



2815/04 Methods of Analysis and Detection

January 2004

Mark Scheme

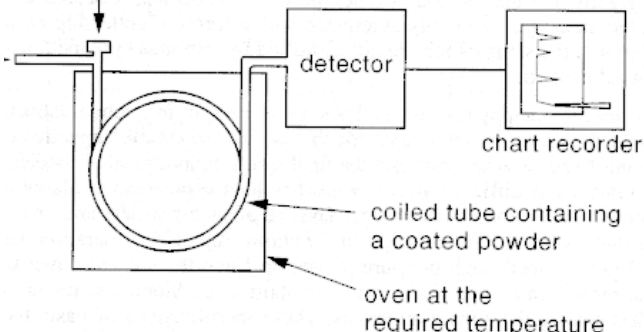
The following annotations may be used when marking:

X	=	incorrect response (errors may also be underlined)
^	=	omission mark
bod	=	benefit of the doubt (where professional judgement has been used)
ecf	=	error carried forward (in consequential marking)
con	=	contradiction (in cases where candidates contradict themselves in the same response)
sf	=	error in the number of significant figures

Abbreviations, annotations and conventions used in the Mark Scheme:

/	=	alternative and acceptable answers for the same marking point
;	=	separates marking points
NOT	=	answers not worthy of credit
()	=	words which are not essential to gain credit
<u> </u> (underlining)	=	key words which <u>must</u> be used
ecf	=	allow error carried forward in consequential marking
AW	=	alternative wording
ora	=	or reverse argument

Question	Expected Answers	Marks
1(a)	Electrons exist in discrete energy levels/quantised	1
(b)	(i) Electrons move from higher to lower energy levels	1
	This process emits energy as radiation	1
	(ii) $E = hf = 6.63 \times 10^{-34} \times 5.68 \times 10^{14} \times 6.02 \times 10^{23}$	1
	= 227000(226703) J mol ⁻¹ (units not required)	1
(c)	The lines converge because the energy levels get closer together towards the 'edge' of the atom	1
(d)	Any suitable example e.g. analysis of blood serum	1
	To include preparing a solution, making a suitable dilution	1
	Use of calibration / standards	1
		Total : 9

Question	Expected Answers	Marks
<p>2(a)</p> <p>(b)</p> <p>(c)</p>	<p>Reagent : 6M hydrochloric acid (accept conc.) NOT Restriction enzymes</p> <p>Conditions : Reflux / heat : long period of time / 24 hours</p> <p>(i) W</p> <p>(ii) Large [H⁺] / pH5 is acid the NH₂ group is protonated and no COO⁻ exist or shown on diagram</p> <p>Diagram below could score 3 if appropriately annotated.</p>  <p>Sample is injected into spectrometer Carrier gas carries it through the column Column is heated Samples are separated by their attraction for the column/partition Different components have different times of emergence Samples may be analysed by mass spectrometry or by using standards</p> <p>Correct terminology e.g. carrier gas, mobile phase, stationary phase QoWC</p>	<p>1</p> <p>1</p> <p>1</p> <p>1</p> <p>1</p> <p>1</p> <p>1</p> <p>1</p> <p>1</p> <p>1</p> <p>1</p> <p>1 max 5</p> <p>1</p>
<p>(d)</p>	<p>(i) R_f value is the distance moved by a component divided by the distance moved by the solvent</p> <p>(ii) Retention time is the time between injection and the emergence of a component</p>	<p>1</p> <p>1</p> <p>Total : 15</p>

Question	Expected Answers	Marks
3(a)(i)	M : M+1 = 52 : 2.3	1
	No. of carbon atoms = $\frac{2.3 \times 100}{52 \times 1.1} = 4.02 = 4$ carbons	1
(ii)	C ₄ H ₆ O	1
(b)	To give <i>m/e</i> 55 sample has lost 15 units hence CH ₃	1
	To give <i>m/e</i> 41 sample has lost 29 units hence C ₂ H ₅ or CHO (Penalise +ve ions -1)	1
(c)	<i>m/e</i> 41 could be C ₃ H ₅ ⁺ (NOT C ₂ HO ⁺ if CHO given in (b)) (check ecf from (a)(ii))	1
(d)	They will be equal	1
(e)	Any reasonable structure (check ecf from (a)(ii))	1
(f)	(i) CO and C ₂ H ₄	2 x 1
	(ii) Suitable calculation showing working to identify C ₂ H ₄	1
		Total : 11

Question	Expected Answers	Marks
4(a)	Mass peak is at m/e 72 If there is one O atom, the carbon and hydrogen atoms must add up to 56 mass units Since D is saturated this is C_4H_8 /or other valid method Hence D is C_4H_8O (alternative method using M:M+1 acceptable)	1 1 1
(b)	<p style="text-align: center;">Any 7 of the following points</p> I.r. spectrum shows a strong absorption at 1720 cm^{-1} which suggests C=O No other obvious absorptions in the i.r. (i.e. no C-O, no -OH) N.m.r shows protons in 3 environments Splitting patterns shows one group of 3 protons ($-CH_3$) at $\delta = 2.0$, and a C_2H_5 group C_2H_5 group is shown by triplet and quartet (or equiv.) Major peak in mass spectrum is at m/e 43 which could be $C_3H_7^+$ or CH_3CO^+ $C_3H_7^+$ does not fit the n.m.r. evidence OR more likely to be CH_3CO^+ (Other worthy points to be decided once scripts seen) D is $CH_3CH_2COCH_3$	1 1 1 1 1 1 1 1 1 Total : 10