and whether you observe any contamination.

Other

List of annexes:

1. Safety data sheets for all chemical reagents in the Competition, in digital format.
2. Basic infrastructure list for this module.
3. Printed Reference Paper.
4. Table of physicochemical properties of the reactants and the product.

Check if you have received all these annexes and immediately inform the Experts if any are missing.

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