Carbonyls, Carboxylic Acids & Derivatives

Mark Scheme

Level	International A Level
Subject	Chemistry
Exam Board	Edexcel
Topic	Chemistry Lab Skills 2
Sub Topic	Carbonyls, Carboxylic Acids & Derivatives
Booklet	Mark Scheme

Time Allowed: **93 minutes**

Score: **/77**

Percentage: **/100**

Grade Boundaries:

A*	A	B	C	D	E	U
>85%	'77.5%	70%	62.5%	57.5%	45%	<45%

<table>
<tr><th>Question Number</th><th>Acceptable Answer</th><th>Reject</th><th>Mark</th></tr>
<tr><td>1(a)</td><td>
<table>
<tr><th>Functional group</th><th>Test</th><th>Re</th><th></th></tr>
<tr><td rowspan="2">Alkene</td><td>Bromine (water / solution)</td><td>(Brown /orange /yellow to) colourless</td><td rowspan="2">(2)</td></tr>
<tr><td>OR
Acidified potassium manganate(VII)</td><td>(Purple to) colourless</td></tr>
<tr><td>Ketone
ALLOW carbonyl</td><td>2,4-dinitro phenylhydrazine

OR

Brady’s reagent

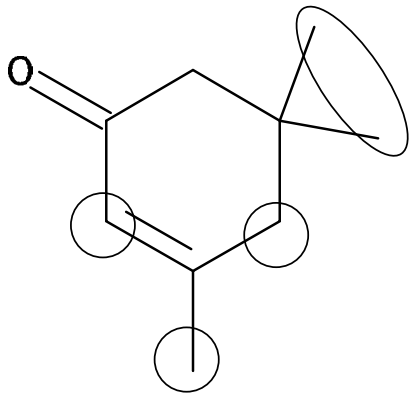
ALLOW (2,4-)DNP(H)</td><td>Orange / yellow / red and precipitate</td><td>(2)</td></tr>
</table>
For each functional group:
all three points scores 2;
functional group and test reagent alone scores 1;
test and result alone scores 1

Use of just C=C for alkene and / or just C=O for ketone max 3
IGNORE
Test for aldehyde group with negative result

No TE on incorrect test

If no other marks scored then identifying both functional groups scores 1
</td><td></td><td>4</td></tr>
</table>

Question Number	Acceptable Answer	Reject	Mark
1(b)(i)	Singlet because neither of the adjacent /neighbouring carbon atoms has a hydrogen / proton attached ALLOW Singlet because no adjacent /neighbouring / nearby hydrogen(s) / proton(s) ‘No splitting’ / one peak for ‘singlet’	Just ‘singlet’	1

Question Number	Acceptable Answer	Reject	Mark
1(b)(ii)	 **MP1** (standalone mark) The top methyl group proton environments fully identified **and** linked in some way **(1)** **MP2** The other three proton environments **(1)** MP2 may only be awarded if there are no errors on the rest of the molecule apart from the top methyl groups Likely errors: omission of a proton environment / incorrectly linked proton environments / additional protons ALLOW Any other labels of the proton environments		2

(Total for Question 1 = 7 marks)

Question Number	Acceptable Answers	Reject	Mark
2(a)	Sulfuric acid reacts very exothermically with water ALLOW The reaction with acid is exothermic OR (Sulfuric) acid will shoot out of container OR The reaction of water with (sulphuric) acid is vigorous/splashes OR Prevent splashing of acid	nitric acid Prevent splash alone	1

Question Number	Acceptable Answers	Reject	Mark
2(b)	(Pale/straw-coloured) yellow / brown / red-brown	Red/purple/blue/black/blue-black/orange	1

Question Number	Acceptable Answers	Reject	Mark
2(c)(i)	(Freshly prepared) starch (solution)		1

Question Number	Acceptable Answers	Reject	Mark
2(c)(ii)	(If starch is added too early) starch iodine complex formed (doesn't re-dissolve) ALLOW Iodide for iodine ALLOW (Black) solid/precipitate / complex forms OR Insoluble compound forms		1

Question Number	Acceptable Answers	Reject	Mark
2(c)(iii)	From blue-black to colourless ALLOW From blue / black/ dark blue/ deep blue to colourless	...to clear	1

Question Number	Acceptable Answers	Reject	Mark
2(d)(i)	24.1(0) 23.8(0) 23.55 23.45 (cm^3)		1

Question Number	Acceptable Answers	Reject	Mark
2(d)(ii)	The third and fourth / 23.55 and 23.45 (cm^3) **and** They are concordant OR Within ±0.2/0.1 (cm^3) IGNORE Anomalous		1

Question Number	Acceptable Answers	Reject	Mark
2(d)(iii)	23.5(0) (cm^3) ALLOW TE including second titre value, mean = 23.6(0) (cm^3)		1

Question Number	Acceptable Answers	Reject	Mark
2(d)(iv)	Correct answer 74.6% / 75% OR 74.9% (TE from 23.60 average titre) Ignore SF except 1SF With no working **(5)** Number of mol of thiosulfate $= \frac{23.50 \quad 0.200}{1000}$ (1) $= 4.70 \times 10^{-3}/0.00470$ **Second mark** EITHER Number of mol of iodine $= \frac{4.70 \times 10^{-3}}{2}$ $= 2.35 \times 10^{-3}/0.00235$ **AND** Number of moles of copper ion $= 2 \times 2.35 \times 10^{-3}$ (1) $= 4.70 \times 10^{-3}/0.00470$ in 10 cm^3 OR From equations amount of iodine is half amount of thiosulfate and amount of copper is twice amount of iodine, so amount of copper equals amount of thiosulfate for this mark Number of moles of copper in solid		5

	= $10 \times 4.70 \times 10^{-3}$ (1) = $4.70 \times 10^{-2}/0.0470$ Mass of copper in solid = $4.70 \times 10^{-2} \times 63.5$ (g) (1) = 2.9845 Percentage copper = $\frac{2.9845 \times 100}{4.00}$ = 74.6125 = 74.6% (1) Using 23.60 by averaging titres 2, 3 and 4 $4.72 \times 10^{-3}/0.00472$ (1) 2.36×10^{-3}0.00236 AND $4.72 \times 10^{-3}/0.00470$ (1) $4.72 \times 10^{-2}/0.0470$ (1) 2.9972 (1) 74.9% (1) Answers greater than 100% max 3		

Question Number	Acceptable Answers	Reject	Mark
2(d)(v)	**irst Mark** Uncertainty in titre value: $\frac{2 \times 0.05}{23.55} \times 100 =$ (±)0.42/0.425/0.4246% (1) **Second Mark** Uncertainty in the mass measurement: $\frac{2 \times 0.005 \times 100}{4.0} = (\pm)0.25\%$ **OR** $\frac{1 \times 0.005 \times 100}{4.0} = (\pm)0.125\%$ so it would / would not be worth using a 3 dp balance (1) Ignore SF including 1 SF		2

(Total for Question 2 = 15 marks)

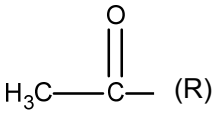
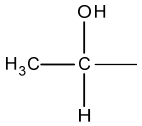
Question Number	Acceptable Answers	Reject	Mark
3(a)(i)	Red /orange / yellow **and** precipitate ALLOW Bright and correct colour Combinations of these colours AND Solid / crystals /ppt /ppte / precipitate (ie recognisable spelling for 'precipitate')	Colour alone Pale yellow Other colour combinations eg red / brown	**1**

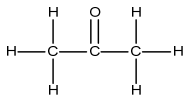
Question Number	Acceptable Answers	Reject	Mark
3(a)(ii)	Aldehyde **OR** ketone (both needed) Either order. ALLOW Carbonyl (compound/group) RCOR IGNORE contains C=O or other formulae		**1**

Question Number	Acceptable Answers	Reject	Mark
3(b)(i)	**First mark** Solutions: (aqueous) silver nitrate / $AgNO_3$ **(1)** **Second mark** (aqueous) ammonia / NH_3 / NH_4OH **(1)** Reagents in any order. IGNORE NaOH **NOTE** Ammoni(a)cal silver nitrate scores first 2 marks BUT Silver diam(m)ine / $[Ag(NH_3)_2]^+(NO_3^-)$ scores only 1 mark **Third mark** dependant on **any** silver salt and ammonia / ammoni(a)cal silver nitrate / silver diam(m)ine / $[Ag(NH_3)_2]^+$ Condition: Clean test tubes / warm / heat ALLOW heat to below 50^0C **(1)** **Fourth mark** stands alone - independent Positive result: Silver mirror / grey or black precipitate ALLOW Silver (alone) silver solid / silver ppt / ppte / metallic silver OR Silver mirror solution **(1)**	Other silver salts Silver / Ag^+ ions Silver compounds Ammonium / NH_4^+ ions Anything else eg nitric acid Shake vigorously Heat under reflux $20\text{-}25^0C$ Silver colour / silver coloured solution	**4**

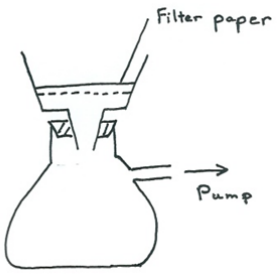
Question Number	Acceptable Answers	Reject	Mark
3(b)(ii)	Ketone ALLOW Keytone OR Carbonyl group in a ketone OR C=O in a ketone ALLOW ketone with additional correct general, displayed / structural formulae	Just 'carbonyl group' Just 'C=O'	**1**

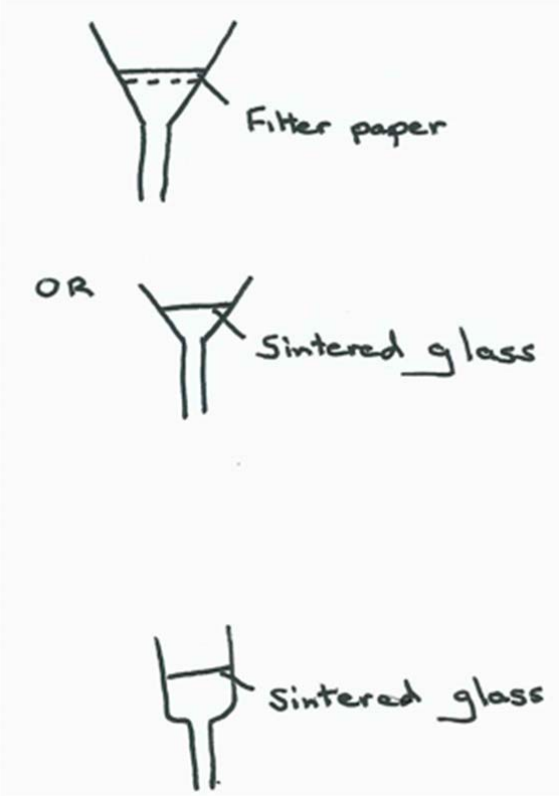
Question Number	Acceptable Answers	Reject	Mark
3(c)(i)	(pale) yellow precipitate / solid / crystals Qualification of yellow is allowed like pale, light, creamy, or bright, but not dark. **Both** colour **and** state ALLOW 'Cloudy yellow' alone OR Recognisable spelling or abbreviations for precipitate like ppt / ppte / percipitate IGNORE antiseptic smell	Dark yellow	**1**

Question Number	Acceptable Answers	Reject	Mark
3(c)(ii)	Methyl ketone / CH_3CO (group) OR Contains  OR Methyl attached to a carbonyl (group)/C=O OR It is a 2-one ketone	 Secondary alcohol Functional group on second carbon atom	**1**

Question Number	Acceptable Answers	Reject	Mark
3(d)	**First mark** One hydrogen / proton environment OR One kind of hydrogen / proton ALLOW Hydrogen ion for proton **(1)** **Second mark** (Singlet so) no neighbouring hydrogens (on adjacent carbons) OR Chemical shift is correct for a ketone **(1)** **Third mark**  Hydrogens must be shown, but methyl groups do not have to be displayed. **(1)** ALLOW Correct structural or skeletal formula	Just 'it is a singlet' Just 'not split(ted) by any other hydrogen' Chemical shift is the same as a methyl group (alone) Chemical shift is at 2.2 (alone)	**3**

Question Number	Acceptable Answers	Reject	Mark
3(e)(i)	Faster **(1)** Solid / precipitate / crystals are drier OR more solvent / solution / filtrate removed OR Reverse argument for normal filtering **(1)** IGNORE More efficient / more effective / increases yield / more pure Reducing pressure reduces boiling temperature		**2**

Question Number	Acceptable Answers	Reject	Mark
3(e)(ii)	 **First mark** Buchner / side-armed flask **(1)** **Second mark** Side arm connected to pump **and** bung/rubber around neck of funnel OR Quickfit flask funnel shown with clear sealed join and pump labelled  **(1)** ALLOW aspirator for pump, drawings of tap pump IGNORE Blocked outlet to pump **Third mark** Buchner funnel with **flat** filter paper The filter paper must be labelled OR be drawn flat **and** clearly shown	Heated flask Large gap between funnel and flask Pressure pump (alone) Suction (alone) Filter paper which goes up the sides of the funnel Fluted filter paper	**3**

	above the pores of the funnel OR Hirsch funnels see diagrams below  Notice sintered / fitted glass does not need a filter paper IGNORE Blocked outlet to funnel **(1)** Notice heating of funnel / solvent can be ignored		

Question Number	Acceptable Answers	Reject	Mark
3(e)(iii)	**First mark** Dissolve in / mix with MINIMUM / SMALL volume / amount of HOT ethanol / solvent (to dissolve most of the solid / make a saturated solution) **(1)** **Second mark** Filter HOT (to remove insoluble impurities)# AND Cool / allow to crystallize **(1)** **Third mark** Filter (under reduced pressure) (to remove soluble impurities)# AND Wash with COLD / minimum volume of solvent **(1)** **Fourth mark** **Dry** between filter papers / with **paper** towel / in desiccator Both dry **and** method of drying ALLOW Use of **cool / warm** oven OR oven at specified temperature below 100°C Use of hair drier / electric hand drier **(1)** **Note** penalties may be applied: *Penalise use of water only once #If impurities are wrong way round penalise only once Both these penalties can be applied to reduce the mark by 2	Water as solvent* Filter alone (Wash) with water* Drying agents unless in desiccator	**4**

Question Number	Acceptable Answers	Reject	Mark
3(e)(iv)	Measure the melting temperature **(1)** IGNORE References to sharp melting temperature Compare with literature / data (book) / known value **(1)** Second mark conditional on melting temperature mark IGNORE IR spectroscopy and check fingerprint region	Boiling temperature alone Chromatography and compare R_f values If melting temperature is clearly for propanone and not propanone derivative.	**2**

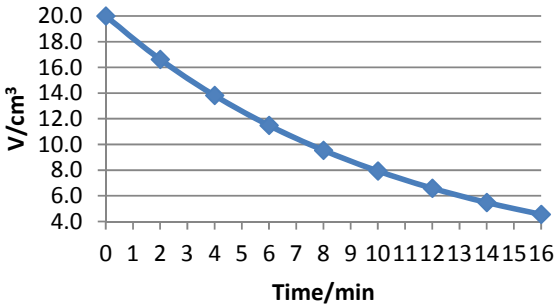
Total for Question 3 = 23 marks

Question Number	Acceptable Answers	Reject	Mark
4(a)	To quench/stop/slow the reaction ALLOW freeze IGNORE: to reduce reactivity exothermic reaction/reaction gives our heat		**1**

Question Number	Acceptable Answers	Reject	Mark
4(b)	Phenolphthalein **and** one of the following: (Indicator) range /colour change corresponds to steep change in pH. OR (Indicator) range /colour change corresponds to vertical/steep region of pH titration curve OR (change in) pH range 7.1 - 12/ above 7 OR (change in) pH range of methyl orange is below 7/ range 6.9 - 3 OR pK_{in} is greater than 7, or any number greater than 7 and less than 14. (correct value is 9.3) OR changes colour at/near equivalence point OR carboxylic acid is a weak acid OR weak acid – strong base titration	strong acid – strong base titration	**1**

Question Number	Acceptable Answers	Reject	Mark
4(c)(i)	Catalyst/hydrochloric acid/HCl reacts with the sodium hydroxide ALLOW Acid in (initial) solution		1

Question Number	Acceptable Answers	Reject	Mark
4(c)(ii)	(More) (carboxylic) acid is formed	More product is formed	1

Question Number	Acceptable Answers	Reject	Mark
4(d)	 Axes labelled with quantity and **units** Note unit for time must be min not s **(1)** Points correctly plotted using at least half the graph paper in both dimensions Smooth curve through points **(1)** Allow one mis-plot provided curve is smooth Two half lives found in range 7.2 - 7.8 (mins) Ignore seconds for units (half lives need not be successive) **(1)** First order (this mark depends on two close values in third mark) **(1)** Note: If second half life is 15.2 etc. , then 3rd and 4th marks lost		4

Question Number	Acceptable Answers	Reject	Mark
4(e)	Orange to green/blue/brown Both colours required IGNORE: Qualifications of colour, e.g. dark green	Combinations of blue and green	1

Question Number	Acceptable Answers	Reject	Mark
4(f)(i)	**Correct** names or formulae are acceptable, e.g. sodium hydrogencarbonate (allow sodium bicarbonate)	Indicators sodium hydroxide	2

Reagent **(1)**	Observation **(1)**
Na_2CO_3(aq)/$NaHCO_3$ (aq)/$CaCO_3$((s))	Effervescence/ fizzing
Allow solid Na_2CO_3/$NaHCO_3$	Allow: Testing gas with limewater which turns cloudy; Neutralises large volume
PCl_5	Misty/steamy/white fumes
Alcohol (+ mineral acid)	Fruity smell
Na / Mg	Effervescence/ fizzing

$NaCO_3$
Loses reagent mark

$LiALH_4$

White smoke

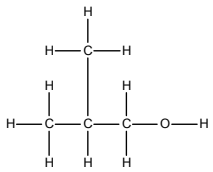
Just ester formed

Question Number	Acceptable Answers	Reject	Mark
4(f)(ii)	2,4-dinitrophenylhydrazine/2,4-DNP(H) /DNP(H)/Brady's reagent **(1)** red/orange/yellow **precipitate** **(1)** Or Iodine and sodium hydroxide **(1)** Yellow precipitate **(1)** Ignore references to antiseptic smell	Colour only Colour only	2

	Ignore references to Tollens, Bendict's, Fehling's and result (ie no TE)		

Question Number	Acceptable Answers	Reject	Mark
4(g)	Primary ALLOW: 1° OR $-CH_2OH$ IGNORE: Names		1

Question Number	Acceptable Answers	Reject	Mark
4(h)(i)	**4 different H/hydrogen/proton environments**	4 different kinds of H 4 different environments	1

Question Number	Acceptable Answers	Reject	Mark
4(h)(ii)	 accept $-OH/CH_3$ ALLOW correct skeletal formula correct structural formula e.g. $CH_3CH(CH_3)CH_2OH$ OR Part structural, part displayed formula OR	Molecular formula OHC/O-H-C where there are clearly two bonds to hydrogen	1

	Vertical bond to OH wherever it finishes		

Question Number	Acceptable Answers	Reject	Mark
4(h)(iii)	H on the OH group OR OH group ALLOW TE for OH/H on wrong isomer	O alone COH	1

(Total for Question 4 = 17 marks)

Question Number	Acceptable Answers	Reject	Mark
5(a)	Hazard: flammable Precaution: keep away from naked flames/ use electrical heating/mantle or use water bath **(1)** Hazard: corrosive Precaution: wear gloves **(1)** Precaution must relate to appropriate hazard 2 correct hazards with no precautions **(0)** IGNORE Use of fume cupboard	Keep away from sources of heat Avoid direct heat	2

Question Number	Acceptable Answers	Reject	Mark
5(b)	Round-bottom/pear shape flask with vertical reflux condenser , drawn or labelled **(1)** Condenser jacket drawn at with water in at bottom, out at top **(1)** Heating source e.g. heating mantle/electric heater/water bath/ oil bath **(1)** ALLOW Water/oil bath heated by Bunsen burner Fully correct distillation apparatus **(1 max)** If both reflux and distillation diagrams drawn, then 2 marks max	Conical flask Bunsen burner Arrow (labelled or unlabelled) Closed apparatus/ large air gaps in wrong places loses an additional mark	3

Question Number	Acceptable Answers	Reject	Mark
5(c)	Prevents superheating/ localised heating ALLOW: Violent boiling OR Promotes smooth/even/uniform boiling OR Promotes smooth/even/uniform heating IGNORE prevents vigorous reactions/(large)bubbles/splashing	Just "stops bumping" Just: Violent reaction Just: Prevent explosion Just: Prevent mixture rising up condenser	1

Question Number	Acceptable Answers	Reject	Mark
5(d)	To remove/react with/neutralize the (unreacted)(ethanoic) **acid**		1

Question Number	Acceptable Answers	Reject	Mark
5(e)	Anhydrous sodium sulfate **(1)** Others would react with/decompose product/ester Sodium sulfate does not react with/ decompose product/ester **(1)** **Second mark depends on first**	Others "too strong" Easiest to separate The only neutral one	2

Question Number	Acceptable Answers	Reject	Mark
5(f)	Start 139-141^{o}C End 143-145^{o}C Both required for the mark	Single temperature	1

Question Number	Acceptable Answers	Reject	Mark
5(g)(i)	Mass 3-methylbutan-1-ol = 0.81 x 10.0 **(1)** = 8.10(g) Mol 3-methylbutan-1-ol = 8.10/88.0 = 0.09204545 Mol product = 0.09205 (0.0920 and 0.092 are both allowed for this step) **(1)** Mass of product = 0.09205 x 130.0 = 12.0 (g) to 3 sf **(1)** Correct answer with no working 3 marks **(3)** With consequential marks, the last mark is lost if the candidate's data is not rounded correctly to 3 sf. 0.09 gives 11.7 **(2 max)** 0.092 gives 12.0 **(3)**	0.09/0.0921	3

Question Number	Acceptable Answers	Reject	Mark
5(g)(ii)	**EITHER** % yield = 9.45/(ans to 4(g)(i)) x 100 **(1)** = correct value **(1)** N.B. correct value: % yield = 9.45/12.0 x 100 = 78.75% = 79% **OR** $\frac{9.45}{130}$ = 0.07269 (mol) **(1)** $\frac{0.07269}{0.09205} \times 100$ = 78.9680% = 79% **(1)** Accept any answer that rounds to 79 to two sf Allow TE from (i) for full credit unless greater than 100% in which case (**1max**)		2

(Total for Question 5 = 15 marks)