

Benzene structure and electrophilic substitution

Chemistry grades 11-12 (A Level · IB · Class 11-12) · Lab

Name: _____ Date: _____ Class: _____

Methyl benzoate is nitrated by a mixture of concentrated acids at low temperature. The lab lets you carry out an electrophilic substitution, calculate the percentage yield and judge the purity of the product from its melting point.

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Aim

To prepare methyl 3-nitrobenzoate by electrophilic substitution, measure the percentage yield and test purity with the melting point. Benzene itself is not used in school because it is carcinogenic.

Equipment

- methyl benzoate (about 2 g), concentrated sulfuric acid, concentrated nitric acid
- boiling tubes or small conical flasks, dropping pipettes
- ice-water bath, thermometer (–10 to 110 °C)
- crushed ice, Buchner funnel and filter paper, ethanol for recrystallisation
- balance, melting point apparatus

Safety

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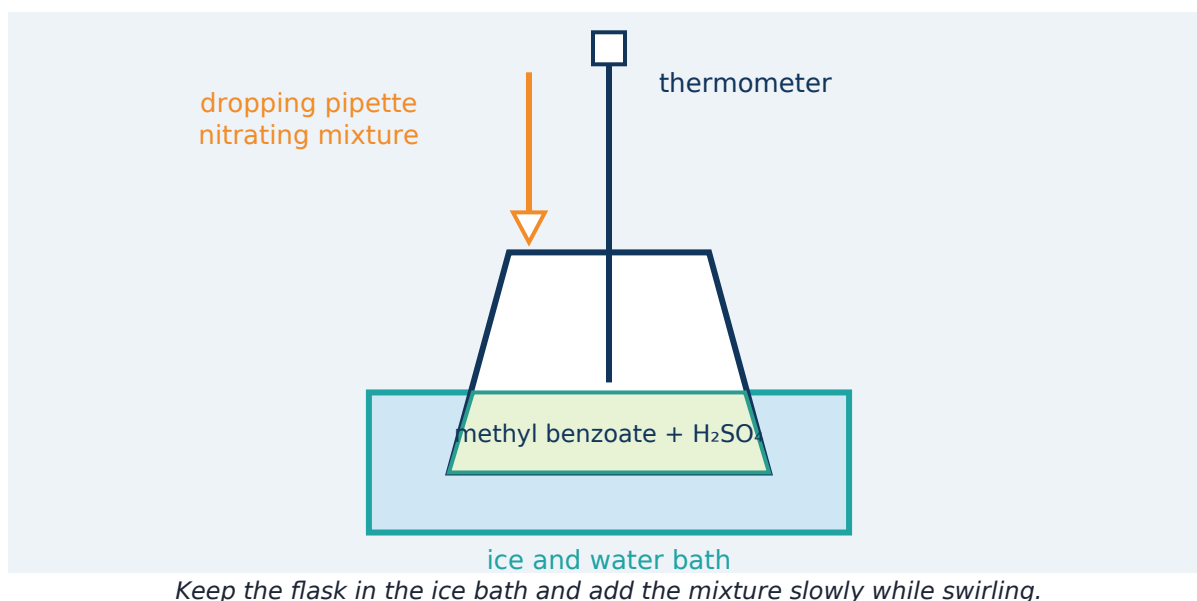
Wear goggles, gloves and a lab coat. Concentrated acids are corrosive and the nitrating mixture can burn skin: add it slowly, keep the flask cold and work in a fume cupboard. Rinse spills with plenty of water.

Method

1. Weigh 2.04 g of methyl benzoate into a flask and add 4 cm³ of concentrated sulfuric acid slowly. Cool in the ice bath below 10 °C.
2. In a separate tube, cool a mixture of 1.5 cm³ concentrated sulfuric acid and 1.5 cm³ concentrated nitric acid in the ice bath.
3. Add the nitrating mixture drop by drop over 10 minutes, swirling and keeping the

temperature below 10 °C.

4. Leave at room temperature for 15 minutes, then pour onto 20 g of crushed ice and stir.
5. Filter, wash with cold water, and recrystallise from a little hot ethanol. Dry, weigh and measure the melting point.



Results

Temperature during the addition of the nitrating mixture:

Time (min)	0	2	4	6	8	10
T (°C)	5	6	8	9	8	7

Masses and melting point:

Quantity	Value
mass of methyl benzoate	2.04 g
mass of dry product	1.63 g
melting point	76-78 °C (literature value 78 °C)

Analysis questions

Question 1 Moles of reactant

Calculate the moles of methyl benzoate, $\text{C}_8\text{H}_8\text{O}_2$ ($M = 136.0 \text{ g/mol}$).

Question 2 Theoretical yield

Calculate the theoretical mass of methyl 3-nitrobenzoate, $\text{C}_8\text{H}_7\text{NO}_4$ ($M = 181.0 \text{ g/mol}$).

Question 3 Percentage yield

Calculate the percentage yield and suggest two reasons it is below 100 %.

Question 4 Temperature control

Explain why the temperature must stay below 10 °C and use the data to comment on the experiment.

Question 5 Role of sulfuric acid

Write the equation for the formation of the electrophile and state the role of sulfuric acid.

Question 6 Melting point

Comment on the melting point range as evidence of purity.

Question 7 Recrystallisation

Explain the purpose of washing with cold water and recrystallising, and suggest one improvement.