OCR
RECOGNISING ACHIEVEMENT

GCE

Chemistry

Advanced GCE **2816/03**

Practical

Mark Scheme for June 2010

Oxford Cambridge and RSA Examinations

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This mark scheme is published as an aid to teachers and students, to indicate the requirements of the examination. It shows the basis on which marks were awarded by Examiners. It does not indicate the details of the discussions which took place at an Examiners' meeting before marking commenced.

All Examiners are instructed that alternative correct answers and unexpected approaches in candidates' scripts must be given marks that fairly reflect the relevant knowledge and skills demonstrated.

Mark schemes should be read in conjunction with the published question papers and the Report on the Examination.

OCR will not enter into any discussion or correspondence in connection with this mark scheme.

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ADVICE TO EXAMINERS ON THE ANNOTATION OF SCRIPTS

1. Please ensure that you use the **final** version of the Mark Scheme.
   You are advised to destroy all draft versions.

2. Please mark all post-standardisation scripts in red ink. A tick (✓) should be used for each answer judged worthy of a mark. Ticks should be placed as close as possible to the point in the answer where the mark has been awarded. The number of ticks should be the same as the number of marks awarded. If two (or more) responses are required for one mark, use only one tick. Half marks (½) should never be used.

3. The following annotations may be used when marking. No comments should be written on scripts unless they relate directly to the mark scheme. Remember that scripts may be returned to Centres.
   - o x = incorrect response (errors may also be underlined)
   - o ^ = omission mark
   - o bod = benefit of the doubt (where professional judgement has been used)
   - o ecf = error carried forward (in consequential marking)
   - o con = contradiction (when candidates contradict themselves in the same response)
   - o sf = error in the number of significant figures

4. The marks awarded for each part question should be indicated in the margin provided on the right hand side of the page. The mark total for each question should be ringed at the end of the question, on the right hand side. These totals should be added up to give the final total on the front of the paper.

5. In cases where candidates are required to give a specific number of answers, (e.g. 'give three reasons'), mark the first answer(s) given up to the total number required. Strike through the remainder. In specific cases where this rule cannot be applied, the exact procedure to be used is given in the mark scheme.

6. Correct answers to calculations should gain full credit even if no working is shown, unless otherwise indicated in the mark scheme. (An instruction on the paper to 'Show your working' is to help candidates, who may then gain partial credit even if their final answer is not correct.)

7. Strike through all blank spaces and/or pages in order to give a clear indication that the whole of the script has been considered.

8. An element of professional judgement is required in the marking of any written paper, and candidates may not use the exact words that appear in the mark scheme. If the science is correct and answers the question, then the mark(s) should normally be credited. If you are in doubt about the validity of any answer, contact your Team Leader/Principal Examiner for guidance.

Abbreviations, annotations and conventions used in the Mark Scheme	/	= alternative and acceptable answers for the same marking point
	;	= separates marking points
	NOT	= answers which are not worthy of credit
	( )	= words which are not essential to gain credit
	___	= (underlining) key words which **must** be used to gain credit
	ecf	= error carried forward
	AW	= alternative wording
	ora	= or reverse argument

2816/03 Mark Scheme
Practical Examination: June 2010

PLAN: Skill P [16 marks out of 18 available]

Identification tests: 14 marks available

2 marks are available for each of the six compounds.

Each tick awarded must be accompanied by its code letter.

There are **four** marking points (bullets) for each compound

- Two marks are awarded if all four bullets are correct.
- One mark is awarded if two or three bullets are correct.

Mark the tests in a way which maximises the mark awarded.

The mark scheme below shows the most common tests only. Other tests may also be suitable.

When marking equations

- Accept any formula which shows both the functional group and its position
  (eg Allow C_3H_7OH but not C_3H_8O for propan-1-ol, but not C_3H_7OH for methylpropan-2-ol)
- Subtract one scoring bullet for poor ICT (eg C2H5OH) on **one** occasion only.
- Penalise a trivial error (eg missing out one H atom) once only
- Penalise a repeated error (eg C_2H_5 for "prop") once only
- Ignore state symbols

Some candidates may add sodium metal in order to divide the six compounds into two groups of three. Then they will attempt to distinguish between the compounds within each group of three. The marks for using sodium can then be awarded for the **third** compound discussed. Some will begin by using 2,4-DNP in a similar way to "eliminate" two carbonyl compounds

If a test reagent is used twice, the test that would score the lower mark will be awarded 0.
A negative test scores 0, **except** for the $K_2Cr_2O_7$ test for a tertiary alcohol, which can score 1.

If the test **reagent** is unsuitable, 0 marks out of 2 will normally be awarded.
How a response is marked will depend of the sequence in which the tests are described.

X Flow chart (2 marks)

A clear flow chart that would enable five or six compounds to be identified. [1]

The identity of the test reagent and the outcome of the test are required
The flow chart must show positive and negative branches throughout.

The Plan has a logical narration, explaining the sequencing of each test [1]

A good flow chart will normally earn this mark in addition to X1.
*This mark **not** awarded if there is a list of tests with no explanation of the order.*

A 1-iodopropane (2 marks)

- Heat with NaOH, then neutralise (or add HNO_3) then add **silver nitrate**
  or add silver nitrate dissolved in ethanol
- [Pale] yellow precipitate formed
- Precipitate not soluble in [excess aqueous] ammonia
- Equation: $Ag^+ + I^- \rightarrow AgI$

B Propanoic acid (2 marks)

- Add **magnesium *or*** a **named metal carbonate *or*** suitable named indicator
- Observation: fizzing/bubbles/effervescence ***or*** appropriate colour change for indicator
  Do not accept "gas" or the name of the gas as an observation
- Positive test for gas (H_2 ***or*** CO_2) described
- Equation: eg $Mg + 2C_2H_5COOH \rightarrow (C_2H_5COO)_2Mg + H_2$ (***or*** ionic equation)

Or

- Add **ethanol/methanol *and*** heat in presence of concentrated sulphuric acid
- Sweet/fruity smell produced (when mixture poured into cold water)
- Correct name of ester produced
- Equation: $C_2H_5COOH + CH_3OH \rightarrow C_2H_5COOCH_3 + H_2O$

C Propanal (2 marks)

- Warm with ammoniacal silver nitrate / **Tollen's reagent**
  or warm with Fehling's solution(s) / add Benedict's reagent
- Silver mirror/black precipitate formed
  or brown/red precipitate obtained
- Propanal is a reducing agent ***or*** a correct redox statement about the reaction
  or Cu_2O identified as the precipitate in Fehling's test
- Equation: $C_2H_5CHO + [O] \rightarrow C_2H_5COOH$ ***or*** $Ag^+ + e^- \rightarrow Ag$

Or

- Add **Schiff's reagent**
- Goes red
  Maximum 1 mark for this test

D Propanone (2 marks)

- Add a solution of **2,4-dinitrophenylhydrazine** (***or*** Brady's reagent ***or*** "2,4-DNP")
  "Add 5 cm^3"(owtte) implies that a solution was used.
- Orange/yellow precipitate obtained
- Melting point of solid determined ***and*** compared to literature value
  or this is a condensation reaction ***or*** reference to formation of hydrazone
  or specific statement that this is a test for any carbonyl compound (aldehyde + ketone)
- Equation: $CH_3COCH_3 + C_6H_3(NO_2)_2N_2H_3 \rightarrow C_6H_3(NO_2)_2NHN=C(CH_3)_2 + H_2O$

Or

- Add concentrated aqueous sodium hydroxide followed by excess iodine
  or potassium iodide with excess sodium chlorate(I)
  *Accept **either** "concentrated" **or** "excess" as sufficient detail of conditions*
- Yellow precipitate formed (of CHI_3)
- Test for CH_3CO- group
- Equation: $CH_3COCH_3 + 3I_2 + 4NaOH \rightarrow CHI_3 + CH_3COONa + 3NaI + 3H_2O$

E Propan-1-ol (2 marks)

- Heat with acidified **potassium dichromate** (VI).
  Do not penalise absence of the oxidation state. Allow $KMnO_4$, but insist on the "VII"
- Colour change to green/turquoise solution
  or colour change from orange to green *(no state needed)*
- [$Cr_2O_7^{2-}$ is] reduced to Cr^{3+} *(redox statement **and** reference to Cr^{3+} needed))*
- Equation: $C_3H_7OH + [O] \rightarrow C_2H_5CHO$
  or $C_3H_7OH + 2[O] \rightarrow C_2H_5COOH + H_2O$
  or $3C_3H_7OH + Cr_2O_7^{2-} + 8H^+ \rightarrow 3C_2H_5CHO + 2Cr^{3+} + 7H_2O$

Or

- Add small piece(s) of **sodium** [metal]
  some reference to small quantity needed
- Fizzing observed [faster than for a tertiary alcohol]
- Hydrogen produced
- Equation: $2Na + 2C_3H_7OH \rightarrow H_2 + 2C_3H_7O^-Na^+$ *(charges not required)*

Or

- Add phosphorus pentachloride to dry sample of unknown liquid
  Alternative chlorinating reagents are acceptable: "dry" (owtte) must be stated
- Steamy/acidic fumes produced
- Test for –OH group ***and*** reaction produces hydrogen chloride
- Equation: $C_3H_7OH + PCl_5 \rightarrow HCl + POCl_3 + C_3H_7Cl$

F Methylpropan-2-ol (2 marks)

- Add concentrated hydrochloric acid with zinc chloride
- Goes cloudy quickly [relative to propan-1-ol]
- Reference to nucleophilic substitution ***or*** Lucas test named
- Equation: $(CH_3)_3COH + HCl \rightarrow (CH_3)_3CCl + H_2O$

Or

- Heat with acidified potassium dichromate (VI).
  Do not penalise absence of the oxidation state. Allow $KMnO_4$, but insist on the "VII"
- No reaction (or no colour change) since this is a tertiary alcohol
  Only one mark is available for this test, since it is a negative one.

S Safety and Sources etc – 4 marks

S1 A significant hazard stated for <u>one</u> of the materials or tests ***and*** safety
Appropriate safety must be more than "safety spectacles" or "lab coat" **[1]**

Possibilities include:

- *corrosive nature of hot alkalis*
- *flammability/ corrosiveness of using sodium metal*
- *flammability of alcohols, carbonyls or esters when heated*
- *toxicity of 2,4-DNP*

S2 Two secondary sources quoted in the text ***or*** as footnotes ***or*** at end of plan. **[1]**

Book reference(s) must have chapter or page numbers
Internet reference(s) must go beyond the first slash of web address
*Accept **one** <u>specific</u> reference to a "Hazcard" (by name or number)*
*Allow one reference to a specific past paper (but **not** to teaching notes etc)*

S3 **QWC**: text is legible ***and*** spelling, punctuation and grammar are accurate **[1]**

*Award S3 if there are fewer than **six** errors in legibility, spelling, punctuation or grammar.*

- *Penalise a specific error only once: mis-spelling of the same word is one error.*
- *[Repeated] failure to put a capital letter at start of sentences is one error.*
- *IT: [Repeated] lack of superscripts ("cm3") counts as one error.*
- *Other types of repeated IT error are also penalised once.*
- *Don't penalise an error if penalised in a balanced equation.*
- *Do not award S3 if more than **five** different words in which the letters are illegible.*
- *Bullet pointed methods/ listed descriptions are acceptable*

S4 **QWC**: information is organised clearly and coherently **[1]**

Can you say "yes" to all three of the following questions?

- *Is a word count given and within the limits 450 – 1050 words?*
  Accept a total word count or any word numbering in the margin
  Photocopied/downloaded material must be counted within the total
- *Is scientific language used correctly – allow one error without penalty.*
  Is there a terminology error
  - eg "burn" for "heat" or "hydrous" for "hydrated"?
  Is there an incorrect chemical formula in the text (eg $NaCO_3$)?
  Is there an incorrect equation not required by the mark scheme?
  If units are quoted are they [normally] correct? (eg mol dm^3)
- *Is the Plan free of excessive repetition or verbosity?*

Enter the total mark for Skill P on the front cover of the Test booklet.

Practical Test, Part B

Part 1

Page 3 (Part 1 – Skill I) [14 marks]

Put a page total at the bottom of each of pages 3 - 5, not ringed.
The section total (out of 30) should be ringed at the bottom of page 5
No "negative" marks are awarded in any marking sub-section

Presentation of weighings **[1]**

- Readings clearly labelled
- Both weighings **and** the difference in mass all quoted consistently to 2 or 3 d.p.
- Unit (g) given for both weighings
- Subtraction correct

Presentation of titration data **[2]**

Check the following bullet points.
All 8 bullets correct = 2 marks; 7 correct = 1 mark.

- Correctly labelled table (initial, final and difference - *aw*) used to record burette data
- A table grid, showing at least three grid lines, **must** be drawn.
- All **accurate** burette data and titres (including 0.00 cm^3 at start) are quoted to 0.05 cm^3
- All subtractions are correct
- Units (cm^3 *or* ml) are shown in the header for each row/column ***or*** for each reading
- Readings from three or more titrations are recorded
- No inverted sets of readings
- No readings recorded greater than 50 cm^3

Recording the start volume as "50.00 cm^3" scores 0 out of 2.
However, assume that the candidate meant to write "0.00" when assessing accuracy.

Self-consistency of titres **[2]**

If any two **accurate** titres agree within 0.10 cm^3, award 1 mark
*Assume that the **first** reading is the trial when assessing this mark.*

If the two **ticked** titres agree within 0.20 cm^3, award 1 mark.
If the candidate has altered his/her figures to "fiddle", award 1 mark maximum

Mean titre correctly calculated **[1]**

- *The mean should normally be calculated using the [two closest] accurate titres.*
  ***However** the trial <u>may</u> be used if it is closer than one of the accurate readings.*
- *All three readings may be used if all are within 0.10 cm^3*
- *The mean must be correctly quoted **either** to 2 d.p **or** to 0.025/0.075*
  If "rounding" is attempted, it must be done correctly.

Accuracy – [7 marks]

- *Write down the supervisor's mean titre, rounded to 0.05 cm^3 and ringed.*
  Check that supervisor's subtractions are correct.
- *For a mass of **1.50 g**, the mean titre should be approximately **25.00 cm^3***
  In the absence of results from a supervisor, use the above as default values.

- *The candidate's **own** mean should normally be used for assessment of accuracy.*
  *However, if there was a subtraction error or an **obvious** error in selecting which titres to use to calculate the mean, re-calculate the mean using the candidate's two [closest] **accurate** readings (ignore the first reading, however it was labelled) and use this corrected value to assess accuracy.*
  Use candidate's mean to nearest 0.05 cm^3

- ***Calculate the corrected candidate's mean titre (T) using the formula below***

  $$T = \text{candidate's mean titre} \times \frac{\text{supervisor's mass}}{\text{candidate's mass}}$$

- ***Compare supervisor's mean titre with candidate's corrected mean titre (T).***
  ***Put "δ = __" on the script** to show the difference between the two mean titres.*

- ***Use the conversion chart below to award the mark out of 7 for accuracy.***

Candidate's mean titre is within **1.50 cm^3** (incl) of supervisor's mean titre	**[1 mark]**
Candidate's mean titre is within **1.00 cm^3** (incl) of supervisor's mean titre	**[2]**
Candidate's mean titre is within **0.80 cm^3** (incl) of supervisor's mean titre	**[3]**
Candidate's mean titre is within **0.60 cm^3** (incl) of supervisor's mean titre	**[4]**
Candidate's mean titre is within **0.40 cm^3** (incl) of supervisor's mean titre	**[5]**
Candidate's mean titre is within **0.30 cm^3** (incl) of supervisor's mean titre	**[6]**
Candidate's mean titre is within **0.20 cm^3** (incl) of supervisor's mean titre	**[7 marks]**

Spread penalty

("Spread" relates to the titres actually used by the candidate to calculate his/her mean)

If the titres have a spread of more than 0.30 cm^3, deduct 1 mark from accuracy mark.
If the titres have a spread of more than 0.60 cm^3, deduct 2 marks from accuracy mark.
If the titres have a spread of more than 1.00 cm^3, deduct 3 (max) from accuracy mark.

Accuracy marks should be indicated as figures on the script: for example " +5 -1"

Safety [1 mark]

One reason, relating to the harmful/irritant nature of materials ***or*** to hot apparatus ***and*** one corresponding precaution

- Use clips/tongs to remove hot conical flask from tripod
- Wear gloves ***or*** safety spectacles to protect from harmful/irritant/hot solutions
- Use of pipette filler to prevent harmful ethanedioic acid from entering mouth

Page 4 - 8 marks

M_r of $KMnO_4$ = 158 [1]
Give this mark if all relative atomic masses are listed and final answer is correct

$[KMnO_4] = {}^{3.00}/_{158} = 0.0190$ mol dm^{-3} [1]
No ecf mark from wrong M_r ***within*** *this part.*
This answer must be quoted to 3 sig fig

(b) $n(KMnO_4)$ in mean titre = ${}^{0.0190 \times titre}/_{1000}$ *(**method** mark)* [1]

Candidate correctly calculates $n(KMnO_4)$ in mean titre [1]
n should work out to approximately 0.0005 mol

(c) Correct half-equation: $MnO_4^- + 8H^+ + 5e^- \rightarrow Mn^{2+} + 4H_2O$ [1]

(d) Combination of the two ionic half-equations [1]
$2\ MnO_4^- + 5(COOH)_2 + 6H^+ \rightarrow 10\ CO_2 + 2\ Mn^{2+} + 8\ H_2O$
$16H^+$ on left with $10H^+$ on right loses the equation mark
"Halved" equation ($MnO_4^- + 2.5H_2C_2O_4$ +..... scores both marks in (d)

Indication that 2:5 mole ratio = 1:2.5 ***or*** explains division by 2 to work out mole ratio ***or*** working shown "equating" the electrons when combining the half equations [1]
*Mark ecf from (c) but, in this case, only award **one** mark (max) in (d)*

(e) $n(\mathbf{H_2X})$ in 25 cm^3 = 2.5 x $n(KMnO_4)$ = 2.5 x "***b***" [1]
Method *mark for correct use of the mole ratio*

Page 5 - 8 marks

(f) $n(\mathbf{H_2X})$ in 250 cm^3, = 10 x "***e***" [1]
Method *mark for scale-up from 25 to 250 cm^3.*

(g) M_r of hydrated acid = ${}^{\text{mass of acid weighed}}/_{\text{moles of acid in 250 cm3}}$ [1]
Method *mark is for substitution of correct figures into the expression above*

*M*r of hydrated acid calculated correctly. [1]
Answer should be 126 but may work out rather lower.
Answer must be integral (since question asks for 3 sig fig in this answer)

*M*r of anhydrous acid = 90 [1]
Award this as a free-standing mark if "90" is seen

x = ${}^{Mr - 90}/_{18}$ *(**Method** mark for correct numerical expression)* [1]
Alternative sequence of calculation steps is also valid
Mass anhydrous acid used (2 marks) then mass water (1) then mole ratio (1)

x correctly calculated and expressed as integer ***or*** to one decimal place [1]

Final answer is shown as x = 2 (integral), with **all** working completely correct [1]
No ecf awarded on this "stretch and challenge" mark

Answers to (b), (e) and (f) are all quoted to **three** sig fig [1]

Pages 6 and 7: Evaluation - 14 marks

There are 15 marking points but the maximum mark available is 14

(a) - 5 marks

Work out mass of anhydrous acid, reading 3 minus reading 1 [1]

Work out mass of water driven off, reading 2 minus reading 3 [1]
Specific reference to the appropriate readings is required for both marks

Calculate number of moles of each chemical..... [1]

..... by dividing each mass by the respective M_r (90 and 18 must be quoted) [1]
Give 1 mark (out of 2) for working out the number of moles of either material

Calculate the mole ratio, $^{n(water)}/_{n(acid)}$ to give the value of **x** [1]
An alternative method via the M_r of the hydrated acid is valid for last two marks

(b) - 4 marks *(mark the "reliability" stand **and** the best second strand)*

Repeat experiment ***and*** use mean values/ ignore anomalies .... [1]

..... Consistent readings indicate <u>reliability</u> [1]

Heat to constant mass*....("longer heating" can score second mark)* [1]

..... To ensure that all of the water (of crystallisation) has been driven off [1]

Allow residue to cool before re-weighing .. [1]

..... For safety reasons ***or*** convection currents affect weighings [1]

Heat gently at first.... [1]

..... To avoid spitting/ frothing *(use of lid can score second mark of these two)* [1]

(c) - 2 marks

Burette has a higher % accuracy than a balance [for small masses] [1]
If "percentage" is not stated, reference to the burette tolerance is acceptable

Titration has an obvious colour change showing when reaction is complete [1]
Accept marking points from (b) if not awarded there

(d) – 4 marks *(but 3 shown on question paper)*

The hydrated solid might lose "extra" mass due to spitting during heating [1]

The anhydrous residue might decompose ***or*** evaporate [1]

Each/either error would make the measured mass loss too large [1]

Each error would also make the mass the anhydrous acid too small,
reducing the number of moles of it and increasing the ratio $^{n(water)}/_{n(anhydrous\ acid)}$ [1]

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