Examiners' Report

June 2013

GCE Chemistry 6CH08 01

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# Introduction

This paper was designed to allow candidates to demonstrate their knowledge of a wide variety of practical techniques and to show their ability to make valid inferences from experimental data.

Some of the questions were very accessible and were designed to allow even the weaker candidates to show their knowledge of the basic practical techniques used in chemistry. Others were more demanding, requiring a real understanding of some less familiar techniques. An important feature of answering such questions is for candidates to try to visualise what is happening in the equipment, exactly as though they were performing the experiment in the laboratory. They should also understand the reasons for what they are doing rather than simply regarding them as processes performed merely because they are routine. Every step has a reason which needs to be understood and explained.

Some of the questions requiring the interpretation of data were more challenging, providing the ablest candidates an opportunity to demonstrate their ability to apply their understanding of analytical techniques. In such questions it is important to use all the data available in order to reach valid conclusions.

# Question 1 (a) (i)

This question was well answered though a few candidates could not remember the colour of aqueous nickel(II) salts. Others gave incorrect formulae for the carbonate ion.

| Test | Observation | Inference |
|---|---|---|
| (i) Add dilute sulfuric acid to compound **Z** | Bubbles of a colourless gas are released. The gas turns limewater milky<br><br>and<br><br>a _green_ coloured solution is formed (1) | Name of gas released is<br>_carbon dioxide_<br><br>Formula of anion in **Z** is<br>$CO_3^{2-}$<br>(2)<br><br>Formula of the complex ion formed is<br>$[Ni(H_2O)_6]^{2+}(aq)$ |

**ResultsPlus**
**Examiner Comments**

A perfect answer!

**ResultsPlus**
**Examiner Tip**

Learn the colours of aqueous solutions of transition metal ions.

| Test | Observation | Inference |
|---|---|---|
| (i) Add dilute sulfuric acid to compound **Z** | Bubbles of a colourless gas are released. The gas turns limewater milky<br><br>and<br><br>a _Green_ coloured solution is formed (1) | Name of gas released is<br>_Carbon dioxide_<br><br>Formula of anion in **Z** is<br>$CO^{2-}$<br>(2)<br><br>Formula of the complex ion formed is<br>$[Ni(H_2O)_6]^{2+}(aq)$ |

**ResultsPlus**
**Examiner Comments**

A good answer spoiled by an incorrect formula for the carbonate ion.

**ResultsPlus**
**Examiner Tip**

Learn the formulae of common molecular ions.

# Question 1 (a) (ii)

This question was well answered though some candidates failed to appreciate that the negative charges on the chloride ions would determine the overall charge on the complex ion.

| | | | |
|---|---|---|---|
| (ii) | Add concentrated hydrochloric acid to the solution containing $[Ni(H_2O)_6]^{2+}$ ions | Yellow-brown solution forms | Formula of the complex ion formed is $[NiCl_4]^{2-}$ (1) |
| (iii) | Add a few drops | Green precipitate forms | Formula of the precipitate |

**ResultsPlus**
**Examiner Comments**

A perfectly correct answer.

**ResultsPlus**
**Examiner Tip**

Remember that chloride ions are large so normally form complexes with a co-ordination number of four.

| | | | |
|---|---|---|---|
| (ii) | Add concentrated hydrochloric acid to the solution containing $[Ni(H_2O)_6]^{2+}$ ions | Yellow-brown solution forms | Formula of the complex ion formed is $[NiCl_2]$ (1) |
| (iii) | Add a few drops | Green precipitate forms | Formula of the precipitate |

**ResultsPlus**
**Examiner Comments**

The candidate has failed to appreciate that a complex ion has been formed.

**ResultsPlus**
**Examiner Tip**

Remember that chloride ions normally form complexes with a co-ordination number of four.

## Question 1 (a) (iii)

Another question that most candidates were able to answer correctly.

| (iii) | Add a few drops of dilute aqueous ammonia to the solution containing $[Ni(H_2O)_6]^{2+}$ ions | Green precipitate forms | Formula of the precipitate formed is $[Ni(H_2O)_4(OH)_2]^0$ (1) |

**ResultsPlus Examiner Comments**

A perfectly correct answer.

**ResultsPlus Examiner Tip**

Remember that the hydrated hydroxide precipitate always has no overall charge.

| (iii) | Add a few drops of dilute aqueous ammonia to the solution containing $[Ni(H_2O)_6]^{2+}$ ions | Green precipitate forms | Formula of the precipitate formed is $[Ni(H_2O)_4(NH_3)_2]^0$ (1) |

**ResultsPlus Examiner Comments**

At low concentration the ammonia acts as base rather than a ligand so the hydroxide is formed.

**ResultsPlus Examiner Tip**

Remember that alkalis always initially cause the formation of the insoluble hydroxide precipitate.

# Question 1 (a) (iv)

Another well answered question though a fairly common error was to state that a green rather than a blue solution was formed.

| (iv) | Add excess dilute aqueous ammonia to the solution containing $[Ni(H_2O)_6]^{2+}$ ions until no further change is observed | Green precipitate dissolves to form clear blue solution <br><br> (1) | Formula of the complex ion formed is $[Ni(NH_3)_6]^{2+}$ |
|---|---|---|---|

**ResultsPlus**
**Examiner Comments**

An excellent answer.

**ResultsPlus**
**Examiner Tip**

Learn the colours of the complexes formed with aqueous ammonia.

| (iv) | Add excess dilute aqueous ammonia to the solution containing $[Ni(H_2O)_6]^{2+}$ ions until no further change is observed | green precipitate doesn't redissolve <br><br> (1) | Formula of the complex ion formed is $[Ni(NH_3)_6]^{2+}$ |
|---|---|---|---|

**ResultsPlus**
**Examiner Comments**

This is incorrect - the precipitate does dissolve in excess ammonia.

**ResultsPlus**
**Examiner Tip**

Learn which transition metal hydroxide precipitates dissolve in excess ammonia.

## Question 1 (b) (i)

Most candidates carried out this calculation correctly.

(b) A 10.0 cm³ sample of a solution containing $[Ni(H_2O)_6]^{2+}$ ions was titrated with a solution of concentration 0.010 mol dm⁻³ with respect to the ligand EDTA⁴⁻ ions. The equation for the reaction is
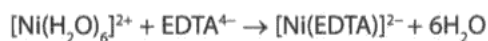
$$[Ni(H_2O)_6]^{2+} + EDTA^{4-} \rightarrow [Ni(EDTA)]^{2-} + 6H_2O$$

(i) The mean titre of the solution containing EDTA⁴⁻ ions was 24.20 cm³. Use this information, and the equation above, to calculate the concentration in mol dm⁻³ of the solution containing $[Ni(H_2O)_6]^{2+}$ ions.

(2)

$$n.o.m = \frac{MV}{1000}$$
(EDTA)

$$= \frac{0.010 \times 24.20}{1000}$$

$$= 2.42 \times 10^{-4} \, mol$$

$$M_{Ni} = \frac{n.o.m. \times 1000}{10.0}$$

$$= \frac{2.42 \times 10^{-4} \times 1000}{10.0}$$

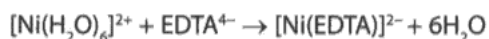$$= 0.0242 \, mol \, dm^{-3}$$

### ResultsPlus
**Examiner Comments**

A good, clearly set out answer.

### ResultsPlus
**Examiner Tip**

Always set out calculations clearly. You are much more likely to score the marks if the examiner can follow your reasoning.

The equation for the reaction is

$$[Ni(H_2O)_6]^{2+} + EDTA^{4-} \rightarrow [Ni(EDTA)]^{2-} + 6H_2O$$

(i) The mean titre of the solution containing $EDTA^{4-}$ ions was 24.20 cm³.
Use this information, and the equation above, to calculate the concentration in mol dm⁻³ of the solution containing $[Ni(H_2O)_6]^{2+}$ ions.

(2)

$$n_{EDTA} = CV = 0.01\left(\frac{24.2}{1000}\right) = 2.42 \times 10^{-3}$$

$$n_{Ni} = CV$$

$$2.42 \times 10^{-3} = C\left(\frac{10}{1000}\right)$$

$$C = 0.242 \text{ mol dm}^{-3}$$

ResultsPlus
**Examiner Comments**

The 'power of ten' error in the calculation of the number of moles loses the first mark. However the method of calculation of the concentration is correct so the second mark is gained.

ResultsPlus
**Examiner Tip**

In cases like this it is very important to set out your workings clearly. The examiner can see that the method of calculation of the concentration is correct, so the second mark is gained despite the initial error.

## Question 1 (b) (ii)

This question was very well answered.

A few candidates incorrectly doubled the error even though the **total** error in the titre was given in the question, not the error in the two individual readings of the burette.

(ii) Assuming the total error in the measurement of the mean titre is ±0.10 cm³, calculate the percentage error in this titre.

(1)

$$\frac{0.1}{24.2} = 0.41\%$$

**ResultsPlus Examiner Comments**

A correct answer.

**ResultsPlus Examiner Tip**

Make sure you know the formula for the calculation of percentage errors.

(ii) Assuming the total error in the measurement of the mean titre is ±0.10 cm³, calculate the percentage error in this titre.

(1)

$$2 \times 0.10 = \pm 0.20$$

$$percentage \ error = \frac{\pm 0.20}{24.20} \times 100\% = 0.83\%$$

(iii) A similar solution, containing the same concentration of [Ni(H₂O)₆]²⁺ ions, also

**ResultsPlus Examiner Comments**

The candidate has incorrectly doubled the error.

**ResultsPlus Examiner Tip**

Read the question carefully - the **total** error is given.

## Question 1 (b) (iii)

Most candidates realised that the titre would increase but not all gave a coherent reason for this and lost the second mark. A common error was to suggest that the presence of impurities lowers the concentration of the nickel ions, so reducing the titre.

(iii) A similar solution, containing the same concentration of $[Ni(H_2O)_6]^{2+}$ ions, also contained a small amount of an impurity, copper(II) sulfate.

Suggest what effect this impurity would have on the titre. Justify your answer.

(2)

More titre is needed for the titration. This is because $Cu^{2+}$ ion in $CuSO_4$ reacts (will also) with $EDTA^{4-}$ to form complex which is $[Cu(EDTA)]^{2-}$.

**ResultsPlus**
**Examiner Comments**

A good answer - the reason for the increase in titre is clearly explained.

**ResultsPlus**
**Examiner Tip**

If the question says "Justify your answer" you must explain the answer you give.

(iii) A similar solution, containing the same concentration of $[Ni(H_2O)_6]^{2+}$ ions, also contained a small amount of an impurity, copper(II) sulfate.

Suggest what effect this impurity would have on the titre. Justify your answer.

(2)

The titre would be lower than the actual value as copper (II) sulfate also reacts with $EDTA^{4-}$ ions

**ResultsPlus**
**Examiner Comments**

The candidate understands that $EDTA^{4-}$ will react with the copper(II) sulfate but fails to appreciate that this would increase the titre.

**ResultsPlus**
**Examiner Tip**

Try to think through your answers logically - this one is contradictory.

## Question 2 (a)

Most candidates answered this question correctly.

2 A colourless liquid, compound **X**, was extracted from raspberries. **X** has the molecular formula $C_{10}H_{12}O_2$ and contains a benzene ring.

(a) What would you expect to see if a sample of compound **X** was burned in air?

(1)

*Smoky flame.*

**ResultsPlus**
**Examiner Comments**

Correct.

2 A colourless liquid, compound **X**, was extracted from raspberries. **X** has the molecular formula $C_{10}H_{12}O_2$ and contains a benzene ring.

(a) What would you expect to see if a sample of compound **X** was burned in air?

(1)

*Grey clouds and vapour*

**ResultsPlus**
**Examiner Comments**

This is clearly wrong.

## Question 2 (b) (i)

This question was quite challenging. Many candidates assumed that the test with bromine water indicated a carbon-carbon double bond, others identified an -OH group but failed to state that it was positioned on a benzene ring.

(b) A series of tests was carried out on compound **X**. In each test, state what you can deduce about the structure of compound **X** from the results described.

(i) **X** forms a white precipitate with aqueous bromine solution.

(1)

X contain phenol group (-OH)

**ResultsPlus**
**Examiner Comments**

A good clear answer.

**ResultsPlus**
**Examiner Tip**

Remember that bromine water reacts with phenols as well as alkenes.

(b) A series of tests was carried out on compound **X**. In each test, state what you can deduce about the structure of compound **X** from the results described.

(i) **X** forms a white precipitate with aqueous bromine solution.

(1)

X is an alcohol  X has -OH group

**ResultsPlus**
**Examiner Comments**

The candidate has failed to state that the -OH group is on a benzene ring.

**ResultsPlus**
**Examiner Tip**

Alcohols do not react with bromine water - this must be a phenol.

## Question 2 (b) (ii)-(iii)

These tests for functional groups were generally well known. Some candidates lost the final mark by not completing the inference that if X was not an aldehyde, it must be a ketone.

(ii) **X** forms an orange precipitate with 2,4-dinitrophenylhydrazine.

(1)

X can be either an aldehyde or a ketone, presence of oxygen double bonded to a carbon.

(iii) Fehling's (or Benedict's) solution remains blue when warmed with compound **X**.

(1)

X is a ketone

**ResultsPlus**
**Examiner Comments**

An excellent answer.

(ii) **X** forms an orange precipitate with 2,4-dinitrophenylhydrazine.

(1)

It or contain carbonyl group . Contain C=O .

(iii) Fehling's (or Benedict's) solution remains blue when warmed with compound **X**.

(1)

It is not an aldehyde contain aldehyde. group

**ResultsPlus**
**Examiner Comments**

The second mark is lost because it is not stated that X is a ketone.

**ResultsPlus**
**Examiner Tip**

A carbonyl that is not an aldehyde must be a ketone.

## Question 2 (c) (i)

This question was poorly answered. Many candidates thought that one of the nmr peaks was caused by the hydrogen atom of the phenol group despite the fact that both peaks were clearly labelled on the spectrum as due to two hydrogen atoms.

(i) Which hydrogen atoms in compound **X** are most likely to have caused the peaks at 6.5 ppm and 7.2 ppm?

(1)

Hydrogen atom that attach to the arene ring.

**ResultsPlus**
**Examiner Comments**

This is correct.

(i) Which hydrogen atoms in compound **X** are most likely to have caused the peaks at 6.5 ppm and 7.2 ppm?

(1)

X contain phenyl group. So H atom in -OH group.

(ii) Compound **X** has a side chain containing four carbon atoms attached to the

**ResultsPlus**
**Examiner Comments**

This is incorrect.

**ResultsPlus**
**Examiner Tip**

Look at the spectrum carefully - both peaks are caused by two hydrogen atoms. The OH of the phenol group only has one hydrogen atom.

# Question 2 (c) (ii)-(iii)

This was a very challenging question. Only the ablest students were able to put together all the information from previous parts of the question - the presence of a phenol group and a ketone group, a molecular formula of $C_{10}H_{12}O_2$ and data from the nmr spectrum to deduce the correct structure for X.

Many failed to realise that one oxygen atom was present in the phenol group, so only one could be in the side chain.

Many knew the 'n+1 rule' but could not apply it in this situation. The number of hydrogen atoms causing each nmr peak was frequently ignored.

(ii) Compound X has a side chain containing four carbon atoms attached to the benzene ring. Show all the atoms on this side chain and label each hydrogen environment on the side chain with its splitting pattern.
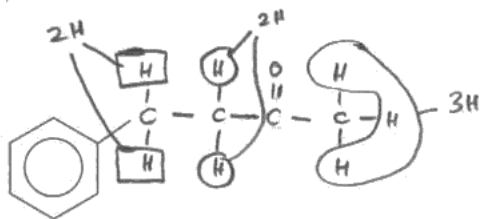


(3)

The 3 hydrogen atoms bonded to the last carbon in the methyl (C CH₃) group is the correspond to the 3H hydrogen environment with singlet peak at around 2 ppm. While the two hydrogen atoms bonded to the second carbon atom correspond to the 2H hydrogen environment with triplet doublet peaks, and the same applies to the 2 hydrogens bonded to first carbon in the side chain. (2H)

(iii) Suggest the structural formula of X.

(1)

ResultsPlus
**Examiner Comments**

An excellent answer.

ResultsPlus
**Examiner Tip**

Effective use of labelling on a diagram is a very good way of communicating chemical information.

(ii) Compound **X** has a side chain containing four carbon atoms attached to the benzene ring. Show all the atoms on this side chain and label each hydrogen environment on the side chain with its splitting pattern.

$C_{10}H_{12}O_2$
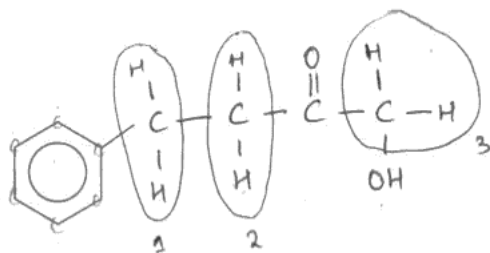


$C_6H_5$

$(C_6H_5CH_2)CH_2COCH_2OH$

(3)

Hydrogen environment 1 - $-CH_2$ group adjacent to a $-CH_2$ group therefore a triplet. Same for hydrogen environment 2.

Hydrogen environment 3 - no adjacent hydrogen environment but next to $-OH$ group (this is a singlet), therefore a doublet.

The $C=O$ group has no hydrogen atoms attached to the carbon atom and so no split will be seen, it is a singlet.

(iii) Suggest the structural formula of **X**.

(1)

~~$(C_6H_5CH_2)CH_2COCH_2OH$~~

~~$C_6H_5(CH_2)CH_2$~~

$C_6H_5CH_2CH_2COCH_2OH$
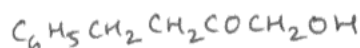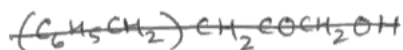
**ResultsPlus**
**Examiner Comments**

The side chain has an incorrect extra -OH group but some marks are salvaged because the two $CH_2$ groups would be triplets cause by two hydrogen atoms and the ketone group is on the correct carbon atom.

The complete structure in part (ii) has no phenol group so cannot score.

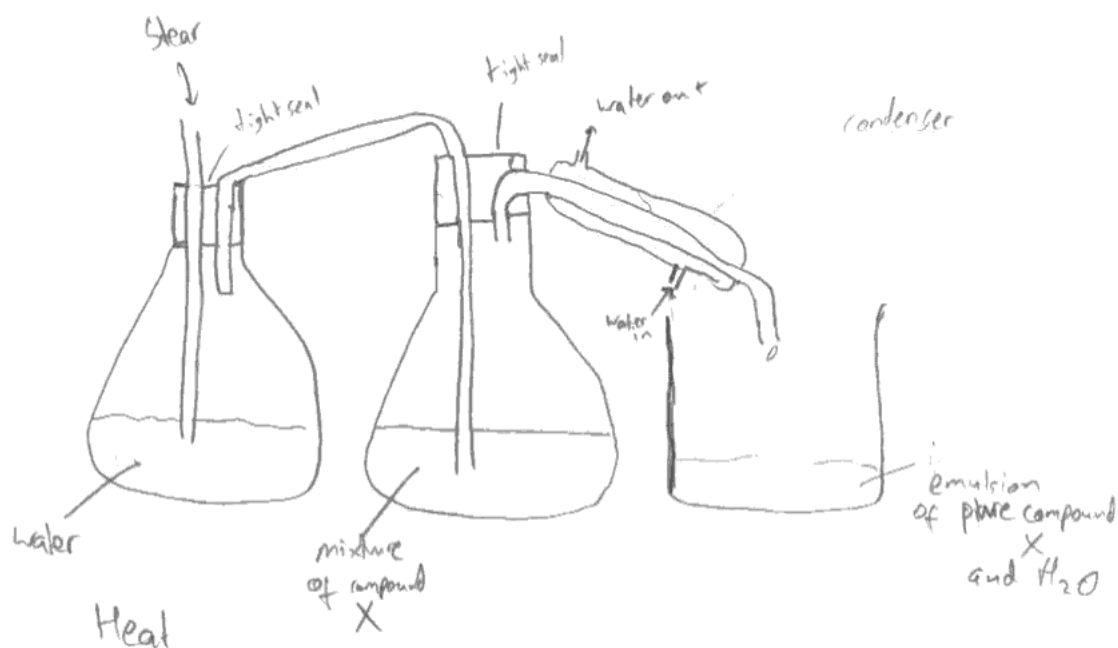**ResultsPlus**
**Examiner Tip**

Even incorrect answers with **some correct reasoning** can score marks.

## Question 2 (d)

The technique of steam distillation was better known than in previous years though poor diagrams often spoiled reasonable answers.

(d) Compound **X** can be extracted from raspberries by steam distillation. Draw a labelled diagram of the apparatus you could use to carry out this steam distillation.

(3)



**ResultsPlus**
**Examiner Comments**

An excellent answer.

(d) Compound **X** can be extracted from raspberries by steam distillation. Draw a labelled diagram of the apparatus you could use to carry out this steam distillation.

(3)
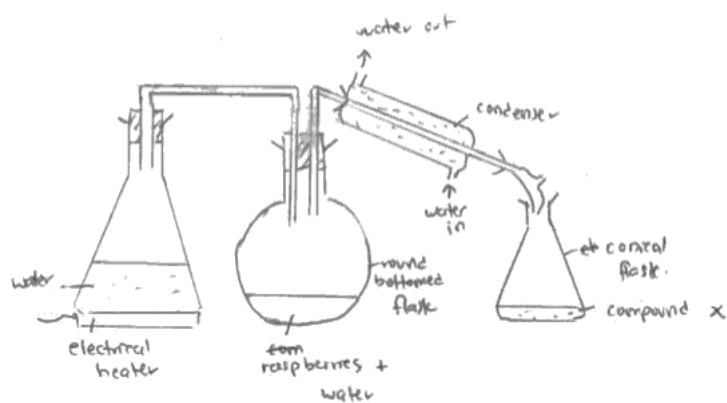


## ResultsPlus
### Examiner Comments

The delivery tube from the steam generator does not dip into the raspberry extract, so the technique would not work and so one mark is lost.

## ResultsPlus
### Examiner Tip

Think about the practical use of apparatus you draw. Be sure that it would really work.

## Question 3 (a) (i)

Most students correctly chose to use a pipette or burette because of their accuracy.

A measuring cylinder can be used but it must be made clear that this is because it would allow measurements to be made which are accurate enough for finding orders of reaction.

(a) (i) Which piece of equipment should be used to measure out the volumes used in each experiment? Justify your choice.

(2)

A pipette. This is because a pipette gives a more accurate and exact volume of the solution being measured. Furthermore, there is no excess solution being measured.

### ResultsPlus
**Examiner Comments**

Correct.

(a) (i) Which piece of equipment should be used to measure out the volumes used in each experiment? Justify your choice.

(2)

use pipette to measure the volume as the volume to all the volume that needed for each solution is less than 25 cm³

### ResultsPlus
**Examiner Comments**

The correct equipment is used but the reason is quite inadequate.

### ResultsPlus
**Examiner Tip**

Pipettes and burettes are used because they enable volumes to be measured accurately.

## Question 3 (a) (ii)

Generally well answered.

(ii) What colour change would you see in step 3?

(2)

From ...... purple ...... to ...... colourless ......

**ResultsPlus**
**Examiner Comments**

Correct.

(ii) What colour change would you see in step 3?

(2)

From ...... purple ...... to ...... clear ......

**ResultsPlus**
**Examiner Comments**

'Clear' means transparent, not 'colourless'.

**ResultsPlus**
**Examiner Tip**

Don't write 'clear' when you mean 'colourless'.

## Question 3 (a) (iii)

This question was fairly well answered with most candidates appreciating the need to have the same total volume in each of the experiments.

(iii) Explain why water was added to the flask in experiments B, C and D.

(1)

To keep the total volume of solution constant and consistent for a fair controlled experiment

**ResultsPlus**
**Examiner Comments**

A good answer.

(iii) Explain why water was added to the flask in experiments B, C and D.

(1)

To dilute the concentration of sulfuric acid.

**ResultsPlus**
**Examiner Comments**

The candidate has missed the point.

## Question 3 (a) (iv)

Most candidates correctly suggested the use of colorimetry, though a few threw away the mark by writing "c**a**lorimetry" instead of "c**o**lorimetry". Those who opted for a titration based method often failed to specify the reagent to be used to titrate the potassium manganate(VII) and therefore lost the mark.

(iv) Suggest a technique that could be used to continuously monitor the change in concentration of potassium manganate(VII) during the reaction.
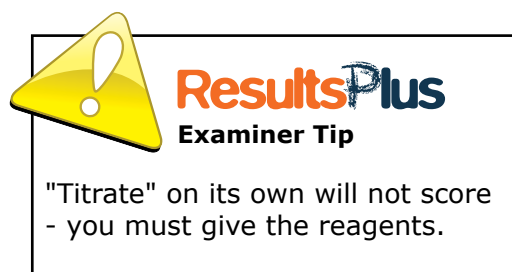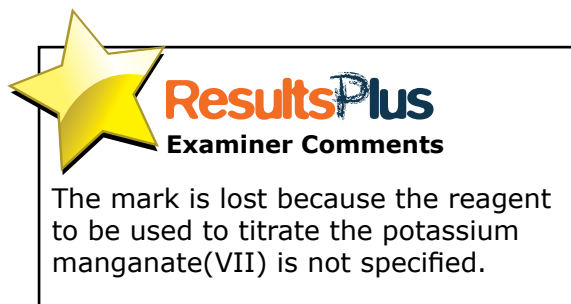
(1)

colorimetry to measure the change in colour intensity

**ResultsPlus**
**Examiner Comments**

Correct.

(iv) Suggest a technique that could be used to continuously monitor the change in concentration of potassium manganate(VII) during the reaction.

kmno4

(1)

quench the solution and titrate

**ResultsPlus**
**Examiner Comments**

The mark is lost because the reagent to be used to titrate the potassium manganate(VII) is not specified.

**ResultsPlus**
**Examiner Tip**

"Titrate" on its own will not score - you must give the reagents.

## Question 3 (a) (v)

The majority of candidates were able to calculate the orders of reaction - often using rather over-elaborate mathematical calculations. Marks were frequently lost in the writing of the rate equation with the rate constant being omitted, or the expression "rate equation" being used instead of "rate".
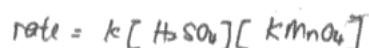
(v) State the order with respect to glucose, sulfuric acid and potassium manganate(VII) and hence write the rate equation for the reaction.

∴ (3)

Partial order of glt glucose is zero order.

Partial order of sulfuric acid is first order.

Partial order of potassium manganate (VII) is first order.

rate = k [ H₂SO₄ ][ KMnO₄ ]

**ResultsPlus**
**Examiner Comments**

A clear correct answer.

(v) State the order with respect to glucose, sulfuric acid and potassium manganate(VII) and hence write the rate equation for the reaction.

(3)

order with respect to potassium manganate (VII)
= first order  0

order with respect to glucose
= First order

order with respect to sulfuric acid
= first order

rate equation = [ KMnO₄ ][glucose]¹[H₂SO₄]¹

**ResultsPlus**
**Examiner Comments**

Only one order is correct so two marks are lost. The final mark could have been obtained with a rate equation consistent with the stated orders but the rate constant was omitted and "rate equation" was written instead of "rate".

**ResultsPlus**
**Examiner Tip**

Learn the standard format of a rate equation.

## Question 3 (b)

In part b(i), very many candidates lost a mark by failing to use all of the graph paper and constricting the graph to less than half of the available space. The graph paper printed allowed straightforward scales which filled most of the space.

In part b(ii), again many candidates failed to calculated the gradient correctly.
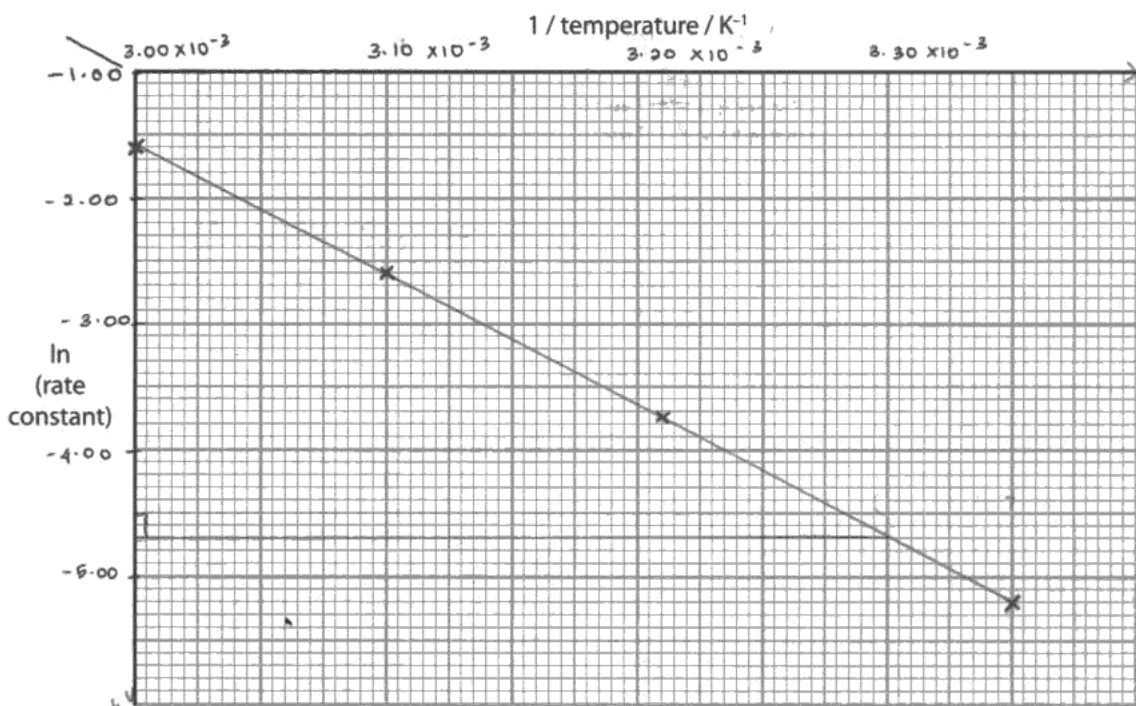
In part b(iii), one mark was often lost for incorrectly stating the units to be J or kJ rather than $Jmol^{-1}$ or $kJmol^{-1}$.
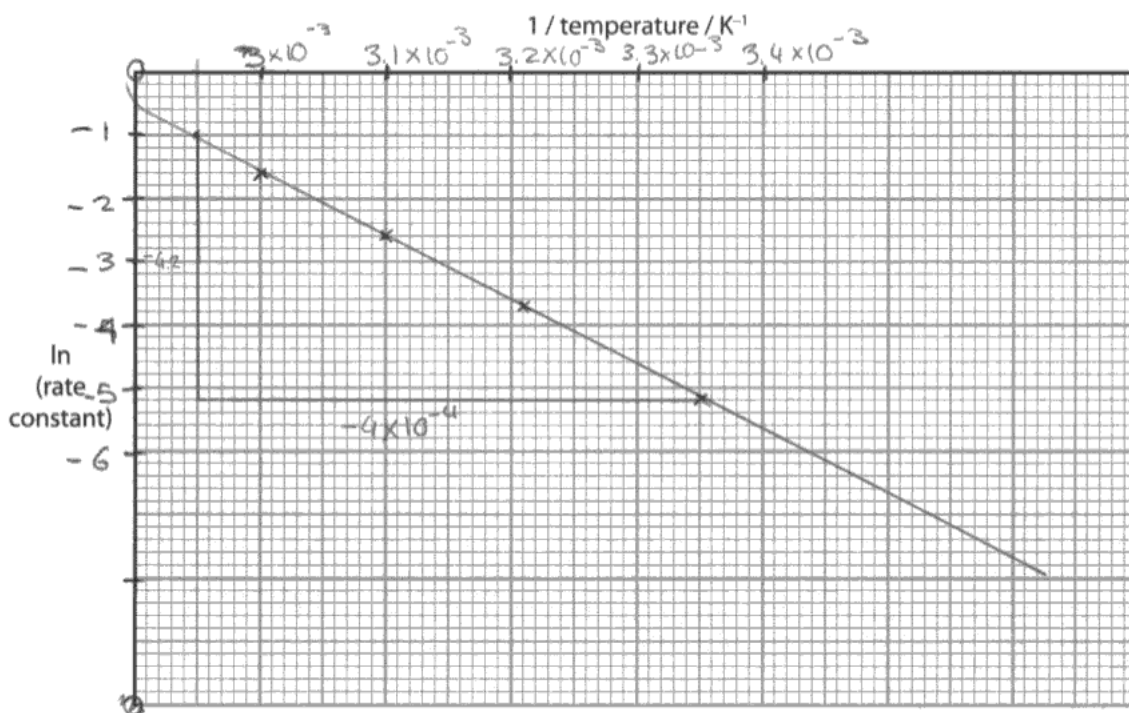
(b) Experiment A was repeated at different temperatures and the time taken for the potassium manganate(VII) to change colour was recorded. The results were processed to find values of 1/temperature and ln (rate constant) and these are shown in the table below.

| Experiment | 1 / temperature / K⁻¹ | ln (rate constant) |
|------------|------------------------|---------------------|
| E | $3.00 \times 10^{-3}$ | −1.60 |
| F | $3.10 \times 10^{-3}$ | −2.60 |
| G | $3.21 \times 10^{-3}$ | −3.75 |
| H | $3.35 \times 10^{-3}$ | −5.20 |

(i) Plot a graph of ln (rate constant) against 1 / temperature on the axes below.

(3)



(ii) Calculate the gradient of the graph.

(1)

$$M = \frac{-4.7 - (-1.6)}{3.3 \times 10^{-3} - 3 \times 10^{-3}}$$

$$= -10\ 333.33$$

$$\frac{-5.2 - (-1.6)}{3.35 \times 10^{-3} - 3 \times 10^{-3}}$$

$$= -10\ 285.71$$

(iii) Use your answer to (ii) and the relationship below to calculate the activation energy, $E_a$, for this reaction. Include a sign and units in your answer.

$$\text{Gradient} = \frac{-E_a}{R}$$

$R = 8.31 \, J \, K^{-1} \, mol^{-1}$

(2)

$-10\,333.33K = \dfrac{-Ea}{8.31 k^{-1} mol^{-1}}$

$Ea = 85\,870 \, Jmol$
$85\,870 \, Jmol^{-1}$

$-10\,285.7 \, k = \dfrac{-Eo}{8.31 \, Jk^{-1} \, mol^{-1}}$

$Ea = 85\,474.29 \, Jmol^{-1}$
$85\,474.29 \, Jmol^{-1}$

(b) Experiment A was repeated at different temperatures and the time taken for the potassium manganate(VII) to change colour was recorded. The results were processed to find values of 1/temperature and ln (rate constant) and these are shown in the table below.

| Experiment | 1 / temperature / K$^{-1}$ | ln (rate constant) |
|---|---|---|
| E | $3.00 \times 10^{-3}$ | −1.60 |
| F | $3.10 \times 10^{-3}$ | −2.60 |
| G | $3.21 \times 10^{-3}$ | −3.75 |
| H | $3.35 \times 10^{-3}$ | −5.20 |

(i) Plot a graph of ln (rate constant) against 1 / temperature on the axes below.

(3)



(ii) Calculate the gradient of the graph.

(1)

$-4.2 \times (-4 \times 10^{-4}) = 1.68 \times 10^{-3}$

$$\frac{-4.2}{(-4 \times 10^{-4})} = 10500$$

(iii) Use your answer to (ii) and the relationship below to calculate the activation energy, $E_a$, for this reaction. Include a sign and units in your answer.

$$\text{Gradient} = \frac{-E_a}{R}$$

$R = 8.31 \text{ J K}^{-1} \text{ mol}^{-1}$

(2)

10,500
$\cancel{1.68 \times 10^3} = \dfrac{-E_a}{8.31 \text{ J K}^{-1}\text{mol}^{-1}}$

10,500
$\cancel{1.68 \times 10^3} \times 8.31 \text{ J K}^{-1}\text{mol}^{-1} = -E_a$

$87,255 \text{ J K}^{-1}\text{mol}^{-1} = -E_a$

$E_a = -87,2500 \text{ J K}^{-1}\text{mol}^{-1}$

## Question 4 (a) (i)

Many candidates were clearly unfamiliar with this technique and scored poorly. Practical detail was required so it was not enough to simply say that the two layers must be separated. To score the mark it was necessary to explain **how** they were to be separated. A significant number of candidates ignored the density data and assumed that the aqueous layer was the lower layer.

---

4   The procedure outlined below can be used to extract caffeine from tea.

1.   Add 25 g of tea, 10 g of calcium carbonate and 250 cm³ of water to a large beaker.

2.   Gently boil the mixture for 15 minutes.

3.   While the mixture is still warm, filter using suction filtration.

4.   Transfer the filtrate to a separating funnel and separate the caffeine from the aqueous mixture using solvent extraction, with dichloromethane as the solvent.

5.   Dry the extract.

6.   Remove the solvent.

[Density of dichloromethane = 1.32 g cm⁻³]

(a) (i)   Outline how to carry out the solvent extraction in **step 4**, to obtain a solution of caffeine dissolved in dichloromethane.

(3)

Once filtrate is transferred to seperating funnel add excess $CH_2Cl_2$ to the mixture _in funnel_ and gently shake the funnel and invert it, to mix the solutions. Open the tap and collect the solvent (lower layer) to a beaker. Add more $CH_2Cl_2$ to the remaining mixture in seperating funnel. Stand for few minutes and remove the lower denser layer only. Repeat process until clear aqueous mixture remains.

---

**ResultsPlus**
**Examiner Comments**

An unusually good answer which obtained full marks.

**ResultsPlus**
**Examiner Tip**

In a test of practical chemistry, make sure that you give experimental detail in your answers.

**4** The procedure outlined below can be used to extract caffeine from tea.

1. Add 25 g of tea, 10 g of calcium carbonate and 250 cm³ of water to a large beaker.

2. Gently boil the mixture for 15 minutes.

3. While the mixture is still warm, filter using suction filtration. *~buchner*

4. Transfer the filtrate to a separating funnel and separate the caffeine from the aqueous mixture using solvent extraction, with dichloromethane as the solvent.

5. Dry the extract. ~

6. Remove the solvent. ~

*aqueous 1.32*
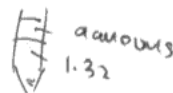
[Density of dichloromethane = 1.32 g cm⁻³]

(a) (i) Outline how to carry out the solvent extraction in **step 4**, to obtain a solution of caffeine dissolved in dichloromethane.

(3)

add ~~add~~ add dichloromethane to aqueous mixture in a separating funnel, ~~so~~ two layers are formed. with ~~aqueous~~ ~~mixture~~ dichloromethane ~~k~~ at the bottom of separating funnel and aqueous mixture on top. Open the ~~sep~~ tap of separating funnel allowing the ~~dichlor~~ solvent with caffein dissolved ~~to~~ into a beaker. ~~it~~ and close it when all the solvent has poured off. Discard the aqueous layer.
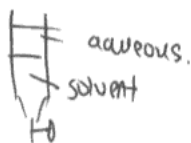
*aqueous.*
*solvent*

## Question 4 (a) (ii)

Most candidates could name a suitable drying agent but few went on to explain how it could be separated from the dried liquid after its use.

(ii) How would you dry the extract in **step 5**? Include the name of a suitable drying agent in your answer.

(2)

You could add ~~the~~ anhydrous calcium chloride, and then filter the mixture

**ResultsPlus**
**Examiner Comments**

A good answer.

(ii) How would you dry the extract in **step 5**? Include the name of a suitable drying agent in your answer.

(2)

Add anhydrous $CaCl_2$ which will act as a drying agent by removing excess $H_2O$ from the solution. (It become hydrated $CaCl_2$

**ResultsPlus**
**Examiner Comments**

The drying agent is correct but there is no indication of how it is to be removed after use.

**ResultsPlus**
**Examiner Tip**

A two mark question will require two separate points in the answer.

## Question 4 (b) (i)

Almost all candidates would wear gloves but a worrying number would use a face mask rather than a fume cupboard to handle a solvent which is harmful if inhaled.

(b) (i)  The solvent dichloromethane is harmful and can enter the body through inhalation and skin absorption. Suggest a possible way to minimise each of these risks when using dichloromethane.

(2)

Inhalation  *Carry out the experiment in a fume cupboard*

Skin absorption  *Wear gloves when handling dichloromethane.*
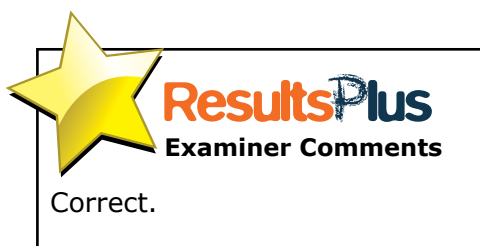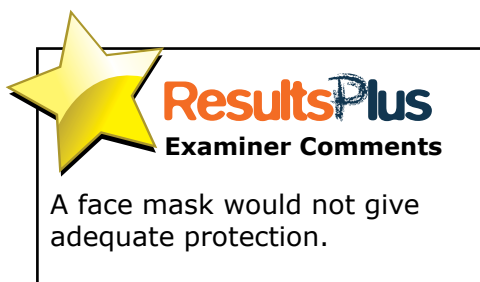
ResultsPlus
**Examiner Comments**

Correct.

(b) (i)  The solvent dichloromethane is harmful and can enter the body through inhalation and skin absorption. Suggest a possible way to minimise each of these risks when using dichloromethane.

(2)

Inhalation  *or wear mask.*

Skin absorption  *wear glove and lab coat .*

ResultsPlus
**Examiner Comments**

A face mask would not give adequate protection.

## Question 4 (b) (ii)

Unfamiliarity with the technique of solvent extraction was evident again here. Many candidates suggested wholly inappropriate techniques such as recrystallization to attempt to remove the solvent.

(ii) Suggest a suitable way to remove the solvent in **step 6**.

(1)

*Carry out distillation since the boiling points of caffeine and dichloromethane differ (Boil dichloromethane out of solution).*

**ResultsPlus**

**Examiner Comments**

A good answer.

**ResultsPlus**

**Examiner Tip**

Learn the basic techniques for purifying solids and liquids.

(ii) Suggest a suitable way to remove the solvent in **step 6**.

(1)

*Evaporation*

**ResultsPlus**

**Examiner Comments**

The idea is correct but no practical detail is given so the mark is lost.

**ResultsPlus**

**Examiner Tip**

In this paper details of practical techniques are required.

## Question 4 (c)

Most candidates appreciated that liquid carbon dioxide was the less harmful solvent. Some of the more astute realised that it would also be much easier to remove. Either argument gained the mark.

(c) The extraction can also be carried out using liquid carbon dioxide. Suggest an advantage of using this rather than dichloromethane.

(1)

Carbon dioxide can be easily removed by heating the extract.

**ResultsPlus Examiner Comments**

An excellent answer.

(c) The extraction can also be carried out using liquid carbon dioxide. Suggest an advantage of using this rather than dichloromethane.

(1)

no harmful gases are released

carbon dioxide is safer to handle than dichloromethane.

**ResultsPlus Examiner Comments**

This answer is too vague to score - it must be clear how the carbon dioxide is safer.

**ResultsPlus Examiner Tip**

Don't be vague!

## Question 4 (d)

This question was generally very well answered though a few candidates could not convert from milligrams to grams.

(d) A student carrying out this extraction obtained 85 mg of caffeine. Calculate the percentage by mass of caffeine obtained from the sample of tea used.

(2)

theoretical

$\text{theoretical} =$
$\text{mass obtained}$

$\% \text{ mass} = \dfrac{85 \times 10^{-3} \text{ g}}{25 \text{ g}} \times 100$

$= 0.34\%$

**ResultsPlus**
**Examiner Comments**

A good clear answer.

(d) A student carrying out this extraction obtained 85 mg of caffeine. Calculate the percentage by mass of caffeine obtained from the sample of tea used.

(2)

$= \dfrac{85}{90} \times 100\%$

$= \dfrac{85}{275} \times 100\%$

$= 31\%$

**ResultsPlus**
**Examiner Comments**

The candidate failed to convert from 'mg' to 'g'. However the incorrect mass was correctly used in the calculation of the percentage so the second mark is gained.

**ResultsPlus**
**Examiner Tip**

Read the question carefully!

## Question 4 (e)

This question was well answered though some candidates incorrectly opted for distillation.

(e) Caffeine obtained in this way is often a pale green solid, due to impurities. State the name of another technique you could use to further purify the caffeine.
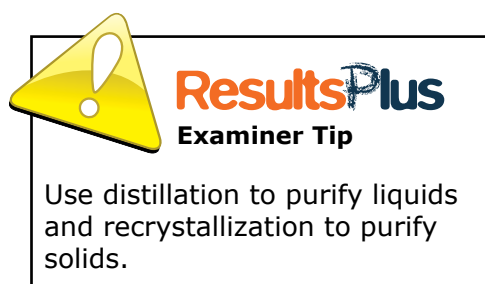
(1)

Recrystallisation

### ResultsPlus
**Examiner Comments**

Correct.

(e) Caffeine obtained in this way is often a pale green solid, due to impurities. State the name of another technique you could use to further purify the caffeine.
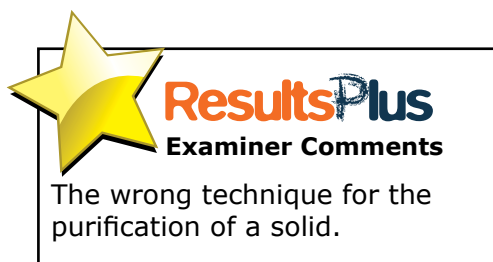
(1)

Distillation in solution

### ResultsPlus
**Examiner Comments**

The wrong technique for the purification of a solid.

### ResultsPlus
**Examiner Tip**

Use distillation to purify liquids and recrystallization to purify solids.

# Paper Summary

The paper worked well, allowing all the candidates the opportunity to show their knowledge and understanding of practical chemistry. The paper was accessible to the weaker candidates who were able to show their knowledge of basic techniques and the more challenging interpretation questions allowed the ablest to show their ability.

Based on their performance on this paper candidates are offered the following advice.

• When drawing graphs use the whole of the grid printed on the question paper not just a small portion.

• Practise calculating the gradient of graphs correctly.

• When interpreting nmr spectra make sure you understand the 'n+1' rule.

• In the interpretation of the nmr spectrum to deduce a structure do not ignore the number of hydrogen atoms responsible for a peak.

While the technique of steam distillation was much better known than in previous papers, many candidates were unfamiliar with the technique of solvent extraction. Make sure you can describe the techniques listed in the specification and that you understand their use and the chemical principles involved in each.

# Grade Boundaries

Grade boundaries for this, and all other papers, can be found on the website on this link:

http://www.edexcel.com/iwantto/Pages/grade-boundaries.aspx