CHEMISTRY
THE NEXT GENERATION

6
ACTIVITY

organic molecules day

16-17 age group
7 HRS
60 STUDENTS
12 STUDENTS PER GROUP
8 SUPERVISORS
£2000

background

A wide range of organic molecules, both naturally occurring and man-made, such as therapeutic medicines and dyes tend to be aromatic molecules. In this activity students will carry out the nitration of a simple aromatic compound and then look at the product using various analytical techniques.

pre-planning required

weeks before
Book the laboratory and technicians. Organise demonstrators, book the nuclear magnetic resonance (NMR) spectrometer, mass spectrometer and infrared spectroscopy (IR) facilities, and order the chemicals.

days before
Get the final list of students attending and divide then into groups. It works best if the schools are spread within groups and is probably most efficient if the students work in pairs. Print the copies of spectra from the ChemSoc website database, **www.chemsoc.org/networks/learnnet/spectra/index2.htm**.

facilities required

Appropriate lecture theatre and laboratory

materials required

- concentrated sulfuric acid
- concentrated nitric acid
- methyl benzoate
- deionised water
- crushed ice
- ice baths
- thermometers
- dropping pipettes (*eg* 'calibrated' plastic)
- Buchner flasks
- Buchner funnels and suitable sized filter paper
- ethanol
- steam baths
- melting point apparatus
- conical flasks
- beakers
- stirring rods
- water bottles
- large filter papers

Suggested timings for the day

09.45	Arrival
10.15	Lecture on chirality and its importance in chemistry, biology and pharmacology
10.45	Lecture looking at the discovery of new medicines
11.30	Break
11.45	Careers lecture – why study chemistry?
12.30	Lunch
13.15	Organic practical session
14.00	Groups are taken to analyse the starting material by either MS, NMR or IR. Try to finish by 14.45.
14.50	Groups are taken to analyse their product by either MS, NMR or IR.
15.10	Another group is taken to analyse the product using mass spectrometry.
15.45	Break
16.00	Lecture on up to date research being done at the university
17.00	Finish

SAFETY

A risk assessment must be done for this activity.

This resource is based on an activity run by Dr Alethea Tabor, University College, London.

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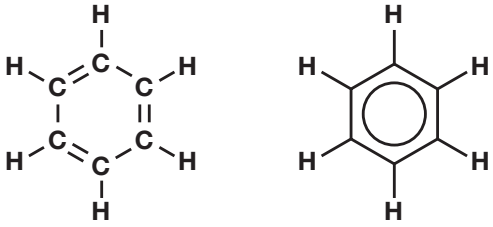
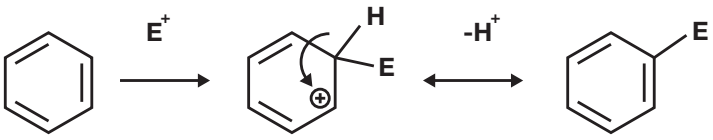
organic molecules day

This activity is designed to give you an insight into what organic chemists do in their day to day work.

A wide range of organic molecules, both naturally occurring and man-made, such as therapeutic medicines and dyes are aromatic compounds. The simplest aromatic compound is benzene.



Aromatic compounds normally undergo substitution reactions – rather than addition reactions. This is because substitution reactions retain the delocalised π-electron system of the aromatic nucleus.



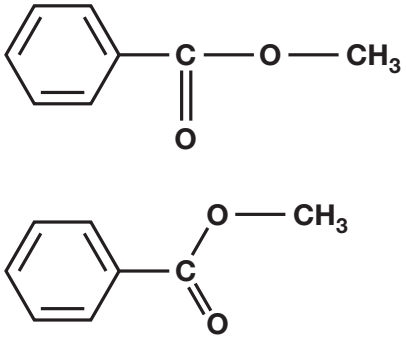
One of the most important substitution reactions of aromatic compounds is nitration – *ie* the addition of an NO_2 group.

SAFETY

Laboratory coats and gloves must be worn at all times. Concentrated sulfuric acid and concentrated nitric acid must be measured in a fume cupboard. No naked flames should be present while ethanol is being used.

The experiment

In this experiment you will nitrate methyl benzoate, which is a relatively simple non-toxic derivative of benzene. You will use a mixture of nitric acid and sulfuric acid to carry out the nitration and then purify the product by recrystallisation.



Finally you will determine the structure of the nitro compound that you have made. How many nitro groups you have added to the ring, and where? You will use a range of spectroscopic techniques – mass spectrometry (MS), infrared (IR) spectroscopy and nuclear magnetic resonance (NMR) spectroscopy – to work out the answer.

The procedure

1. Measure 2.5 cm^3 of methyl benzoate into a small conical flask and then dissolve it in 5 cm^3 of concentrated sulfuric acid. When the liquid has dissolved, cool the mixture in ice.

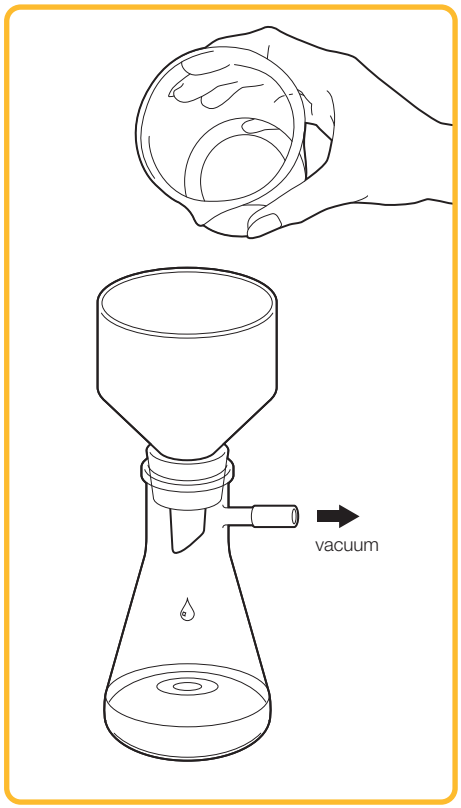
2. Prepare the nitrating mixture by carefully adding 2 cm^3 of concentrated

sulfuric acid to 2 cm^3 of concentrated nitric acid. Cool this mixture in ice.

3 Add the nitrating mixture – drop by drop from a teat pipette – to the solution of methyl benzoate. (NB: Do not allow the nitrating mixture to get into the rubber teat.) Stir the mixture with a glass rod and keep the temperature below 10 ºC. When you have completed the addition, leave the mixture to stand at room temperature for another 15 minutes.

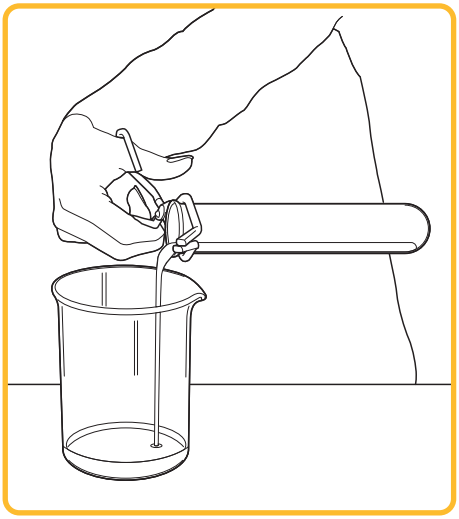
4 After 15 minutes, pour the reaction mixture on to about 25 g of crushed ice and stir until all the ice has melted and the crystalline nitro derivative forms.

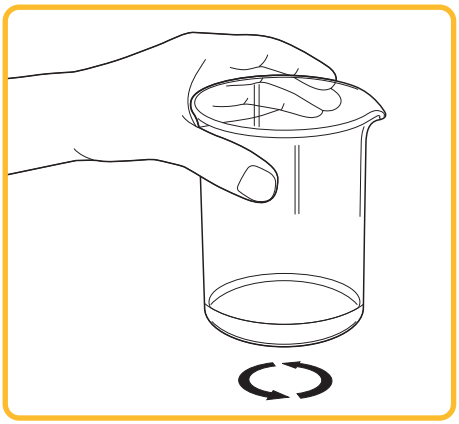
5 Filter the crystals using a Buchner funnel, wash thoroughly with cold water and then transfer to a small beaker.

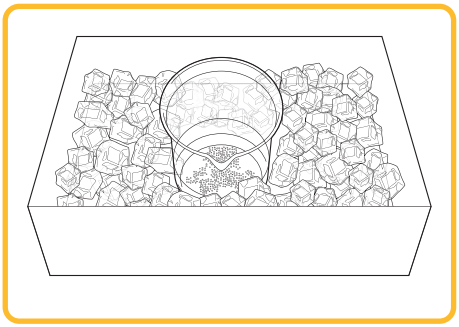


6 Recrystallise the product from hot ethanol. The idea of recrystallisation is to dissolve the impure product in the minimum possible volume of hot solvent. When you cool the solution, the product that you want crystallises out of solution. This is because there is not enough solvent to dissolve it all at the lower temperature. However, the impurities stay behind in solution, because there are less of these impurities there is enough solvent to keep them dissolved. You then filter off the crystals of the product from the remaining solution.

7 Put 15 cm^3 of ethanol in a boiling tube. Warm it to about 50 °C on the steam bath. Dissolve all the crystals in the minimum possible volume of this hot ethanol. Allow the solution to cool to room temperature, then immerse the beaker in ice to complete the crystallisation.







8 Filter the crystals and dry them between filter paper. If there is time, measure the melting point of the crystals.

Analysis

Infrared spectroscopy Compare the spectra of the methyl benzoate and the product. What are the differences? What can you deduce?

Mass spectrometry Compare the spectra of the methyl benzoate and the product. How many nitro groups have been added to the benzene ring? Can you say what position(s) have been substituted?

Nuclear magnetic resonance spectroscopy Compare the spectra of the methyl benzoate and the product. Can you tell how many nitro groups have been added? Can you work out where? Are there any impurities present? Which technique do you find most useful and why?

The theory of these analytical techniques is available on a separate sheet.