InkChemistry

ANSWER BOOK

Proton and carbon-13 NMR spectroscopy

Organic analysis · Unit C19

AQA 3.3.15 · OCR A 6.3.2

16 questions · 49 marks

NAVY EDITION

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SECTION A · Short, 1 to 2 marks

A1 PCN-Q7

B1 the hydrogen of the O–H group exchanges with deuterium from D_2O, forming O–D. **B1** deuterium does not absorb in a proton NMR spectrum, so the O–H peak disappears. [2]

A2 PCN-Q10

M1 the ratio 2.4 : 1.6 : 2.4 simplifies to 3 : 2 : 3, a total of 8 parts. **A1** the formula has 8 hydrogens, so the environments hold 3, 2 and 3 hydrogens. [2]

A3 PCN-Q11

M1 four environments: the two equivalent CH_3 groups, the CH, the CH_2 and the OH. **A1** the areas are in the ratio 6 : 1 : 2 : 1, in any order that matches the groups. [2]

A4 PCN-Q19

B1 oxygen is electronegative and draws electron density away from the carbon and its hydrogens. **B1** the hydrogens are less shielded, so they absorb at a higher chemical shift. [2]

SECTION B · Standard, 3 to 5 marks

B1 PCN-Q1

B1 1,1-dichloropropane, $CHCl_2CH_2CH_3$: three different carbons, 3 peaks. **B1** 1,2-dichloropropane, $CH_2ClCHClCH_3$: three different carbons, 3 peaks. **B1** 1,3-dichloropropane, $ClCH_2CH_2CH_2Cl$: the two CH_2Cl carbons are equivalent, 2 peaks. **B1** 2,2-dichloropropane, $CH_3CCl_2CH_3$: the two CH_3 carbons are equivalent, 2 peaks. [4]

B2 PCN-Q3

B1 $CDCl_3$ has deuterium, ^2H, in place of ^1H, and deuterium does not absorb at the frequency of ^1H. **B1** so the solvent, present in large excess, gives no peak that could swamp or be confused with the sample's peaks. **B1** CCl_4 contains no hydrogen at all, so it gives no proton peak, though it dissolves only non-polar compounds well. **B1** glucose is polar and dissolves in D_2O, which has no ^1H, where it would barely dissolve in $CDCl_3$ or CCl_4. [4]

B3 PCN-Q5

B1 the n + 1 rule: hydrogens with n hydrogens on the adjacent carbons give a peak split into n + 1 lines. **B1** the CH_3 has two neighbouring hydrogens, on the CH_2, so it is a triplet. **B1** the CH_2 has four neighbouring hydrogens, three on the CH_3 and one on the CHO, so it is split into five lines. **B1** the CHO hydrogen, at 9.0–10.0 ppm, has two neighbouring hydrogens on the CH_2, so it is a triplet. [4]

B4 PCN-Q9

B1 four peaks, in the integration ratio 3 : 2 : 2 : 3. **B1** the OCH_3 is a singlet of 3H at about 3.7 ppm, in the RCOO–C–H range. **B1** the CH_2 bonded to the C=O carbon is a triplet of 2H at about 2.3 ppm, in the H–C–C=O range. **B1** the middle CH_2 is a sextet of 2H, with five neighbours, and the end CH_3 a triplet of 3H at about 0.9 ppm. [4]

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B5 PCN-Q16 **B1** butan-1-ol has four carbon environments, so four peaks is consistent with it. **B1** butan-2-ol and methoxypropane also have four carbon environments, so the count alone does not decide. **B1** methoxypropane would show two peaks in the C–O range, 50–90 ppm, whereas each butanol shows one. **B1** the proton NMR spectrum is needed to separate the butanols: butan-1-ol has a CH_2 triplet of 2H near 3.6 ppm, where butan-2-ol has a single CH hydrogen of area 1. [4]

B6 PCN-Q2 **B1** all its hydrogens, and all its carbons, are equivalent, so it gives a single sharp peak. **B1** its peak is at a lower shift than almost all organic compounds, so it does not overlap their peaks. **B1** it is inert and volatile, so it does not react with the sample and is easily removed afterwards; allow non-toxic for inert. [3]

B7 PCN-Q4 **B1** pentan-3-one, $CH_3CH_2COCH_2CH_3$, gives 3 peaks. **B1** 2,2-dimethylpropane, $C(CH_3)_4$, gives 2 peaks. **B1** butanone, $CH_3COCH_2CH_3$, gives 4 peaks. [3]

B8 PCN-Q6 **B1** the CH_3 hydrogens have two hydrogens on the adjacent carbon, so their peak is a triplet. **B1** the CH_2 hydrogens have three hydrogens on the adjacent carbon, so their peak is a quartet. **B1** by the n + 1 rule: 2 + 1 = 3 lines and 3 + 1 = 4 lines, with integration 3:2. [3]

B9 PCN-Q8 **B1** there are two hydrogen environments, the CH_3 and the COOH, with areas in the ratio 3 : 1. **B1** the CH_3 is bonded to the carbonyl carbon, which has no hydrogens, so n = 0 and it is a singlet near 2.1 ppm. **B1** the COOH hydrogen, at 10.0–12.0 ppm, exchanges rapidly and has no hydrogens on its neighbour, so it is also a singlet. [3]

B10 PCN-Q14 **B1** only about 1.1% of carbon atoms are carbon-13. **B1** so two carbon-13 atoms are almost never bonded to each other in one molecule, and one carbon peak is not split by another. **B1** the splitting that attached hydrogens would cause is switched off by the instrument as the spectrum is recorded. [3]

B11 PCN-Q15 **B1** both give one peak per chemical environment, of carbon and of hydrogen respectively, with shifts read from TMS. **B1** only proton NMR gives an integration ratio for the number of atoms in each environment; carbon-13 peak heights are not used. **B1** only proton NMR shows splitting, which gives the number of hydrogens on adjacent carbons. [3]

B12 PCN-Q18 **B1** a doublet of 6H and a septet of 1H show a $(CH_3)_2CH$ group, six equivalent hydrogens split by one neighbour and one hydrogen split by six. **B1** the CH at 4.1 ppm is at a high shift because it is on the carbon bonded to the electronegative chlorine atom. **B1** the compound is 2-chloropropane, $(CH_3)_2CHCl$. [3]

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