Determining the structure of compounds

This resource accompanies the article **How to teach spectroscopy at post-16** in *Education in Chemistry* which can be viewed at: rsc.li/3IEhyi2. The article provides ideas and resources to help your learners master spectroscopic techniques.

Learning objectives

1 Understand, analyse and summarise a scientific text.

2 Know that a combination of data from instrumental analysis is required to determine the structure of a compound.

3 Determine the structure of compounds by analysing data from mass spectrometry (MS), infrared (IR) and ^{1}H nuclear magnetic resonance (NMR) spectra.

Introduction

Providing context to your learners helps them make real-world connections to the content they learn in the classroom. Analytical chemistry's uses in sport and anti-doping can be an engaging hook for your lessons on spectroscopy and structure determination. Inspire your post-16 learners with interesting chemistry careers. For example, Nicola's job profile about her role as an assistant analyst at a drug control centre, available from rsc.li/3k2Hw4E.

How to use this resource

These tasks will sit well within a series on structure determination and spectroscopy. Learners will need to understand the three basic techniques of mass spectrometry, infrared spectroscopy and NMR spectroscopy and have basic organic chemistry knowledge. You could present the resource as a revision guide as well as using it to support your learners' development of understanding.

Task 1 provides the opportunity for information analysis and comprehension. Learners will read background information on nandrolone, an anabolic steroid. They will need to synthesise, share and query ideas in answering the questions and writing the summary to athletes and their national governing bodies. The summaries will indicate if they have understood the key points. Use the discussion on banned substances based on your learners' advice to develop their communication, listening and peer assessment skills. Challenge your learners to think of additional techniques that may help resolve drug testing cases.

Introduce the second task by making links between the information on nandrolone and the techniques used to determine the structure of compounds. The task requires the synthesis of complex data and is a great opportunity for you to provide groups or individuals with formative assessment and feedback. Explain to learners that they will use mass spectra, infrared spectra and ^{1}H NMR spectra to determine the compounds' structures. Make sure they have a data book or sheet available to help them identify key bonds and groups present.

Modelling how to complete the task using the 'I do, we do, you do' approach could be beneficial in helping your learners feel comfortable with the activity. The *Education in Chemistry* CPD article Teaching structure determination post-16, available from rsc.li/3CCNrnl, provides logical steps to help make spectra less puzzling.

Differentiation

The compounds are in order of difficulty from 1 to 7, so differentiation is also possible. You could split the class into groups and give them one compound each or task them to work through the series. The student worksheet includes some clues. You can give further prompts if individuals/groups are struggling, such as:

- Mass spectrometry provides information about molecular formula – this is a good starting point.
- IR spectra provide information on key functional groups.
- Integrated ^{1}H NMR spectra indicate the relative numbers of hydrogen atoms in different environments.

Answers

Task 1

1. An anabolic steroid sold as Deca-Durabolin; a dietary supplement for building body mass.
2.

(a) Testosterone has an additional methyl group.
(b) Nandrolone's and 19-norandrosterone's –OH and =O groups are in opposite positions.

3. Oxidise the –OH group, reduce the =O group.
4. Use of HRMS and HPLC.
5. Building body mass artificially by drug use is against the rules of sport.
6. The substance must be detected at very low levels (2 ng ml^{-1}).
7. 19-Norandrosterone is $C_{18}H_{29}O_2$ and its molecular ion peak is 277. The main fragments on the mass spectrum would be:
   - 242 loss of =O, –OH and two other Hs.
   - 201 loss of 5-C ring (C_5O = 76).
   - 185 loss of 6-C ring (C_6H_4O = 92).

- 145 molecule splits (loss of C_9H_8O = 132).
- Other corresponding peaks at 132, 92, 76.

8. High levels have been claimed to be a result of eating non-organic beef obtained from cattle treated with the steroids, having sex and that 19-norandrosterone can be formed from the legal substance creatine found in high-protein milkshakes.
9. Learner-led discussion.

Summary answers will be learner dependent and discussed by the class.

Task 2

Compound 1 is propan-2-ol $CH_3CH(OH)CH_3$

- *m/z* 45 loss of CH_3
- IR wavenumber / cm^{-1} 3010 O–H (alcohol)
- ^{1}H NMR, δH / ppm (the hydrogens in each environment are emboldened below.)
  - 1.1 (doublet, integration 6) $\mathbf{CH_3}CH(OH)\mathbf{CH_3}$
  - 3.9 (septet, integration 1) $CH_3\mathbf{CH}(OH)CH_3$
  - 4.1 (singlet, integration 1) $CH_3CH(O\mathbf{H})CH_3$ – lost on shaking sample with D_2O

Compound 2 is ethyl phenyl ether $C_6H_5OCH_2CH_3$

- *m/z* 94 loss of CH_3CH_2 and protonation of fragment remaining
- IR wavenumber / cm^{-1} 1600 and 1500 benzene ring
- ^{1}H NMR, δH / ppm (the hydrogens in each environment are emboldened below.)
  - 1.4 (triplet, integration 3) $C_6H_5OCH_2\mathbf{CH_3}$
  - 4.0 (quartet, integration 2) $C_6H_5O\mathbf{CH_2}CH_3$
  - 7.5-6.8 (integration 5) $C_6\mathbf{H_5}OCH_2CH_3$

Compound 3 is methyl 2-methylprop-2-enoate $CH_3OCOC(CH_3)CH_2$

- *m/z* 69 loss of CH_3O; 41 loss of CH_3OCO
- IR wavenumber / cm^{-1} 1740 C=O; 1660 C=C
- ^{1}H NMR, δH / ppm (the hydrogens in each environment are emboldened below.)
  - 2.0 (singlet, integration 3) $CH_3OCOC(\mathbf{CH_3})CH_2$
  - 4.0 (singlet, integration 3) $\mathbf{CH_3}OCOC(CH_3)CH_2$
  - 6.2 and 5.7(two singlets, each integration 1) $CH_3OCOC(CH_3)\mathbf{CH_2}$

Compound 4 is benzyl ethanoate $CH_3COOCH_2C_6H_5$

- *m/z* 108 loss of CH_3CO and protonation of fragment; 91 loss of CH_3COO
- IR wavenumber / cm^{-1} 1710 C=O
- ^{1}H NMR, δH / ppm (the hydrogens in each environment are emboldened below.)
    - 2.0 (singlet, integration 3) $\mathbf{CH_3}COOCH_2C_6H_5$
    - 5.0 (singlet, integration 2) $CH_3COO\mathbf{CH_2}C_6H_5$
    - 7.3 (broad, integration 5) $CH_3COOCH_2C_6\mathbf{H_5}$

Compound 5 is 1-(4-chlorophenyl) ethanone $ClC_6H_4COCH_3$

- *m/z* 139 loss of CH_3; 111 loss of CH_3CO
- IR wavenumber / cm^{-1} 1700 C=O; 1600 and 1500 benzene ring
- ^{1}H NMR, δH / ppm (the hydrogens in each environment are emboldened below.)
    - 2.6 (singlet, integration 3) $ClC_6H_4CO\mathbf{CH_3}$
    - 7.5 (doublet, integration 2) $ClC_6\mathbf{H_4}COCH_3$
    - 8.0 (doublet, integration 2) $ClC_6\mathbf{H_4}COCH_3$

Compound 6 is cyclohexanone $(CH_2)_5CO$

- *m/z* 42 $[CH_2CO]^+$
- IR wavenumber / cm^{-1} 1700 C=O
- ^{1}H NMR, δH / ppm (the hydrogens in each environment are emboldened below.)
    - 1.9 (broad, integration 6) $(\mathbf{CH_2})_5CO$
    - 2.3 (broad, integration 4) $(\mathbf{CH_2})_5CO$ – CH_2 groups closest to C=O

Compound 7 is 2-aminobutane $CH_3CH_2CHNH_2CH_3$

- *m/z* 58 loss of CH_3; 44 loss of CH_3CH_2
- IR wavenumber / cm^{-1} 3400 NH_2
- ^{1}H NMR, δH / ppm (the hydrogens in each environment are emboldened below.)
    - 1.6-1.0 (broad, integration 10) $\mathbf{CH_3CH_2}CHNH_2\mathbf{CH_3}$
    - 3.0-2.6 (broad, integration 1) $CH_3CH_2\mathbf{CH}NH_2CH_3$