Loughborough University

New Techniques and Technologies in Chemistry

22CMD119

Semester 1 2022/23 In-Person Exam paper

This examination is to take place in-person at a central University venue under exam conditions. The standard length of time for this paper is **2 hours**.

You will not be able to leave the exam hall for the first 30 or final 15 minutes of your exam. Your invigilator will collect your exam paper when you have finished.

Help during the exam

Invigilators are not able to answer queries about the content of your exam paper. Instead, please make a note of your query in your answer script to be considered during the marking process.

If you feel unwell, please raise your hand so that an invigilator can assist you.

You may use a calculator for this exam. It must comply with the University's Calculator Policy for In-Person exams, in particular that it must not be able to transmit or receive information (e.g. mobile devices and smart watches are **not** allowed).

Answer **All** questions

1. (a) Discuss the potential benefits and limitations of using flow chemistry compared to conventional batch chemistry. You should include examples to illustrate key arguments in your answer.

[13 marks]

b) Describe, with examples, how it is possible to monitor flow reactions. Use your examples to show different methods on monitoring, showing the advantages and disadvantages.

[12 marks]

2. (a) In a synthetic experiment, the following series of experiments were carried out varying 3 factors: A = temperature; B = time; C = catalyst. Quantify the main effects and the interaction effects, and state which ones are influencing the reaction.

Expt	A	B	C	Conversion
1	+	+	+	82
2	+	+	-	83
3	+	-	+	49
4	+	-	-	61
5	-	+	+	82
6	-	+	-	90
7	-	-	+	60
8	-	-	-	67

[10 marks]

b) It was subsequently decided to look at the effect of an additional factor; the use of different pressures. With four factors, what number of experiments would be required to completely cover the reaction space, with high (+) and low (-) values for each of the four factors? Describe how it would be possible to adjust the experimental design to be more efficient.

[5 marks]

c) With your more efficient design, describe methods you might use to test the reproducibility of the experiments. Take care to describe what exactly you are testing in your proposed method, and what information this could give you.

[5 marks]

d) Describe how you might further optimise the reaction using an automated system. What are the principles involved in an automated optimisation?

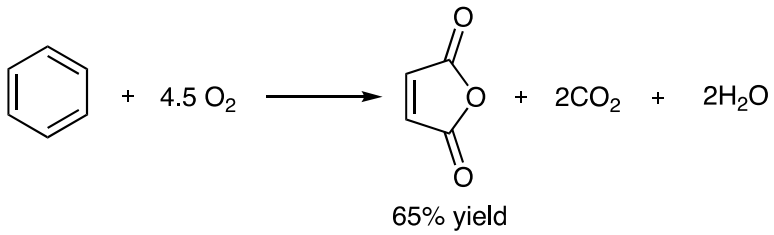
[5 marks]

3. a) Define the term Atom Economy and show how it can be applied in a specific Green Chemistry example.

[4 marks]

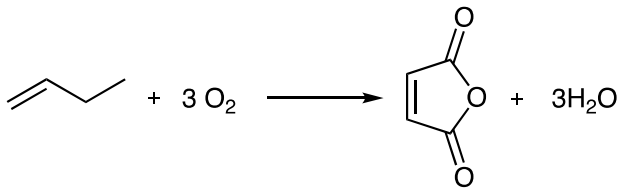
b) Maleic anhydride can be prepared via two routes, **Route A** through the oxidation of benzene or **Route B** through the oxidation of but-1-ene.

Route A



65% yield

Route B



55% yield

i) Assuming each reaction occurs in the gas phase and that no additional chemicals are required calculate the atom economy and the effective mass yield of both reactions (you should assume that O_2, CO_2 and H_2O are non-toxic).

[8 marks]

ii) Which route would you recommend to industry and why?

[3 marks]

c) Describe, with specific examples, two of the other twelve principles of Green Chemistry

[10 marks]

4. A chemical manufacturer creates their product by reacting the precursor with a reducing agent. The reaction takes place at the interface between two immiscible solutions. The precursor is a divalent cation, that is soluble in an aqueous solution. Whilst the reducing agent is a small molecule soluble in 1,2-dichloroethane. The product is also water soluble and a monovalent cation. If the two immiscible liquids are shaken, the reaction yield increases. The manufacturer wishes to understand the reaction mechanism more.

a)

i) What electrochemical technique can be used to elucidate the reaction mechanism? [1 mark]

ii) what are the types of reactions that take place, [11 marks]

iii) and what would be the signal to justify the mechanism? [12 marks]

b) Why would the reaction yield increase upon shaking? [1 mark]

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