

Teacher Resource Bank

GCE Chemistry

PSA18: A2 Organic Chemistry

- Testing the purity of an organic solid



Technical Sheet

To test the purity of an organic solid by measuring its melting point

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

- Pure benzenecarboxylic acid
- Other pure organic solids as desired by the Centre
- Thermometer (0 °C to 250 °C range)
- Melting point apparatus to include either:
 - an electrothermal m.p. apparatus
 - or
 - oil bath (small beaker half-filled with mineral oil)
 - tripod, gauze and Bunsen burner
 - rubber ring to attach m.p. tube to thermometer
- Melting point tubes
- Watch glass
- Spatula

The Centre may choose to use a range of organic solids, but the target should be to ensure that

- the solid does not decompose on heating
- the m.p. of each solid is in the range 100 °C to 200 °C
- the solid gives a precise and sharp melting point temperature

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



Student Sheet

The aim of this experiment is to test the purity of an organic solid by measuring its melting point.

Introduction

The purity of an organic solid can be determined in part by measuring its melting point and comparing the value with the known Data Book value of the melting point for that compound. A pure dry solid will melt at a precise temperature whereas an impure solid will melt over a range of temperatures which are lower than the melting point of the pure solid.

Melting point apparatus varies in type from the most simple using an oil bath to the more sophisticated electrothermal devices. In every case, the same general principle applies that the heating of a small quantity of the solid in a thin-walled melting point tube should be undertaken slowly and with care. When melting occurs, the solid should collapse into a liquid without any change in temperature and the way in which this occurs can give a clue to the purity of the solid. Repeat measurements should be taken with further samples of the organic solid to verify the reliability of the value obtained.

The method will not work if the solid decomposes on heating.

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

- a) Powder a sample of the organic solid by crushing it gently with a spatula onto the surface of a filter paper.
- b) Fill three melting point tubes with the organic solid to a depth of approximately 0.5 cm.
- c) Set up the melting point apparatus provided and mount one of the melting point tubes ready for taking a measurement.
- d) Heat the apparatus gently and observe the temperature at which the solid collapses into a liquid. The melting point will be in the range 100 °C to 200 °C
- e) Allow the melting point apparatus to cool and repeat the measurement of the melting point of the solid with the other two samples. If the first reading is taken as an approximate value, then the subsequent heating of the other

A2 Organic Chemistry

PSA18 Testing the purity of an organic solid



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two samples can be done more slowly as this approximate value is approached.

- f) On the basis of the three measurements that you have taken, record the melting point of the organic solid.
- g) Ask your teacher for the Data Book value of the melting point for the solid that you have tested and compare this value with your own.



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 used and the preparation of the solid are appropriate (e.g. dry, powder)

The apparatus set-up is safe and appropriate.

Heating is carried out with due care and only as long as necessary, giving an accurate value for the melting point.

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

Either the quantity used or the preparation of the solid is inappropriate **OR**

The apparatus set-up is unsafe or inappropriate **OR**

Heating is longer than necessary and the m.p. is inaccurate.

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

Either the quantity used or the preparation of the solid is inappropriate.

The apparatus set-up is unsafe or inappropriate.

Heating is longer than necessary and the m.p. is inaccurate.

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 fill melting point tubes with appropriate quantities of solid, set up and use the melting point apparatus provided and obtain an accurate melting point for the pure organic solid provided.

Pure benzenecarboxylic acid melts into a colourless liquid at a precise temperature and a judgement of the care taken by candidates can be made on the basis of observing their work and considering the values that they obtain from repeat melting point measurements.

A2 Organic Chemistry

PSA18 Testing the purity of an organic solid



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If centres are concerned that candidates may know the answer to the melting point before taking a measurement, it is quite acceptable to provide a range of solids across any given set of students (see the technical sheet for the criteria).

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.**