

Teacher Resource Bank

GCE Chemistry

PSA17: A2 Organic Chemistry

- Purify an organic solid



Technical Sheet

To purify an organic solid

Whenever possible, students should work individually.

If it is essential to work in a pair or in a small group, because of the availability of apparatus, supervisors must be satisfied that they are able to assess the contribution from each student to the practical activity.

Requirements

- Impure benzenecarboxylic acid (with methyl orange)
- Decolourising charcoal
- Deionised (or distilled) water in a wash bottle
- Reduced pressure filtration apparatus to include:
 - water pump
 - Buchner or Hirsch funnel
 - Buchner or Hirsch flask
 - Buchner or Hirsch filter paper
- Glass stirring rod
- Two 250 cm³ conical flasks
- Plastic bowl to use as an ice bath
- Filter papers and funnel (short stem funnel if available)
- Weighing bottle
- Tripod, gauze and Bunsen burner
- Ice
- Spatula

The vapour from the flask containing heated benzenecarboxylic acid is unpleasant if inhaled and students should be advised to avoid this.

It is suggested that the amount of methyl orange should be approximately 10 drops in 30 g of the solid.

Centres are expected to carry out and be responsible for their own safety risk assessments.



Student Sheet

The aim of this experiment is to purify an organic solid

Introduction

Most preparations of organic compounds lead to the formation of impure products which require purification before the compound can have any use either in the laboratory or commercially. Impurities can occur for many reasons; two of the most common are contamination with reactants due to incomplete reaction and the presence of other compounds due to alternative competing reactions during the preparation.

One of the commonest techniques used to purify organic solids is recrystallisation.

To achieve recrystallisation, the solid is dissolved in the **minimum volume** of a suitable hot solvent and any insoluble impurities are removed by filtration of the hot solution (quickly through a fluted filter paper). Unwanted colouring matter may be removed by heating the solution with a small amount of decolourising charcoal before hot filtration; the charcoal absorbs the coloured material.

The hot solution, when appropriately concentrated, is allowed to cool to enable crystallisation to occur. Trituration (scrapping the inside of the flask with a glass rod to provide nucleation particles on which crystals may form) may aid crystal formation from the cooled solution. Any soluble impurities should remain in solution in the 'mother liquor'.

The crystals are usually filtered off under reduced pressure at a water pump, using either a Buchner or a Hirsch funnel depending on quantity. The crystals are washed with a little of the cold solvent (to remove impure 'mother liquor') and are then left to dry (possibly in a dessicator).

Purity is determined by a melting point determination on the purified dried crystals; the impure solid will melt over a range of temperatures and at a lower temperature than the pure solid. The pure solid should melt at a sharp melting point.

In this experiment a sample of benzenecarboxylic acid has been contaminated by a coloured dye (an indicator) and will be purified by recrystallisation from hot water. The production of pure white crystals of benzenecarboxylic acid at the end of this recrystallisation is a good sign that the purification has been a success.

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It is the responsibility of the student to carry out and be responsible for their own safety risk assessment before carrying out this experiment. Wear safety glasses at all times. Assume that all of the reagents and liquids are toxic, corrosive and flammable.

Experiment

- Weigh out accurately approximately 3 g of the impure benzenecarboxylic acid provided and place it in a 250 cm³ conical flask.
- Dissolve the solid in the minimum volume of hot water.
- Add two spatula measures of decolourising charcoal and heat the mixture, stirring it with a glass rod. Maintain the volume of the boiling solution by adding water from a water bottle.
- Filter the hot solution a little at a time using a fluted filter paper, a hot funnel and a 250 cm³ conical flask to receive the filtrate. It is possible to place the conical flask on top of a tripod and gauze and warm the filtrate gently during the filtration and maintain a hot funnel for the filtration.
- Cool the filtrate rapidly using an ice bath and triturate the solution with a glass rod, allowing the crystals to form.
- Filter the crystals under reduced pressure using a Buchner (or Hirsch) flask and funnel.
- Wash the crystals with a little **cold** water and allow them to dry between filter papers.
- Weigh the dried crystals.

The ability to process the data is NOT part of the PSA but this is a useful task to complete.

Your teacher can help you with this part of the work.

- Calculate the percentage yield of pure benzenecarboxylic acid from your recrystallisation.



Teacher Notes and Marking Guidance

The specific marking guidance in the specification is as follows

2 marks: All areas of the task are carried out competently.

The quantity of solvent is appropriate.

The recrystallisation process is carried out safely and with due care.

The quantity and quality of recrystallised product are both appropriate.

1 mark: One of the areas of the task is performed poorly.

The quantity of solvent is inappropriate **OR**

The recrystallisation process is carried out with insufficient care **OR**

Either the quantity or quality of recrystallised product is inappropriate.

0 marks: At least two of the areas of the task are performed poorly.

The quantity of solvent is inappropriate.

The recrystallisation process is carried out with insufficient care.

Either the quantity or quality of recrystallised product is inappropriate.

Guidance for Teachers

Teachers are expected to exercise professional judgement in assessing the competence of their candidates in following the instructions.

Candidates should have been given guidance in the correct use of equipment and this guidance **can continue during the practical session** for which this PSA forms a part.

If, however, the guidance required is fundamental or frequent, then the student should **not** be awarded 2 marks.

Most judgements of 2 marks, 1 mark or 0 marks will depend on whether the candidates are able to measure out the quantities of solvent and impure solid with due care and attention to the hazards associated with each. It is also possible to judge whether each of the tasks

- purification using decolourising charcoal;
- the recrystallisation;
- the reduced pressure filtration,

is carried out with safety and care and leads to a purified solid which is satisfactory in terms both of its quantity and its visual quality.

The method yields crystals of benzenecarboxylic acid which can be inspected visually for traces of coloured indicator, traces of charcoal and for their crystallinity.

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Wear Eye
Protection

It is important to remember when marking these practical exercises that PSA is about student competence and that for a student to score full marks on this exercise **perfection is neither expected nor required.**