21. Planning an experiment to identify a carbonyl compound

Student Sheet

In this exercise you will use your knowledge of the chemistry of carbonyl compounds, most of which is covered in the AS syllabus, to plan a series of experiments to identify a carbonyl compound from a small number of possibilities. You will also need to use spectroscopic data to confirm your identification.

Intended lesson outcomes

By the end of this planning exercise you will be able to:

- identify the relevant structural features of carbonyl compounds
- select suitable tests to use to identify an unknown carbonyl compound
- give outline details of each test, including possible observations
- outline the preparation and purification of a solid derivative
- discuss the use of spectroscopic data in confirming identity
- assess the risks involved and suggest appropriate safety precautions

The exercise

In this exercise you will be assessed on your ability to plan experiments to identify a carbonyl compound and to use spectroscopic data to confirm your identification.

Background Information

The carbonyl compound you have to identify is one of the following.

- propanal
- propanone
- butanal
- butanone
- pentan-2-one
- pentan-3-one

In your plan you must first identify the structural features of these compounds by which you will be able to distinguish between them. You should then propose an appropriate series of tests and spectral analyses to perform.

- Your plan does not need to include quantities but should give reagents and conditions.
- The carbonyl compounds are all colourless, flammable liquids.
- Your plan for the laboratory exercises should allow for the compound being any one of those listed. However, when describing the use of spectroscopic data, you may use one of the compounds as an example.
- You may use the NMR data tables provided to find chemical shift values.

Plan

Your plan should follow the sequence outlined below.

1 Identify the structural features of the carbonyl compounds that you will make use of in your tests and spectral analyses.

Chemical testing

2 Give an outline, including any possible observations, of a test you would perform to confirm that the unknown is a carbonyl compound.

3 Give an outline, including any possible observations, of a test you would perform to distinguish between an aldehyde and a ketone.

4 Give an outline, including any possible observations, of a test you would perform to identify all the compounds with a β-keto group (CH_3CO–).

5 Describe how you would confirm the identity of the compound by preparing a solid derivative. You should include full practical details of how the derivative would be purified and how it would be used to confirm the identity of the carbonyl compound.

Spectral analysis

6 In this part of the plan you may use any one of the compounds listed. **Using one** of the possible compounds, describe how you would make use of spectroscopic data on the carbonyl compound to confirm your identification. The techniques you should consider are:

(i) Low-resolution nuclear magnetic resonance spectroscopy

(ii) Mass spectroscopy (including fragmentation patterns)

Safety considerations

7 Perform a risk assessment of your plan.

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Teachers' Notes

Planning exercises work best when students have sufficient knowledge of the topic not to need the support of books or notes. As planning may be a new skill to many students it is recommended that at the beginning of the course, group work is used so students can pool ideas and learn from each other. As students become more confident, they can progress to individual work, and finally to practising planning exercises under exam conditions.

From the student's point of view, often the most difficult part of a planning exercise is getting started. Be prepared to give help early on in the learning process, but encourage students to be more independent as the course progresses.

In this exercise, students will need access to NMR spectroscopic data. These data should be no more than a simple table showing the chemical shift/δ value ranges for common proton environments.

The main questions you might wish to consider when assessing the plan are

- Is the proposed overall method clearly described?
- Is the proposed overall method appropriate, logical and complete?
- Are the practical details provided adequate and workable?
- Is the scope of the plan comprehensive enough?
- Have all the appropriate safety factors been properly considered?

The following list of points will help you work through the students' plans methodically and give appropriate feedback or marks.

Structural Features

- Propanal and butanal have –CHO group and so are **aldehydes**
- Propanone, butanone, pentan-2-one and pentan-3-one have –CCOC– group and so are **ketones**.
- Propanone, butanone and pentan-2-one have a **β-keto group** (CH_3CO–) group and so give positive results with the **tri-iodomethane** (**iodoform**) test.

Carbonyl test

- Add compound to 2,4-dinitrophenylhydrazine solution
- Yellow/orange precipitate with all five compounds

Aldehyde/ketone test

- Add compound to ammoniacal silver nitrate and warm or to Fehling's solution and heat
- If silver mirror/red precipitate formed then compound is an aldehyde
- If no silver mirror/red precipitate formed then compound is not an aldehyde so, as it is a carbonyl compound, it is a ketone.

Tri-iodomethane (iodoform) test

- To compound add KI(aq) and NaC*l*O(aq) and warm the mixture.
- Cool the mixture; fine yellow crystals form with β-keto compounds.
- If no yellow precipitate, then compound does not contain a β-keto group.

Derivative

- Prepares 2,4-dinitrophenylhydrazine derivative (a 2,4-dinitrophenylhydrazone)

- Suggests a suitable quantity to prepare (students could be told to give details of quantities, or a reference to making 'sufficient for a melting point determination' might be acceptable).

Recrystallisation

- Filter off 2,4-DNP derivative and dissolve in **minimum volume** …
- … of **hot solvent**
- Details of how to filter solution **hot** (e.g. fluted filter paper/pre-heated funnel/use Buchner funnel
- Cool solution and crystals form
- Filter crystals using Buchner funnel and flask, wash crystals with cold solvent and dry

Melting point

- Find melting temperature of crystals – brief details of technique to be used
- Compare melting temperature with those listed in data book or similar

Spectroscopic analysis of chosen example

- Quotes formula and correct *m*/*e* value for the molecular ion
- Quotes the formula and *m*/*e* value for one correct fragment ion from given compound
- Deduces number of peaks in nmr spectrum
- Deduces expected height/area under peaks/integration trace ratio in NMR spectrum

Safety

- Avoid use of naked flame/use of water bath because liquids flammable
- Wear eye protection at all times

Technical information

- Access to hazard cards or equivalent
- Access to the chemical shift/δ value ranges for common proton environments
- No access to outside sources
- When candidates have some experience in planning, this exercise could be carried out in exam-style conditions with a time limit of around 1 hour

Note: Make a record of any help given to a student, and the extent of that assistance. However, students will need to be given access to appropriate hazard cards and spectroscopic data tables.

Safety

If this exercise is extended so that the students carry out their plans, you must check their risk assessments against the appropriate MSDS sheets and ensure that they are fully aware of any hazards.