

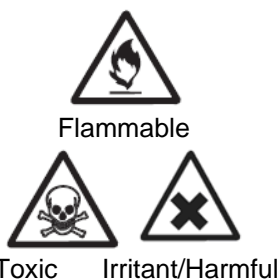
Activity 2: The characteristic test for a carbonyl compound and the use of the 2,4-DNPH (Brady's reagent) derivative to identify an unknown carbonyl compound

Objective

- Be able to perform the characteristic test for a carbonyl compound.
- Use the techniques of recrystallisation and melting point determination to identify an unknown carbonyl compound.

Safety

- Wear safety spectacles and nitrile disposable gloves.



Procedure

1. Dissolve five drops of propanal in 0.5 cm³ of methanol. Add 3 cm³ of 2,4-dinitrophenylhydrazine solution.
2. Stopper and agitate the solution by rocking it from side to side. Record your observations.
3. Repeat using propanone instead of ethanal.
4. Now *identify one of the unknown carbonyl compounds*.
5. Put 0.5 cm³ of methanol in a test tube and add 10 drops of one of the unknown aldehydes or ketones. If the unknown compound does not dissolve, add more methanol drop-wise.
6. Add 5 cm³ of 2,4-dinitrophenylhydrazine to this solution. Stopper the test tube and shake. If a precipitate does not readily form then add a few drops of dilute sulfuric acid until one does.
7. Filter the precipitate under reduced pressure using a small Buchner funnel. Wash with a minimum volume of cold methanol.
8. Recrystallise the precipitate using a solvent of ethanol and water (mixed in equal volumes). Warm the solvent using hot water and dissolve the crystals in a minimum volume. Remove any undissolved solids by filtering the solution (using an ordinary filter funnel, filter paper, etc.) while hot and then allow the filtrate to cool to room temperature. Crystals should form as the filtrate cools. Filter the pure crystals under reduced pressure and dry at room temperature or in an oven maintained below 60 °C.
9. Find and record the melting point of the pure, dry product.

Disposal: All the ammoniacal silver nitrate solution should be washed down the foulwater drain with plenty of water.

Equipment/materials

- Propanal and propanone (highly flammable)
- 1.5 cm³ methanol (highly flammable and toxic by inhalation and contact with skin)
- 11 cm³ 2,4-dinitrophenylhydrazine (2,4-DNPH, Brady's reagent) (toxic by inhalation, dangerous to the environment and can cause explosions when dry)
- Three unknown carbonyl compounds (highly flammable)
- 0.5 mol dm⁻³ sulfuric acid solution (irritant)
- 10 cm³ ethanol (harmful and highly flammable)
- Graduated dropping pipette
- Four dropping pipettes
- Two 10 cm³ measuring cylinders
- Four test tubes
- Stoppers
- Filter funnel, filter paper and 100 cm³ conical flask
- Access to vacuum filtration, melting point apparatus and boiling water
- Oven set below 60 °C

Analysis of results

- What do you notice about the products from the test with 2,4-DNPH?
- Compare the melting point of your pure 2,4-DNPH derivative with the melting points shown in the data.
- Identify the unknown carbonyl compound.

From the examiner

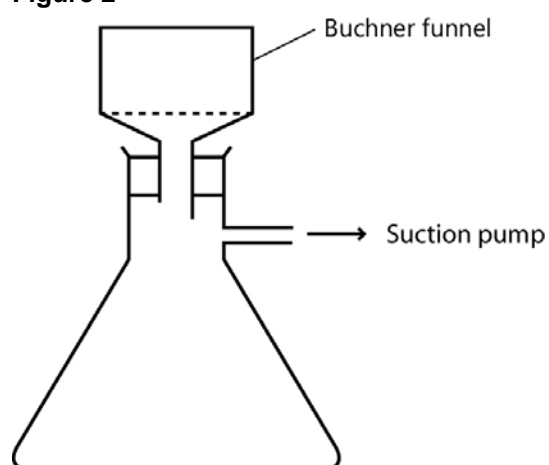
- Demonstrate skilful and safe practical techniques.
- Make and record valid observations.
- Interpret experimental results to reach valid conclusions.

Questions

1. What type of reaction occurs between the carbonyl compound and the 2,4-dinitrophenylhydrazine?
2. Why is it necessary to recrystallise the sample?
3. What would happen to the value of the melting point if the pure crystals were not completely dry?
4. Which methods of spectroscopy might you use as alternatives to determine which carbonyl compound you have?
5. Explain what each method will contribute to the identification of the compound.

Data/diagram**Table 1** Melting points of some 2,4-DNPH derivatives

Carbonyl compound	Mpt of derivative/°C
Ethanal	168
Propanal	155
Benzaldehyde	237
Propanone	128
Butan-2-one	115
4-methylphenylethanone	258

Figure 2

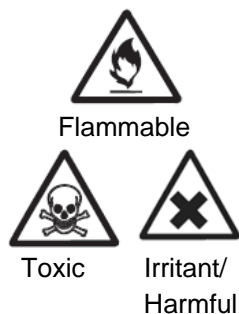
Activity 2: The characteristic test for a carbonyl compound and the use of the 2,4-DNPH (Brady's reagent) derivative to identify an unknown carbonyl compound

Objective

- Be able to perform the characteristic test for a carbonyl compound.
- Use the techniques of recrystallisation and melting point determination to identify an unknown carbonyl compound.

Safety

- Wear safety spectacles and nitrile disposable gloves.
- Propanal, propanone, methanol, carbonyl compounds and ethanol are flammable.
- Methanol and 2,4-DNPH are toxic.
- Sulfuric acid solution is irritant.
- Ethanol is harmful.



From the examiner

- Demonstrate skilful and safe practical techniques.
- Make and record valid observations.
- Interpret experimental results to reach valid conclusions.

Analysis of results and answers

- The product of the reaction between a carbonyl compound and 2,4-DNPH is a yellow/orange crystalline solid.
 - Z is propanal.
 - Y is propanone.
 - X is benzaldehyde.
1. The reaction between a carbonyl compound and 2,4-DNPH is a condensation reaction.
 2. The product is recrystallised to remove any impurities – any remaining impurity will lower the melting point.
 3. Any water/ethanol still present will behave as an impurity and will lower the melting point.
 - 4/5. One method alone would not be sufficient.

Notes on procedure

1. Dissolve five drops of propanal in 0.5 cm³ of methanol. Add 3 cm³ of 2,4-dinitrophenylhydrazine solution.
2. Stopper and agitate the solution by rocking it from side to side. Record your observations.
3. Repeat using propanone instead of ethanol.
4. Now *identify one of the unknown carbonyl compounds*.
5. Put 0.5 cm³ of methanol in a test tube and add 10 drops of one of the unknown aldehydes or ketones. If the unknown compound does not dissolve, add more methanol drop-wise.
6. Add 5 cm³ of 2,4-dinitrophenylhydrazine to this solution. Stopper the test tube and shake. If a precipitate does not readily form then add a few drops of dilute sulfuric acid until one does.
7. Filter the precipitate under reduced pressure using a small Buchner funnel. Wash with a minimum volume of cold methanol.
8. Recrystallise the precipitate using a solvent of ethanol and water (mixed in equal volumes). Warm the solvent using hot water and dissolve the crystals in a minimum volume. Remove any undissolved solids by filtering the solution (using an ordinary filter funnel, filter paper, etc.) while hot and then allow the filtrate to cool to room temperature. Crystals should form as the filtrate cools. Filter the pure crystals under reduced pressure (if your school does not have the required water pressure use a hand pump) and dry at room temperature or in an oven maintained below 60 °C.
9. Find and record the melting point of the pure, dry product.
10. If there is time the students could repeat this with another unknown carbonyl compound.

Infrared spectroscopy

The spectrum will have an absorption at 1680–1750 cm⁻¹, showing the presence of the carbonyl group or C=O bond.

Mass spectrometry

The molecular ion peak (the peak furthest to the right) will give the relative molecular mass of the compound. Fragmentation will also produce ions which include the carbonyl group as they are relatively stable.

NMR

The varying proton environments can be found from the chemical shifts of the peaks. The relative areas under the peaks will give the relative number of protons present in the peaks. Any splitting patterns observed will give the number of protons in the adjacent environment (according to the *n*+1 rule).

Activity 2: The characteristic test for a carbonyl compound and the use of the 2,4-DNPH (Brady's reagent) derivative to identify an unknown carbonyl compound

Objective

- Be able to perform the characteristic test for a carbonyl compound.
- Use the techniques of recrystallisation and melting point determination to identify an unknown carbonyl compound.

Safety

- Wear safety spectacles.
- Wear nitrile disposable gloves.

Requirements per student*/group of students	Notes
2cm ³ methanol	Highly flammable and toxic by inhalation and contact with skin
12cm ³ 2,4-dinitrophenylhydrazine (Brady's reagent)	Toxic by inhalation, dangerous to the environment and can cause explosions when dry See CLEAPSS recipe card 21 for preparation.
Propanal (available to the whole class in a small bottle)	Extremely flammable and irritating to eyes
Propanone (available to the whole class in a small bottle)	Extremely flammable and irritating to eyes
Propanal in a small bottle labelled with the letter Z	Total volume 25cm ³ Extremely flammable and irritating to eyes
Propanone in a small bottle labelled with the letter Y	Total volume 25cm ³ Extremely flammable and irritating to eyes
Benzaldehyde in a small bottle labelled with the letter X	Total volume 25cm ³ Extremely flammable and irritating to eyes
0.5mol dm ⁻³ sulfuric acid solution	Irritant If prepared fresh for this experiment from conc. sulfuric acid, remember conc. sulfuric acid is corrosive and oxidising.
10cm ³ ethanol	Harmful
Graduated dropping pipette	To measure the methanol
Four dropping pipettes	To measure each carbonyl compound
Two 10cm ³ measuring cylinders	To measure the 2,4-DNPH
Four test tubes and stoppers	
Filter funnel, filter paper and 100cm ³ conical flask	
Buchner funnel, Buchner flask with water (suction) pump and filter paper to fit funnel	
Melting point apparatus and melting point tubes	
Kettle	
Oven set below 60°C	

Notes

The students should have access to a kettle to provide them with boiling water for step 8.

CLEAPSS Hazcards® (30, 34, 40B and 85) should be consulted and a risk assessment carried out using up-to-date information before this practical is carried out.

Disposal: All the ammoniacal silver nitrate solution should be washed down the foulwater drain with plenty of water. It must not be stored (silver filaments form which have been known to explode).