CHEMISTRY B (SALTERS)
Theme: Carbon-13 MMR
October 2015
We will inform centres about any changes to the specification. We will also publish changes on our website. The latest version of our specification will always be the one on our website (www.ocr.org.uk) and this may differ from printed versions.

Copyright © 2014 OCR. All rights reserved.

Copyright
OCR retains the copyright on all its publications, including the specifications. However, registered centres for OCR are permitted to copy material from this specification booklet for their own internal use.


Registered office: 1 Hills Road
Cambridge
CB1 2EU

OCR is an exempt charity.
Introduction

Carbon-13 (\(^{13}\text{C}\)) NMR is a key technique which is used routinely by synthetic chemists in the analysis of the products of chemical reactions. The spectra produced are simpler in appearance than high-resolution proton (\(^{1}\text{H}\)) NMR spectra because the instrument is run in such a way that no splitting patterns are observed. For this reason, it might be considered appropriate to teach carbon-13 NMR before covering proton NMR as learners can develop a solid grasp of the fundamentals before moving onto the challenge of dealing with the complexities of integrals, splitting patterns and the \(n+1\) rule.

The key skill for learners to develop to succeed in this topic is the ability to identify how many unique carbon environments are present in a molecule. Sometimes the representations we use to illustrate molecules (structural, displayed and skeletal formulae) are drawn in such a way that it is difficult for learners to correctly identify atoms that are in the same chemical environment. It is therefore important that learners are able to visualise chemical structures correctly in their minds regardless of the way a structure is depicted. Molecular models may be useful to support this, and 3-D computer graphics such as jmol images are also beneficial. Examples of such images are incorporated in the Spectraschool website (http://spectraschool.rsc.org/) along with the relevant spectra for each compound.

The suggested activities are arranged in an order which supports learners in developing the basic skills required to predict a) the number of peaks that will be present in the carbon-13 NMR spectrum of a particular compound and b) the approximate chemical shift values of the peaks. Having completed these activities, learners will be well placed to interpret carbon-13 NMR spectra and make inferences about chemical structures based on the data.

Learning Outcomes
The resources in this Topic Exploration Pack support the teaching and learning of the following specification statements:
PL(s) (proton and) carbon-13 nuclear magnetic resonance (NMR) spectra for the determination of molecular structure
PL(t) the combination of spectroscopic techniques (mass spectrometry, IR and NMR) to determine the structure of organic molecules
Suggested Activities

It is recommended that learners are introduced to the idea of identifying the different chemical environments in molecules before discussing carbon-13 NMR. Activity 1 features an illustration of how to do this before asking learners to identify for themselves the numbers of carbon environments present in a range of simple hydrocarbons. This activity should take no more than 10–15 minutes, depending on the learners’ abilities to visualise structures in three dimensions. If molecular models are available, it might be valuable to have pre-made models of the molecules in question to hand during the lesson.

Once learners are confident with the assignment of chemical environments in different molecules, the concept of carbon-13 NMR can be introduced. The background theory to NMR spectroscopy is not examinable at A-level, and even the fundamentals are challenging for all but the most able A-level students. As such, it is recommended that the theory is not covered in any depth. The Spectraschool (http://spectraschool.rsc.org/) and Chemguide (www.chemguide.co.uk) websites provide excellent background information for those who are interested, and certainly make good reading for teachers wishing to boost their expertise prior to teaching this material. The resources provided in this pack don’t go into detail regarding background theory. Teachers should decide for themselves what they want to cover in this regard, depending on the abilities/interests of learners and the resources available such as textbooks.

Activity 2 introduces carbon-13 spectra with a simple example (bromoethane) and outlines the key features of such a spectrum and includes discussion of the fact that the chemical shift of a particular signal is influenced by the substituents attached to the carbon atom in question. The worksheet then relates the activities carried out as part of Activity 1 to the prediction of the appearance of carbon-13 NMR spectra. This learning is then consolidated by an activity in which learners are asked to predict the appearances of the carbon-13 NMR spectra of four different compounds and to then match their predictions to the actual spectra of the compounds in question.

Activity 3 involves learners identifying six unknown compounds using spectroscopic data. As well as carbon-13 NMR spectra, proton NMR and infrared spectra are provided, the latter of which will have been studied previously. Mass spectra are also provided, but it is suggested that these are not given to the learners in the first instance as they will simply calculate the formula masses of the compounds based on the structures they’ve been given, which they can then match up with the mass spectra to get the correct assignments in an expedient, but less educationally beneficial,
manner. It is recommended that learners are provided with the NMR and IR data at the start of the activity. Once they have worked out the assignment based on the spectroscopic data, the learners can be issued with the mass spectra so they can confirm that their answers are correct. Furthermore, learners can be asked to identify some of the fragment ions present in the mass spectra if this is something that has been covered already. Learners should be asked to record all of the spectroscopic data for each unknown in a systematic fashion (eg in a table) and they should be able to explain how they used the spectroscopic data to form their conclusions.

If the activity is being done before proton NMR has been taught, the students could be asked to ignore those spectra, as it should be possible to complete the assignment using the carbon-13 and IR spectra alone. However, this might be a useful activity to do after teaching of the whole ‘Modern analytical techniques’ topic in Polymers of life (PL) has been completed as it illustrates the relationship between the different techniques and highlights the importance of collecting a range of evidence to support a conclusion. It might be useful to set an exam style question, perhaps based on something from a past paper, as a follow up so learners can build their confidence in tackling assessment tasks.

Below is a suggested plan for how to run Activity 3:

1. Issue learners with the IR, proton and carbon-13 spectra (see page 13 and beyond in this document) and the Activity 3 handout. It would be a good idea to also issue learners with OCR’s GCE Chemistry B Data Sheet so they get used to working with this document.
2. Explain what is expected in terms of the recording/tabulation of spectroscopic data and annotation of spectra. Learners should also be given appropriate guidance regarding how to get started with the activity depending on their confidence and ability levels.
3. Once learners have completed their initial assignment of spectra to structures, they can be issued with the mass spectra (see page 31 and beyond) which will allow them to confirm whether or not their answers are correct. Learners can also be asked to identify some of the fragment ions in the mass spectra.
4. Learners who finish early and have the correct answers can be asked to go round and help other learners who are struggling.
Correct Answers for Activities

Activity 1 Task 1

Activity 1 Task 2

1. 2,5-Dimethylhexane

3 carbon environments
2. 3-Ethylpentane

\[ \text{H}_3\text{C} - \text{CH}_2 - \text{CH}_2 - \text{CH} - \text{CH}_2 - \text{CH}_3 \]

3 carbon environments

3. Benzene

\[ \text{C}_6\text{H}_6 \]

1 carbon environment

4. Methylbenzene

\[ \text{CH}_3 - \text{C}_6\text{H}_4 - \text{CH}_3 \]

5 carbon environments
5. 1,2-Dimethylbenzene

6. 1,3-Dimethylbenzene
Activity 2 Task

1. Propanal

2. 2-Aminobutane
3. cis-Hex-3-ene

4. Methylbenzoate
Activity 3

1. ![Structure](image1.png) unknown 3 ethyl propanoate

2. ![Structure](image2.png) unknown 5 butanoic acid

3. ![Structure](image3.png) unknown 4 1-phenylmethanamine

4. ![Structure](image4.png) unknown 2 pentan-3-one

5. ![Structure](image5.png) unknown 6 propan-2-ol

6. ![Structure](image6.png) unknown 1 ethanol
Appendix 1

Unknown 1

Infrared spectrum

IR Spectrum

transmittance / %

(1882, 63)

wavenumber / cm⁻¹

4000 3500 3000 2500 2000 1500 1000 500

0 10 20 30 40 50 60 70 80 90 100
Unknown 1

$^1$H NMR spectrum
Unknown 2

Infrared spectrum

transmittance / %

IR Spectrum

(3973, 88.5)

wavenumber / cm⁻¹
Unknown 2

$^1$H NMR spectrum
Unknown 2

$^{13}$C NMR spectrum
Unknown 3

Infrared spectrum

transmittance / \% 

IR Spectrum

wavenumber / cm\(^{-1}\)
Unknown 3

$^1$H NMR spectrum

- 2H
- 3H
- 3H
Unknown 3

$^{13}$C NMR spectrum
Unknown 4

$^1$H NMR spectrum
Unknown 4

$^{13}$C NMR spectrum
Unknown 5

Infrared spectrum

Transmittance / %

IR Spectrum

Wavenumber / cm⁻¹

4000 3500 3000 2500 2000 1500 1000 500
Unknown 5

$^{13}$C NMR spectrum
Unknown 6

Infrared spectrum
Unknown 6

$^1$H NMR spectrum
Unknown 6

$^{13}$C NMR spectrum

signal strength / %

$^{13}$C NMR Spectrum

chemical shift / ppm

0 10 20 30 40 50 60 70 80 90 100
Appendix 2

Unknown 1

Mass spectrum
Unknown 2

Mass spectrum
Unknown 4

Mass spectrum
Unknown 5

Mass spectrum
Unknown 6

Mass spectrum