

Solving structures with mass, IR and NMR spectra

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

Name: _____ Date: _____ Class: _____

Spectra are easier to predict when you know the functional group. In this lab you use quick test-tube reactions to identify three colourless liquids, then predict their mass, infrared and NMR spectra and compare each prediction with what the chemistry showed.

Level: Chemistry grades 11-12 (A Level · IB · Class 11-12) · **Curriculum:** IB S3.2

Aim

To use three simple chemical tests to identify ethanol, propanone and ethanoic acid, then predict the mass, infrared and NMR spectra of each.

Equipment

- Three labelled liquids A, B and C (ethanol, propanone and ethanoic acid, 1 cm³ each)
- Six test tubes and a rack, three dropping pipettes
- 0.5 mol/dm³ sodium carbonate solution
- Acidified potassium dichromate(VI) solution, 0.1 mol/dm³ (teacher prepared)
- 2,4-dinitrophenylhydrazine solution (2,4-DNPH, teacher prepared)
- Hot water bath at 60 °C
- Gloves, eye protection

Safety

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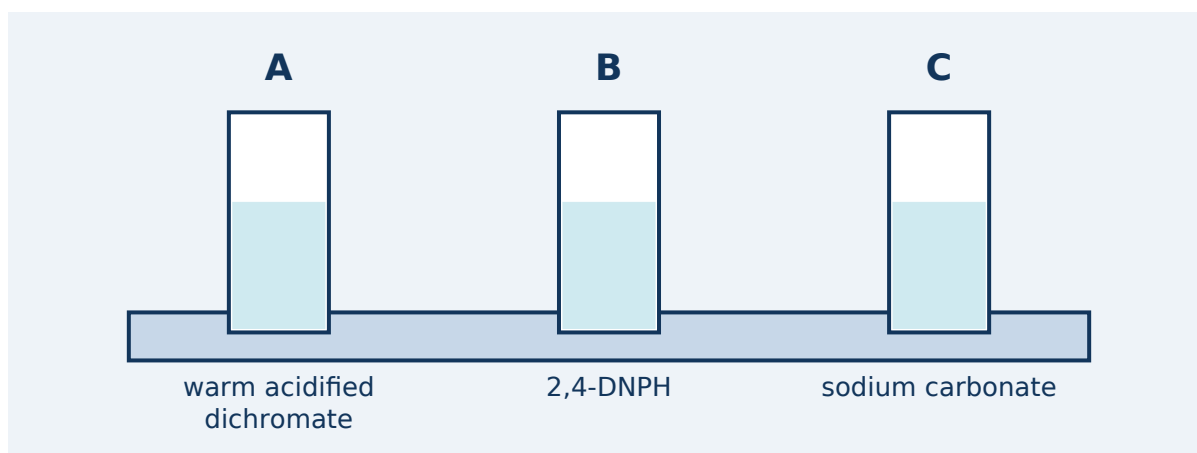
Wear gloves and eye protection throughout. Ethanol and propanone are highly flammable, so keep flames away. Dichromate(VI) is toxic and a suspected carcinogen, 2,4-DNPH is irritant and stains skin, and ethanoic acid is corrosive. Collect all waste in a labelled bottle.

Method

1. Add 1 cm³ of each liquid to its own test tube labelled A, B or C.
2. To a second set of three tubes, add 1 cm³ of each liquid again.
3. To the first set, add 1 cm³ of sodium carbonate solution and note any bubbles.
4. To the second set, add 5 drops of acidified potassium dichromate and warm in the water

bath for 3 minutes.

- Place 2 drops of each liquid into fresh tubes and add 5 drops of 2,4-DNPH. Shake and observe.
- Record every observation in the table. Dispose of waste as instructed.



Each unknown liquid is tested separately in a test tube rack.

Results

Liquid	Sodium carbonate	Warm acidified dichromate	2,4-DNPH
A	no change	orange to green	no change
B	no change	no change, stays orange	orange precipitate
C	steady bubbles of gas	no change, stays orange	no change

Analysis questions

Question 1 Identify the liquids

Use the results to identify A, B and C, and give the evidence for each.

Question 2 Write the carbonate equation

Write a balanced equation for the reaction of ethanoic acid with sodium carbonate and name the gas.

Question 3 Predict the IR spectra

State the key IR absorptions, in cm^{-1} , that would show A, B and C are the compounds you identified.

Question 4 Predict the molecular ions

Calculate the m/z of the molecular ion for each liquid ($\text{C} = 12$, $\text{H} = 1$, $\text{O} = 16$).

Question 5 Predict proton NMR

Give the number of proton NMR signals for each liquid and the splitting of the signals.

Question 6 Predict ^{13}C NMR

State the number of ^{13}C signals for each liquid and say which carbon is at the highest shift.

Question 7 Evaluate the method

Explain one limitation of the 2,4-DNPH test and suggest a way to improve identification.