

Cambridge NATIONALS

SAMPLE LEARNER WORK WITH COMMENTARY

UNIT R074: HOW SCIENTISTS USE ANALYTICAL TECHNIQUES TO COLLECT DATA

SCIENCE IN THE WORKPLACE Level 1/2



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INTRODUCTION

This is a guide for teachers so that you can see how we would mark work, Cambridge Nationals are designed to give the learners the project and let them create the work.

The guide contains sample learner work for this unit and covers all learning objectives, graded at Marking Band 1 (MB1) and Marking Band 3 (MB3).

The accompanying commentary explains why each piece of work was awarded its grade.

For MB1 graded work, additional guidance has been added to suggest improvements that could be made to make it an MB2 graded piece of work.

For MB3 graded work, additional guidance has been added to explain why it was awarded that grade and not the lower grade of MB2.

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Sample Learner Work Marking Band 1

SAMPLE LEARNER WORK

Unit R074: How scientists use analytical techniques to collect data

Overarching Learning Objective

LO1: Be able to apply the principles of good laboratory practice

- prepare samples and standard solutions and calibration
- carries out techniques required
- risk assessment and work safely
- analyses and justifying the techniques
- evaluate outcomes

LO2: Be able to separate and identify the substances present in a mixture

- select and set up equipment
- take and record measurements
- calculate Rf values
- types of chromatography to improve analysis

Health Scenario

The contamination of a batch of sauce with the banned dye Sudan has led to the UK's biggest food recall. More than 400 food products from major manufacturers and retailers have been recalled. Food needs to be tested for traces of Sudan 1.

I have been given a sample of food I will test for Sudan. I will use chromatography.

Risk assessment

The solvent is harmful and flammable. I will do the experiment in the fume cupboard and wear goggles.





Distance moved by solvent = 8 cm Distance moved by 1 = 2 and 5 cm Distance moved by 2 = 6 cm Distance moved by 3 = 2 cm Distance moved by 4 = 3 cm

Rf value of spots in the food = 0.25 and 0.75

The food sample has E127 in it but also an unknown dye. It might be Sudan 1. Scientists would analyse food dyes by gas chromatography or HPLC.

LO3: Be able to examine and record features of samples

- select and set up equipment
- take and record measurements
- calculate magnification and scale
- types of instrumental analysis to improve analysis

Health Scenario

Experts say that our hair says a lot more about us than how closely we follow the latest styles. In fact, the health of our hair and scalp can be a major tip-off to a wide variety of health conditions.

The sun wind and cold dry air also affects our hair.

I decided to look at my hair (natural blonde) and also my friend's hair, which is dyed bright red. I put a drop of glycerol on a microscope slide and put a very small piece of hair on top. I looked at the hair down the microscope.

Risk assessment

You could cut yourself on the slide or coverslip. Glycerol has a very low hazard but I am going to wear goggles to be safe.



My hair

The magnification I used was times 400 I worked this out by multiplying 10 and 40 (the strength of the lenses).



Friend's hair

You can see that my hair is much healthier and in very good condition the edge of my friend's hair is broken and splintered. I was surprised that the edges of her hair were greeny blue while the centre was red.

Scientists would use the same method as I did but would also use electronic microscopes to look at the hair.

LO4: Be able to identify cations and anions in samples

- select and set up equipment
- take and record observations
- types of instrumental techniques to improve analysis

Health Scenario

Too much salt in the diet has been shown to increase health risks and can lead to an increased risk of heart problems. LoSalt has been produced as a salt substitute.

I am going to analyse a sample of LoSalt to see what it contains.

Risk assessment

Because I was using chemicals, I wore goggles throughout the experiment.

Method

We used chemical tests for ions. Each test needed to be done more than once to determine the chemical nature of the substance. We used a variety of tests to determine what the substances were. Some of these were the A to D tests and the flame test.

Test for chloride (Test A)

We added a few drops of dilute nitric acid and a few drops of silver nitrate solution to a solution of LoSalt and normal salt. If chloride ions are present, then a white precipitate is formed.

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Test for sulphate (Test B)

We added a few drops of barium chloride solution and a few drops of dilute hydrochloric acid to a solution of LoSalt and normal salt. If sulphate ions are present, then a white precipitate of barium sulphate is formed.

Test for carbonate (Test C)

A few drops of dilute hydrochloric acid are added to a small amount of LoSalt and normal salt. If carbonate ions are present, then bubbles of carbon dioxide are given off.

Test for cations (Test D)

A few drops of dilute sodium hydroxide are added to the solution of LoSalt and normal salt and the tube is shaken gently. Then more of the dilute sodium hydroxide is added and the tube is shaken.

Flame test (Test E)

For this test we used 2-3 drops of concentrated hydrochloric acid and had the hole to the Bunsen burner slightly open. Then we put the LoSalt and normal salt on two watch glasses. We placed the hoop end of the nichrome wire into the solution and put it into the flame. If the colour of the flame was lilac then the substance was potassium. If the colour was golden yellow, the substance was sodium.

Results table

Salt	Flame Test (test E)	Test A	Test B	Test C	Test D
LoSalt	Bright yellow	white precipitate	*	A few bubbles	nothing
Normal salt	Bright yellow	white precipitate	nothing	No bubbles were given off	nothing

Sodium was present in both samples.

When I'd completed the tests my teacher told me to do them again, but this time look through a piece of blue glass. The flame for the LoSalt was a bright purply red pink colour telling me that potassium was present.

Scientists to identify many ions, would use HPLC instruments to see what's there and how much.

LO5: Be able to determine the concentration of an acid or base using titration

- select indicator and equipment setting it up correctly
- take and record measurements
- process data using mathematical techniques
- type of instrumental technique to improve analysis of samples by titration

Health Scenario

Milk of Magnesium has both a laxative and an antacid action. It helps relieve the symptoms associated with excess acid such as flatulence, heartburn and indigestion as well as constipation that occurs occasionally.

0



6	Secure a burette in a clamp and stand.			
7	Carefully pour the hydrochloric acid into	o the burette.		
	Make sure the burette is at your eye lev top of the burette	el and the filter f	unnel is lifted slig	htly from the
8	Pour the hydrochloric acid into the bure	ette until it is abou	ut 1 cm above the	e 0 ml mark.
9	Run the hydrochloric acid through the t level falls below this, but record the pre	ap of the burette cise volume to th	is on 0 (It does no e nearest 0.05 ml	ot matter if the).
10	Open the tap of the burette and run the hydrochloric acid into the flask SLOWLY.			
11	When the liquid begins to have swirls of pink, add the hydrochloric acid very slowly; then one drop at a time			
12	Record the volume on the burette in the table below when the methyl orange turns ORANGE. The contents of the flask will also become clear at this point (instead of milky).			
13	Repeat the titration twice so that you h	ave three sets of	results.	
14	14 Record your results in the table below. Calculate the average volume of hydrochloric acid needed to neutralise the Milk of Magnesia.			
		First titration	Second titration	Third titration
Final	volume on burette in ml	25.5	23.2	24
Start	volume on burette in ml	0	0	0
Volun	Volume of hydrochloric acid used in ml 25.5 23.2 24			24

15 Calculate the amount of magnesium hydroxide in a dose (5 ml):

Average volume of hydrochloric acid used in ml

Amount of magnesium hydroxide in one dose = average titration volume x 17.4 mg

24.23333

My figure is very close to what's on the bottle.

Scientists would use an automatic process to do their titrations.

LO6: Be able to determine the concentration of coloured substances in solution

- select indicator and equipment setting it up correctly
- take and record measurements
- draw calibration curve and determine concentration of a substance
- type of instrumental technique to improve analysis of samples

Health Scenario

Iron is needed for healthy blood so our diet needs foods that have iron in it.

Finding the amount of iron in cornflakes using colorimetry

Iron is needed for production of red blood cells and is therefore necessary to keep us healthy.

Cornflakes have added iron in the form of tiny iron filings, which dissolve in the acid in our stomachs.

Stage 1: Extracting the iron from cornflakes

- 1 Grind some cornflakes in a mortar.
- 2 Weigh a crucible.
- 3 Transfer about 4 g of the cornflakes to a crucible. Record the mass to the nearest 0.01 g.
- 4 Place the crucible on a pipe-clay triangle on a tripod.
- 5 Heat the crucible strongly with a roaring Bunsen flame.





- 6 Heat the crushed cornflakes until they are reduced to a grey ash.
- 7 Allow the contents of the crucible to cool fully.

Stage 2: Setting up standard reference solutions

- 1 Label five test tubes 1 5.
- 2 Make up the standard reference solutions as shown in the information below.

[Use 10 ml syringes to measure out the distilled water and iron(III) nitrate solution and potassium thiocyanate solution.

Use a 2 ml syringe to measure out the potassium thiocyanate solution.]

	Tube 1	Tube 2	Tube 3	Tube 4	Tube 5
Distilled water (ml)	20	15	10	5	0
Iron nitrate solution (ml)	0	5	10	15	20
Potassium thiocyanate solution (ml)	2.5	2.5	2.5	2.5	2.5
Concentration of iron(III), equivalent to, in mg/100 g of cornflakes	0	7	14	21	28

- 1 Put the cold ash from the crucible into a 100 ml beaker.
- 2 Rinse the crucible with 5 ml of nitric acid and add the washings to the beaker.
- 3 Repeat rinse again with another 5 ml of acid.
- 4 Use a glass rod to stir the contents of the beaker for 5 minutes.

The iron in the ash reacts with the acid to form iron nitrate.

- 5 Add 10 ml of water to the beaker. Mix well.
- 6 Filter the mixture in the beaker into a test tube labelled 'sample'.
- 7 Measure out 2.5 ml of potassium thiocyanate solution using a 5 ml syringe. Add this to the test tube.

Stage 4: Measuring the concentration using the colorimeter

- 1 Pour some of the contents of Tube 1 into the cuvette so the level is about 5 mm from the top.
- 2 Handle the cuvette by its frosted edge and place it into the colorimeter.
- 3 Check that the colorimeter is set on the 'absorbance' scale. Press 'zero'.
- 4 Pipette some of Tube 2 into a clean cuvette.
- 4 Place the cuvette in the colorimeter and take a reading. Record it in your table.
- 5 Repeat the process with Tubes 2-5.
- 6 Pipette some of the sample prepared in Stage 3 into a cuvette and record the absorbance.

Concentration of iron(III), equivalent to, in mg/100 g cornflakes	Absorbance reading
0	0
7	0.12
14	0.23
21	0.39
28	0.52

Cornflakes sample	0.4



Marking commentary on MB1 sample learner work

Basic understanding and skill shown when preparing samples. (Good level of skill shown when preparing serial dilutions, with some teacher intervention). Some comments on hazards and risks and support needed to work safely. Observations recorded at suitable level on provision of support. Some conclusions drawn from patterns in data. Limited comments on the quality of data.

Suggested improvements to progress sample learner work to MB2

Throughout the candidate has been supported by the teacher to achieve MB2. The candidate has to show both the understanding of the science and skills when carrying out practical investigations; this would be shown by independently planning the practical. A more formal risk assessment is required showing how most risks can be managed at MB2 although there can be some support when actually undertaking the practical but it should be done without any significant accident.

Measurements need to be recorded in an appropriate format with the correct headings and units at MB2.

At MB2 data needs to be discussed including accuracy and sources of error, linked to the methods of collection, limitations in the methods of data collection identified and suggestions for improvements given rather than just giving a simple statement.

Learning Objective 2 - Be able to separate and identify the substances present in a mixture

Marking commentary on MB1 sample learner work

Chromatography carried out with significant support. Rf values calculated, again with significant support. HPLC and GC identified as means of improving separation.

Suggested improvements to progress sample learner work to MB2

Chromatography was carried out with significant support. At MB2 independence needs to be shown this would be supported by a plan that could be followed by another person and a risk assessment giving how most risks could be reduced. It is difficult to follow the recorded measurements, a table of results with the correct headings and units would be expected at MB2. Although Rf values have been calculated the equation was given and support was given throughout. At MB2 the candidate should know the correct equation to use and it is expected they would calculate at least one outcome correctly by them. Although two alternative techniques have been quoted some description is required at MB2.

Learning Objective 3 - Be able to examine and record features of samples

Marking commentary on MB1 sample learner work

Support needed to view hair samples at high power. Calculations of magnification used on microscope made. Scanning electron microscopy as an improvement suggested.

Suggested improvements to progress sample learner work to MB2

Significant support was needed when viewing the hair and a photograph given. At MB2 little support should be needed to use the microscope and a drawing should be made (or at least labelling on a photograph).

At MB2 the candidate needs to know the equations for calculating magnification and scale and be able to derive one outcome correctly and not need support throughout.

Although an alternative technique has been quoted some description is required at MB2.

Learning Objective 4 - Be able to identify cations and anions in samples

Marking commentary on MB1 sample learner work

Tests carried out with support. Observations recorded, but not all accurately. Type of instrumental analysis quoted.

Suggested improvements to progress sample learner work to MB2

Tests were carried out with support. At MB2 the candidate must independently select the necessary equipment to carry out analyses, this would be supported by a plan that could be followed by another person and a risk assessment giving how most risks could be reduced, although little support could be given to set up correctly.

Observations were recorded, but not all accurately. At MB2 observations should be recorded using an appropriate level of detail and in an appropriate format rather than using a simple word giving little accuracy or leaving a blank.

Although an alternative technique has been quoted some description is required at MB2.

Learning Objective 5 - Be able to determine the concentration of an acid or base using titration

Marking commentary on MB1 sample learner work

Help given throughout. Basic drop-by-drop titration performed and result recorded. Calculations made with support. Comment made on automated process used by scientists.

Suggested improvements to progress sample learner work to MB2

The candidate was provided with method and equipment in the form of a help sheet and significant support needed to set it up to take measurements. At MB2 the candidate cannot be supplied with a help sheet they have to show some independence. The candidate has been provided with equations but data has been substituted correctly and some calculations carried out correctly. Again at MB2 a help sheet cannot be provided although some help can be given when manipulating the equations. Although an alternative technique has been quoted some description is required at MB2.

Learning Objective 6 - Be able to determine the concentration of coloured substances in solution

Marking commentary on MB1 sample learner work

Help given throughout. Standards made with reasonable accuracy when provided with support. Measurements taken and recorded. Calibration curve drawn with Microsoft Excel, but (incorrect) line of best fit added manually.

Suggested improvements to progress sample learner work to MB2

Support has been given by supplying a help sheet, at MB2 independence needs to be shown and a help sheet cannot be supplied. When drawing the calibration curve drawn, suitable scales and minor errors only in plotting of points; and an appropriate line of best fit drawn, at MB1 help can be given to do this.

At MB2 when using the calibration curve only little support can be given rather than significant support to determine the concentration of a substance in a solution.

Sample Learner Work Marking Band 3

SAMPLE LEARNER WORK

Unit R074: How scientists use analytical techniques to collect data

Overarching Learning Objective

LO1: Be able to apply the principles of good laboratory practice

- prepare samples and standard solutions and calibration
- carries out techniques required
- risk assessment and work safely
- analyses and justifying the techniques
- evaluate outcomes

LO2: Be able to separate and identify the substances present in a mixture

- select and set up equipment
- take and record measurements
- calculate Rf values
- types of chromatography to improve analysis

Forensic Science Scenario

A suicide note had been left at the crime scene. There were two pens in the person's flat.

I wanted to see if either of the pens had been used to write the note.

I have been provided with samples from the two pens and from the suicide note. The samples were prepared by putting refills from the pens in ethanol overnight, the extract from the note by soaking it in ethanol, and then concentrating the extract. I am going to prepare chromatograms from them.

Analysis of ink samples by chromatography

Method

- 1 I first carried out a Risk Assessment for this procedure and got this checked by my teacher.
- 2 I took a thin-layer chromatography (TLC) plate (or chromatography paper), and ruled a line 25 mm from the bottom.
- 3 I made up the solvent system and poured this into the chromatography tank. I did this in a fume cupboard.
- 4 I put the cover on the tank and left it undisturbed in the fume cupboard so the tank became saturated with the solvent vapour.
- 5 Using a micropipette, I placed a small spot of the ink extracted from the letter on the line I had drawn. I built up the spot by adding a drop, letting it evaporate, and then adding another, etc. This was because the ink wasn't very concentrated.
- 6 I spotted one of the inks obtained from one of the pens on another TLC plate, and then the ink from the second pen on another.
- 7 The spots were allowed to dry thoroughly.
- 8 The TLC plate was then placed in the solvent in the tank. I had worked out the volume of solvent I needed so that when I put it in the tank, the level of liquid did not cover and dissolve the spots.
- 9 I then put the lid on the tank. I left the chromatography tank undisturbed but monitored the level of the solvent.
- 10 I allowed the solvent to run up the plate until it was about 30 mm from the top. I removed the cover and quickly before the solvent began to evaporate marked the point reached by the solvent.
- 11 I removed the TLC plate and allowed it to dry on some paper towels in the fume cupboard.
- 12 I scanned the TLC plates, then drew around the spots and re-scanned them. My chromatograms are on pages 17 and 18.

Chemical or activity	Hazard and associated risk	Reducing risk	Comments
Butan-1-ol	Irritant – may irritate respiratory system, skin and eyes. Vapour may cause drowsiness. Risk of catching fire.	Wear eye protection Use in fume cupboard	Make sure CLEAPSS Hazcardss are available. These are on the p: drive. Seek medical help if you come into contact with the liquid or it splashes in your eyes.
Ethanol, absolute	Highly flammable. Breathing vapour may result in sleepiness.	Wear eye protection. Use in fume cupboard and keep away from naked flames	Make sure CLEAPSS Hazcardss are available. These are on the p: drive. Seek medical help if you come into contact with the liquid or it splashes in your eyes.
Ammonia 880	Corrosive. Causes burns. Dangerous to eyes. Causes severe damage if swallowed.	Wear eye protection. Wear gloves. Use in fume cupboard.	Make sure CLEAPSS Hazcardss are available. These are on the p: drive. Remove the casualty to fresh air if inhaled. Seek medical help if you come into contact with the liquid or it splashes in your eyes.
Inks	Low hazard.	Handle carefully.	
Glassware – chromatography tank,	The glass could cut if any of the equipment	Careful handling.	Make sure the broken glass kit is available. Record accidents and



micropipettes

gets broken.

ink extracted from the note



ink extracted from the BIC pen



seek medical help.

ink extracted from the Staedtler pen



The ink on the note was obviously a match to the Staedtler pen because the spots were in the same position. They also had a different range of colours to the BIC pen. To add further proof, I calculated the Rf values. The Rf is the distance moved by the spot relative to that moved by the solvent:

Rf value = <u>distance moved by spot</u> distance moved by solvent

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Calculations

Ink from note:

Spot	Distance moved by solvent in cm	Distance moved by spot in cm	Rf value
1 Yellow	15.85	5.75	0.36
2 Blue	15.85	8.8	0.56
3 Purple	15.85	9.25	0.58
4 Violet	15.85	11.8	0.74
5 Red-purple	15.85	12.6	0.79
6 Orange	15.85	12.85	0.81
7 Blue	15.85	13.75	0.87
8 Black	15.85	14.45	0.91

Ink from BIC pen:

Spot	Distance moved by solvent in cm	Distance moved by spot in cm	Rf value
1 Blue	15.75	11.95	0.76
2 Purple	15.75	12.5	0.79
3 Blue	15.75	12.85	0.82
4 Purple	15.75	13.2	0.84
5 Yellow	15.75	15.0	0.95

Ink from Staedtler pen:

Spot	Distance moved by solvent in cm	Distance moved by spot in cm	Rf value
1 Blue	15.75	5.5	0.35
2 Blue	15.75	5.85	0.37
3 Blue	15.75	6.2	0.39
4 Blue	15.75	7.15	0.45
5 Blue	15.75	7.75	0.49
6 Blue	15.75	8.4	0.53
7 Purple	15.75	8.75	0.56
8 Violet	15.75	11.15	0.71
9 Red-purple	15.75	11.85	0.75
10 Orange	15.75	12.65	0.80
11 Blue-black	15.75	13.75	0.87
12 Black	15.75	14.4	0.91

I have shaded in the spots that match. The Rf values did not always match perfectly, I think that this is because the original spot from the pen had a higher concentration of dyes than that extracted from the note (here the Rf values were sometimes slightly higher because less dye meant that the spots ran a little farther). I could not understand however, why there was a yellow spot in the ink from the note (spot 1) and blue spots from the Staedtler pen in the same position in the pen itself.

I also found it very difficult (for instance, with spots 4, 5, 6, 7 and 8 on the note chromatogram, and 8, 9, 10, 11 and 12 on the Staedtler pen chromatogram) to judge exactly where the centre of the spot was as the spots were not circular or elliptical. All this means, of course, that a Staedtler pen was use to write the 'suicide' note. It could have been written by someone else's Staedtler pen, or a murderer could have written the note with the victim's pen.

Improvements in chromatographic analysis

Drugs can be analysed by gas chromatography (GC) and high performance liquid chromatography (GC). The type of chromatography used depends on whether the compounds to be separated are decomposed by heat. Gas chromatography is used to separate organic compounds that are volatile. A gas chromatograph consists of:

- a moving gas mobile phase, such as helium, nitrogen or argon
- a port to inject the sample
- a separation column containing the stationary phase, in an oven. The stationary phase is liquid at the column operating temperature
- a detector, and
- a data recording system.

High-performance liquid chromatography (HPLC) is a form of liquid chromatography used to separate compounds that are dissolved in solution.

HPLC instruments consist of:

- a reservoir of the liquid mobile phase
- a pump to move the mobile phase
- an injector
- a separation column packed with a column, and
- a detector.

Compounds are separated by injecting the sample mixture onto the column. The different components in the mixture pass through the column at different rates and separate.

LO3: Be able to examine and record features of samples

- select and set up equipment
- take and record measurements
- calculate magnification and scale
- types of instrumental analysis to improve analysis

Ecology Scenario

Examining Marram grass found at Nicholstan Burrows using a microscope.

Marram grass is adapted to withstand the wind and the Sun, as well as salt from the sea. It's leaves are curled into a C shape to reduce water loss (the opening of the C faces downwind). This is to protect the stomata (these are pores on the leaves that allow the plant to absorb carbon dioxide). The stomata are positioned at the bottom of grooves in the leaves.



You can see these grooves on a photo

Width of leaf in photo = 18.5 mmActual width of leaf = 2.5 mm (measured using a ruler) so the magnification = 18.5/2.5 = 7.4

You can't see much detail on the photograph. I looked at part of a leaf in school with a binocular microscope, which is a low power microscope and I saw more detail of the grooves.

Professional scientists use scanning electron microscopes to examine leaf surfaces. They don't use much more magnification, but SEMs allow you to see surface detail, as these have a large depth of field. [21]

In a scanning electron micrograph of a Marram grass leaf, you would see the grooves and the hairs on the ridges. The hairs reduce air movement and reduce water loss.

In school, I looked at a cross-section of a Marram grass leaf.

I calculated the magnification using an eyepiece graticule in my microscope. I need to calibrate it so that if I count the divisions on the eyepiece graticule when I'm looking at a slide, I can find out the true measurement of the structure I'm looking at.

Calibrating my eyepiece graticule

I did this by using an eyepiece graticule in the eyepiece of my microscope, and a stage micrometer, which is a microscope slide with an accurate scale engraved on it.

The stage micrometer has 1 mm divided into 100 divisions. There are 100 divisions on the eyepiece graticule. I placed the stage micrometer on the stage of the microscope, lining up the zero with the zero on the eyepiece graticule.

At x100 magnification, there were 100 stage micrometer divisions for 100 divisions on the eyepiece graticule. 100 divisions on the eyepiece graticule = 1 mm on the stage micrometer.

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1 division on my eyepiece graticule= $1/100 \text{ mm} = 10 \mu \text{m}$.

At x400 magnification, there were 25.5 stage micrometer divisions for 100 divisions on the eyepiece graticule.

Each division on the stage micrometer	= (1 ÷ 100) mm
	= 10 µm
25.5 divisions on the stage micrometer	= (25.5 x 10) μm
255 µm	

1 division on my eyepiece graticule = $255/100 \ \mu m$ = 2.55 μm

This diagram shows my calibration of the eyepiece graticule at x400 magnification. I can now use this to estimate the size of any object I look at with my microscope.



At x100 magnification, I estimated the diameter of the Marram grass leaf was 202 eyepiece graticule divisions.

1 division on my eyepiece graticule = $1/100 \text{ mm} = 10 \mu \text{m}$.

The diameter of the leaf was 202 x 10 μ m = 2020 μ m = 2.02 mm.

At x400 magnification, I estimated the width of one of the leaf folds:

Diameter of fold 1 = 115 eyepiece graticule divisions

Diameter of fold 2 = 90 eyepiece graticule divisions

Diameter of fold 3 = 98 eyepiece graticule divisions

Diameter of fold 4 = 74 eyepiece graticule divisions

Diameter of fold 5 = 86 eyepiece graticule divisions

Average diameter of fold = 93 eyepiece graticule divisions.

1 division on my eyepiece graticule = $2.55 \,\mu$ m.

The average diameter of a leaf fold = (2.55 x 93) μ m = 237 μ m.

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OCR LEVEL 1/2 CAMBRIDGE NATIONALS SCIENCE IN THE WORKPLACE



Improvements in instrumental analysis

Scanning electron microscopy (SEM) is where the tissue is preserved (fixed), and coated with heavy metals. The specimen is bombarded with electrons, which are reflected onto a fluorescent screen, building a 2D image of the surface of the specimen.

Transmission electron microscopy is where the tissue/cells are fixed, embedded in resin and cut into ultra-thin sections using an instrument called a microtome. The sections are stained and put into the electron microscope. The microscope uses a beam of electrons instead of light to view the specimen. Electrons have a much smaller wavelength, so a much greater resolution and magnification are possible than using a light microscope. Electron micrographs reveal many different structures than are possible with the light microscope. An image is focused using condensers instead of lenses. An image is produced on a fluorescent screen and this is photographed. Photographic enlargement makes it possible to increase the magnification further. A drawback is that you can't view living specimens with electron microscopes, and the microscope has to be evacuated to view the specimen.

LO4: Be able to identify cations and anions in samples

- select and set up equipment
- take and record observations
- types of instrumental techniques to improve analysis

Forensic Science Scenario

Do the shoeprints provide any clues? The cyanide evidence suggests that it could have been murder.

Some shoeprints of soil were left at the crime scene.

Soil samples have been taken for analysis. Five suspects have been located who were seen in the area at the time.

Samples of soil were also taken from the soles of five suspects who were seen around the house at the time of the burglary.

I added the soil samples to about 5 cm³ of deionised water in test tubes, shook them and allowed them to stand overnight, then decanted off the liquid.

Qualitative analysis of samples

Qualitative analyses are carried out when the analyst wants to know the types of chemicals in a sample (but not how much of them).

I are going to do three types of test:

- flame tests for cations
- chemical tests for cations
- chemical tests for anions.

Chemical or activity	Hazard and associated risk	Reducing risk	Comments
Hydrochloric acid (concentrated)	Corrosive. Will burn if it comes into contact with skin or eyes.	Wear eye protection and gloves.	Make sure CLEAPSS Hazcards are available. These are on the p: drive. Seek medical help IMMEDIATELY if the liquid splashes on your skin or in your eyes.
Barium chloride	Toxic by ingestion.	Wear eye protection. Use with care. Avoid contact with skin or eyes and wash hands after use. Dispose of in beaker provided.	Make sure CLEAPSS Hazcards are available. These are on the p: drive. Seek medical help if you come into contact with the liquid or it splashes in your eyes.
Calcium chloride	Irritant.	Wear eye protection. Use with care.	Make sure CLEAPSS Hazcards are available. These are on the p: drive. Wash hands if it comes into contact with skin.
Copper(II) sulphate	Irritant.	Wear eye protection. Use with care. Avoid contact with skin or eyes and wash hands after use. Dispose of in beaker provided.	Make sure CLEAPSS Hazcards are available. These are on the p: drive. Seek medical help if you come into contact with the liquid or it splashes in your eyes.
Iron(III) chloride	Corrosive. Will burn if it comes into contact with skin or eyes.	Wear eye protection. Use with care. Avoid contact with skin or eyes and wash hands after use.	Make sure CLEAPSS Hazards are available. These are on the p: drive. Seek medical help if you come into contact with the liquid or it splashes in your eyes.
Lead(II) chloride	Toxic.	Wear eye protection. Use with care. Avoid contact with skin or eyes and wash hands after use. Dispose of in beaker provided.	Make sure CLEAPSS Hazcards are available. These are on the p: drive. Seek medical help if you come into contact with the liquid or it splashes in your eyes.
Lithium chloride	Irritant.	Wear eye protection. Use with care. Avoid contact with skin or eyes and wash hands after use.	Make sure CLEAPSS Hazcards are available. These are on the p: drive. Wash hands if it comes into contact with skin.
Potassium chloride	Irritant.	Wear eye protection. Use with care. Avoid contact with skin or eyes and wash hands after use.	Make sure CLEAPSS Hazcards are available. These are on the p: drive. Wash hands if it comes into contact with skin.
Potassium sulphate	Irritant.	Wear eye protection. Use with care. Avoid contact with skin or eyes and wash hands after use.	Make sure CLEAPSS Hazcards are available. These are on the p: drive. Wash hands if it comes into contact with skin.
Silver nitrate solution	Corrosive. Will burn if it comes into contact with skin or eyes.	Wear eye protection. Use with care. Avoid contact with skin or eyes and wash hands after use. Dispose of in beaker provided.	Make sure CLEAPSS Hazcards are available. These are on the p: drive. Seek medical help if you come into contact with the liquid or it splashes in your eyes.

SAMPLE LEARNER WORK

Chemical or activity	Hazard and associated risk	Reducing risk	Comments
Sodium carbonate	Irritant.	Wear eye protection. Use with care. Avoid contact with skin or eyes and wash hands after use.	Make sure CLEAPSS Hazcards are available. These are on the p: drive. Wash hands if it comes into contact with skin.
Sodium chloride	Irritant.	Wear eye protection. Use with care. Avoid contact with skin or eyes and wash hands after use.	Make sure CLEAPSS Hazcards are available. These are on the p: drive. Wash hands if it comes into contact with skin.
Sodium hydroxide solution	Irritant at this concentration (0.10 mol dm ⁻³).	Wear eye protection. Use with care. Avoid contact with skin or eyes and wash hands after use.	Make sure CLEAPSS Hazcards are available. These are on the p: drive. Seek medical help if you come into contact with the liquid or it splashes in your eyes.
Unknown chemicals (in soil) from suspects and crime scene	Unknown. Treat as irritant/ harmful/toxic.	Wear eye protection. Use with care. Avoid contact with skin or eyes and wash hands after use. Dispose of in beaker provided.	Make sure CLEAPSS Hazcards are available. These are on the p: drive. Seek medical help if you come into contact with the liquid or it splashes in your eyes.
Unknown microorganisms in soil from suspects and crime scene	Unknown – could contain pathogenic microorganisms.	Use with care. Avoid contact with skin or eyes and wash hands after use. Dispose of in beaker provided (technician will autoclave).	
Wire loop	Risk of burning self, hair, others.	Wear eye protection. Use with care. Put on heatproof mat after use.	Inform teacher and seek medical help if you get burnt.
Bunsen burner	Risk of burning self, hair, others.	Wear eye protection. Use with care. Make sure that the Bunsen is positioned carefully on the bench on a heatproof mat. Put on the yellow flame when not in use.	Inform teacher and seek medical help if you get burnt.
Glassware – test tubes and boiling tubes	The glass could cut if any of the equipment gets broken.	Careful handling. Do not walk around with test tube racks.	Make sure the broken glass kit is available. Record accidents and seek medical help.

Flame tests

Equipment list

- goggles
- apron
- gloves wire loop
- heatproof mat
- beaker
- concentrated hydrochloric acid
- Bunsen burner
- chemical samples to test
- chemical samples from suspects
- chemical sample from crime scene

Method

- 1 Put goggles and gloves on to prevent injury from chemicals or the Bunsen flame.
- 2 Clean the wire loop in a roaring Bunsen flame.
- 3 Dip the wire loop in the concentrated hydrochloric acid in the beaker.
- 4 Dip the wire loop in the first one of the chemical samples to test, so as to pick some of the chemical up.
- 5 Put the wire loop just above the centre of the Bunsen flame.
- 6 Observe and record the colour of the flame.
- 7 Clean the loop before picking up and testing some of the next chemical samples.
- 8 Test the sample taken from the crime scene.

Results

Flame tests.

Commis		Matalian measure		
Sample	Test 1	Test 2	Test 3	Metal ion present
barium (chloride)	apple green	apple green	apple green	barium
calcium (chloride)	brick red	brick red	brick red	calcium
copper (sulphate)	blue-green	blue-green	blue-green	copper
lithium (chloride)	red	red	red	lithium
potassium (chloride)	lilac	lilac	lilac	potassium
sodium (chloride)	orange-yellow	orange-yellow	orange-yellow	sodium
from gardener	lilac	lilac	lilac	potassium
from worker at copper smelter	blue-green	blue-green	blue-green	copper
from worker at iron foundry	no colour	no colour	no colour	no conclusion
from worker who had been salting the roads	orange-yellow	orange-yellow	orange-yellow	sodium
from builder	brick red	brick red	brick red	calcium
from crime scene	lilac	lilac	lilac	potassium

Chemical tests for cations

Equipment list

- test tubes
- test tube rack
- dilute sodium hydroxide solution
- pipette
- chemical samples

Method

- 1 Pour 1 cm³ of the first chemical sample into a test tube.
- 2 Add a few drops of dilute sodium hydroxide solution.
- 3 Observe and record what happens in the test tube.
- 4 Repeat the test with the other chemical sample.
- 5 Dissolve a small amount of the chemical from the crime scene in 1cm³ of distilled water.
- 6 Add a few drops of sodium hydroxide solution and observe and record what happens in the test tube.

Chemical tests for cations

Sample	Colour when sodium hydroxide solution added	Metal ion present
calcium chloride	white precipitate	calcium
copper(II) (sulphate)	pale blue jelly-like precipitate	copper(II)
iron(III) (chloride)	red-brown precipitate	iron(III)
lead(II) chloride	off-white precipitate	lead(II)
from gardener	no change. Solutions mixed	potassium
from worker at copper smelter	pale blue jelly-like precipitate	copper(II)
from worker at iron foundry	red-brown precipitate	iron(III)
from worker who had been salting the roads	no change. Solutions mixed	no conclusion
from builder	white precipitate	calcium (supported by flame test)
from crime scene	no change. Solutions mixed	no conclusion

Chemical tests for anions

Equipment list

- test tubes
- test tube rack
- dilute nitric acid
- acidified barium chloride solution
- acidified silver nitrate solution
- pipette
- chemical samples

Method

- 1 Pour dilute nitric acid into a test tube to the depth of 3-4 cm.
- 2 Add the first sample to the liquid. If the sample fizzes, it must be a carbonate.
- 3 If the sample does not fizz, pour half of the mixture into a clean test tube, and the other half into another clean test tube.
- 4 To one test tube, add a few drops of silver nitrate solution. If it goes cloudy, it must contain chloride as the non-metal part (anion).
- 5 To the other test tube, add a few drops of barium chloride solution. If the liquid goes cloudy, the sample must contain sulphate as the non-metal part.
- 6 Repeat the tests with all the chemical samples, and samples from the suspects and the crime scene.

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Chemical tests for anions

Sample	Reaction with nitric acid	Reaction with barium chloride	Reaction with silver nitrate	Anion present
(sodium) carbonate	fizzed	-	-	carbonate
(sodium) chloride	no reaction	no reaction	white precipitate went dark in light	chloride
(potassium) sulphate	no reaction	white precipitate	no reaction	sulphate
from gardener	no reaction	white precipitate	no reaction	sulphate
from worker at copper smelter	no reaction	white precipitate	no reaction	sulphate
from worker at iron foundry	no reaction	no reaction	white precipitate went dark in light	chloride
from worker who had been salting the roads	no reaction	no reaction	white precipitate went dark in light	chloride
from builder	no reaction	no reaction	white precipitate went dark in light	chloride
from crime scene	no reaction	white precipitate	no reaction	sulphate

Conclusion

The flame test colours and the precipitates produced in the chemical tests helped us to identify the chemicals in the samples. The results from the tests narrow the suspects down to one person – the gardener. The chemical sample from crime scene contained potassium sulphate. We know this because the chemical produced a lilac flame test and a white precipitate with barium chloride solution. These are the same results as the soil from the gardener's shoes.

The chemical samples from the other suspects' shoes:

Worker at copper smelter				
metal ion	the sample produced a blue-green flame colour a blue precipitate with sodium hydroxide solution			
non-metal ion	a white precipitate with barium chloride solution			
Therefore, the chemical was copper (II) su	Iphate.			
Chemistry	The copper (II) ions reacted with the hydroxide ions to form insoluble copper hydroxide. The sulphate ions reacted with the barium ions to form insoluble barium sulphate.			
Worker at iron foundry				
metal ion	the sample produced a red-brown precipitate with sodium hydroxide solution			
non-metal ion	white precipitate with silver nitrate solution			
Therefore, the chemical was iron(III) chloride.				
Chemistry	The iron(III) ions reacted with the hydroxide ions to form insoluble copper hydroxide. The chloride ions reacted with the silver ions to form insoluble silver chloride, which darkens on exposure to light (as it's turned to elemental silver).			

Worker who had been salting the roads				
metal ion	the sample produced a orange-yellow flame colour			
non-metal ion	a white precipitate with silver nitrate solution			
Therefore, the chemical was sodium chlo	ride.			
Chemistry	The chloride ions reacted with the silver ions form insoluble silver chloride, which darkens on exposure to light (as it's turned to elemental silver).			
The builder				
metal ion	the sample produced a brick red flame colour a white precipitate with sodium hydroxide solution			
non-metal ion	a white precipitate with silver nitrate solution			
Therefore, the chemical was calcium chlo	pride.			
Chemistry	The calcium ions reacted with the hydroxide ions to form insoluble calcium hydroxide. This conclusion was supported by the flame test, as several metal ions give white precipitates with sodium hydroxide. The chloride ions reacted with the silver ions to form insoluble silver chloride, which darkens on exposure to light (as it's turned to elemental silver).			

Evaluation

The results recorded were repeatable because the experiments were done three times and the same results were produced each time. It was important to repeat the tests as some groups got different colours with their flame tests. Sometimes the samples gave a yellow colour flame when a sample was first tested. This could have been that there were traces of sodium left on the loops so these were cleaned again and the sample retested. This technique could have been improved by viewing the flame colour through cobalt blue glass, which removes the yellow colour from the flame.

Improvements in instrumental techniques

Analytical chemists would only ever use these tests as a quick diagnostic test. They are not as sensitive as the instrumental techniques available. Also, they are not always specific to one ion, e.g. the sodium hydroxide test for metal ions. To identify many ions, chemists would use liquid chromatography (LC). Conventional LC is most commonly used in preparative scale work to purify and isolate some components of a mixture. It is also used in ultra-trace separations (where small disposable columns are used once and then discarded). Analytical separations of solutions for detection or quantification typically use more sophisticated instruments. HPLC instruments use a pump to force the mobile phase through and provide higher resolution and faster analysis time.

Atomic emission spectroscopy (AES) or optical emission spectroscopy (OES) uses quantitative measurement of the optical emission from excited atoms to determine the concentration of metal ions. Atoms in solution are drawn into the instrument into the excitation region, where they are desolated, vaporised, and atomised by a flame, discharge, or plasma. These high-temperature atomisation sources provide sufficient energy to promote the atoms into high energy levels. The atoms decay back to lower levels by emitting light. Since the transitions are between distinct atomic energy levels, the emission lines in the spectra are narrow. The spectral separation of nearby atomic transitions requires a high-resolution spectrometer.

Inductively coupled plasma (ICP) is a very high temperature (7000-8000K) excitation source that efficiently desolates, vaporises, excites, and ionises atoms in the sample. Molecular interferences are greatly reduced with this excitation source.

LO5: Be able to determine the concentration of an acid or base using titration

- select indicator and equipment setting it up correctly
- take and record measurements
- process data using mathematical techniques
- type of instrumental technique to improve analysis of samples by titration

Ecology Scenario

Quantitative Analysis of water samples. Port Talbot is affected by acid rain. The sulphuric acid concentration in a sample of water collected at Port Talbot will be measured.

Equipment list

- graduated pipette (25cm³)
- sodium hydroxide solution, 0.001 mol dm³
- burette
- indicator (bromothymol blue)
- conical flask
- distilled water
- 'acid rain' samples
- filter funnel
- clamp and stand
- goggles

Risk Assessment: Quantitative Analysis

Source of hazard	Hazard and risk	Risk and people at risk	Safety precautions	Emergency action
Sodium hydroxide solution (0.001 mol dm ⁻³)	Irritant.	Could get spilt or splashed and irritate the skin or eyes.	Handle carefully. Wear eye protection. Use with care and avoid contact with skin and mouth. Wash hands after use.	Inform teacher of accident. It may be necessary to get medical help.
Acid rain/ sulphuric acid	Irritant.	Could get spilt or splashed and irritate the skin or eyes.	Wear goggles. Use with care. Wash hands after use.	Inform teacher of accident. It may be necessary to get medical help.
Bromothymol blue	Low hazard.	Not usually classified as hazardous but should be used with caution.	Wear goggles. Skin contamination should be avoided. Wash hands after use.	Inform teacher of accident.
Glassware (burette, pipette and conical flask)	Broken glass can cut. Cuts can become infected.	Risk of cuts.	Handle with care.	Inform teacher of accident. It may be necessary to get medical help.

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SAMPLE LEARN				AMPLE LEARNER WOR
Source of hazard	Hazard and risk	Risk and people at risk	Safety precautions	Emergency action
Filling burette	Acid rain is irritant.	Burette could topple over. Burette could overflow if acid rain not allowed to drain from funnel. Acid rain could get spilt or splashed and irritate the skin or eyes.	Put burette at eye level to fill. Lift filter funnel slightly when filling. Handle carefully. Wear eye protection. Use very small volumes.	Inform teacher of accident. It may be necessary to get medical help.

Method

I first secured a burette into the clamp and stand, and washed it through twice with some of the acid rain sample to be analysed. I used a washed-out pipette to measure out 25 cm³ of sodium hydroxide solution into a clean 250 cm³ conical flask. To this I added 3 to 4 drops of bromothymol blue indicator. I chose bromothymol blue as this has an equivalence point of around pH 7. The reaction between sulphuric acid and sodium hydroxide is one between a strong acid and a strong base, which means that the salt formed is neutral.

Choosing an indicator strong acid + e.g. ammonium sulphate	weak base
weak acid + e.g. sodium ethanoate	strong base

Using a filter funnel, I added my sample of acid rain to the burette and allowed some to run out until it reached the 0 cm³ mark. I then slowly titrated the acid rain into the sodium hydroxide solution, mixing it constantly. When the liquid in the conical flask began to have swirls of the indicator changing colour (turning yellow), I began to add the acid rain one drop at a time. I stopped adding the acid when the liquid had reached the required colour – green – and recorded the volume to the nearest 0.05 cm³. I made a decision to repeat the titration twice for the same sample or until I got two concordant readings. That's within 0.1 cm³ of each other. I then repeated this with another two acid rain samples.

Results

Acid rain sample 1

Ruratta reading	Titration volume (cm ³)			
burette reading	1st	2nd	3rd	Average
Final reading	23.50	47.20	23.70	volume (cm ³)
Start reading	0.00	23.50	0.00	
Volume used	23.50	23.70	23.70	23.70

Acid rain sample 2

Puratta reading		Titration volume (cm ³)	
Burelle reading	1st	2nd	Average
Final reading	21.50	43.10	volume (cm ³)
Start reading	0.00	21.50	
Volume used	21.50	21.60	21.55

Acid rain sample 3

Duratta readina	Titration volume (cm ³)			
Burelle reading	1st	2nd		Average
Final reading	19.50	39.10		volume (cm ³)
Start reading	0.00	19.50		
Volume used	19.50	19.60		19.55

Calculating the concentration of sulphuric acid in the acid rain samples

Acid rain sample 1

23.70 cm³ of acid rain was needed to neutralise 25.00 cm³ of sodium hydroxide sulphuric acid + sodium hydroxide ► sodium sulphate + water H₂SO₄ + 2NaOH 🕨 Na₂SO₄ + H₂O So 1 mole $H_2SO_4 = 2$ molesNaOH $\underline{MA \times VA} = \underline{MB \times VB}$ Na NB Where: $M_A = concentration of acid$ $M_B = concentration of alkali$ $V_A =$ volume of acid $V_B =$ volume of alkali NA = number of moles of acid NB = number of moles of alkali So 1 mole $H_2SO_4 = 2$ molesNaOH so $N_A = 1$ $N_B = 2$ $V_A = 23.70 \text{ cm}^3$ $V_B = 25.00 \text{ cm}^3$ $M_B = 0.001 \text{ mol } dm^3$ $\underline{MA \times VA} = \underline{MB \times VB}$ Na NB $Ma \times 23.70 = 0.001 \times 25.00$ 1 2 $M_A \ge 23.70 = 0.001 \ge 12.5$ $M_A \times 23.70 = 0.0125$ = 0.0125 Ma 23.70 Ma $= 0.00053 \text{ mol dm}^3$ The concentration of sulphuric acid in the first acid rain sample = 0.00053 mol dm⁻³

The same process was used to calculate the concentrations of the other two samples.

LO6: Be able to determine the concentration of coloured substances in solution

- select indicator and equipment setting it up correctly
- take and record measurements
- draw calibration curve and determine concentration of a substance
- type of instrumental technique to improve analysis of samples

Summary of results

Acid rain sample	Concentration, mol dm ⁻³
1	0.00053
2	0.00058
3	0.00064

Conclusion

The sulphuric acid in the acid rain neutralised the sodium hydroxide. My preliminary testing for cations and anions, and with Universal indicator, suggested that it was sulphuric acid that I was titrating (the only ions I detected were sulphate ions). There were quite large differences in the concentrations from one sample to another but this was not unexpected. The bodies of water were different in volume and there could have been different levels of evaporation from the various bodies of water.

Evaluation

The equivalence points for my titrations were not difficult to obtain because of the indicator I used (bromothymol blue). There was a clear change from blue to green, although I was working with low concentrations of alkali and acid, which made it a bit more difficult. Our results were close to each other, and therefore showed good levels of repeatability. Comparing these with other groups, who collected samples from similar locations, the results showed good levels of repeatability.

I am therefore satisfied with the quality of my results. I could have improved this further, however, by changing the glassware I used. I used class B glassware. I could have used class A. The table below illustrates the differences:

Burette accuracy tolerances			
Capacity, cm ³	Subdivision, cm ³	Class A and precision grade accuracy, ±cm ³	Class B and standard grade accuracy, ±cm ³
10	0.05	0.02	0.04
25	0.10	0.03	0.06
50	0.10	0.05	0.10
100	0.20	0.10	0.20

Improvements in instrumental techniques

In industry, scientists use autotitrators.

This is where the whole process of titration is made much easier by making it automated. You simply add a predetermined amount of reactant and the machine will add the other reactant and measure the products to find the end point. So many samples can be done in no time at all. The accuracy is increased due to the finely calibrated computer instead of your eyes. The amount of hands-on interaction is drastically reduced.

Forensic Science Scenario

At the crime scene there was a mug that had a dried coffee stain at the bottom. There was a faint odour of almonds. The mug could have contained cyanide.

A vial containing a small volume of liquid was found in the rubbish bin. Forensic scientists collected the vial and took it back to the Forensic Science lab.

A forensic scientist reacted 20 cm³ of the liquid with potassium polysulfide. Any cyanide would react to produce potassium thiocyanate. The potassium thiocyanate formed was reacted with iron (III) chloride to produce iron(III) thiocyanate. Iron (III) thiocyanate is deep red. Its concentration can be estimated using colorimetry.

Method

I have been given a deep red solution to analyse, so the vial found at the crime scene must have contained cyanide.

- 1 I poured 50 cm³ of the stock solution of 0.15 mol dm⁻³ into a 100 cm³ beaker.
- 2 I poured 50 cm³ of deionised water into a second 100 cm³.
- 3 I then labelled five clean, dry test tubes with the numbers 1 5.
- 4 I prepared my standard solutions by measuring the following volumes from my stock solution and distilled water beakers. I used graduated pipettes (with filler) to measure the correct amount of standard solution or water into each test tube (using a different pipette for each). The solutions were prepared as follows:

Test tube number	Volume of FeSCN ²⁺ solution in cm ³	Volume of deionised water in cm ³	Final concentration of FeSCN ²⁺ in mol dm ⁻³
1	10	0	0.15
2	8	2	0.12
3	6	4	0.09
4	4	6	0.06
5	2	8	0.03

Then

- 1. I stirred the contents of each test tube carefully, with a clean glass stirring rod each time.
- 2. I switched on the colorimeter, turned the dial to the blue filter and set it to read on the absorbance scale.
- 3. I pipetted some of the deionised water from the beaker into the first cuvette and placed it into the colorimeter. I checked that the cuvettes was dry on the outside before putting it into the colorimeter and handled it only by the sides with frosted edges. I checked that the cuvette was free of bubbles.
- 4. I pressed the 'zero' to zero the colorimeter.
- 5. I then put the cuvettes into the colorimeter in turn and recorded the readings.
- 6. I drew a calibration curve from my set of results.
- 7. I then pipetted the reacted sample from the crime scene into a cuvette and took an absorbance reading from this.

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Risk assessment

Chemical or activity	Hazard and associated risk	Reducing risk	Comments
Iron(III) thiocyanate sample produced from crime scene sample	Irritant. Hazardous in case of skin contact. Hazardous in case of eye contact or ingestion.	Wear eye protection.	Information from [16], based on ammonium thiocyanate. Seek medical help if you come into contact with the liquid or it splashes in your eyes.
Iron(III) thiocyanate standards	Irritant. Hazardous in case of skin contact. Hazardous in case of eye contact or ingestion.	Wear eye protection. Use a pipette filler to measure out.	Information from [16]. Seek medical help if you come into contact with the liquid or it splashes in your eyes.
Test tubes and graduated pipette	The glass could cut if the test tubes get broken	Careful handling. Do not move the test tube rack around.	Make sure the broken glass kit is available. Record accidents and seek medical help.
Colorimeter	Electrical. Possible risk of electric shock.	Keep away from taps. Regular portable appliance testing.	Inform teacher of any accident.

Results

Concentration of FeSCN ²⁺ in mol dm ⁻³	Absorbance
0	0
0.03	0.125
0.06	0.242
0.09	0.364
0.12	0.481
0.15	0.599

Absorbance obtained from crime scene sample = 0.440

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SAMPLE LEARNER WORK Absorbance 0.7 0.8 0.5 0.4 0.3 0.2 0.1 0.0 0.02 0.04 0.06 0.08 0.10 0.12 0.14 0.16 Concentration of FeSCN2+ in mol dm-3

Improving of instrumental technique

Forensic scientists would rarely use a colorimeter in the lab. They would use a spectrophotometer. Whereas a colorimeter measures absorbance (or transmittance) using coloured filters, a spectrophotometer can make measurements at a range of different wavelengths. You can therefore choose a wavelength that will give maximum absorbance for the colour you're measuring.

Learning Objective 1 - Be able to apply the principles of good laboratory practice

Marking commentary on MB3 sample learner work

Thorough understanding and level of skill when preparing samples/ standard solutions/ calibration.

Some support given in preparation of standard solutions.

All tasks carried out independently.

Very good risk assessments produced independently. No incidents during practical work.

All experimental procedures described in detail. Some justification of techniques.

Patterns in data described where appropriate. Some evaluation of data.

Why it was awarded MB3 not MB2

The major difference between MB2 and MB3 is that candidates are fully independent when carrying out practicals whereas some support can be given at MB2.

This will be reflected in the planning detail so any person can follow and understand the science behind the activity.

At MB3 a scientifically drawn diagram is in 2D using a ruler, which is correctly labelled and labels under each other connected to the particular equipment with a straight line will support how the equipment is set up.

When selecting equipment candidates could explain why they have selected the equipment:

• what measurements can be taken

• what accuracy the measurements can be taken to.

Candidates would demonstrate the ability to set up equipment, by

- setting up on a stable platform (table)
- setting up in enough space to carry out measurements
- be able to have a line of sight perpendicular to an instruments scale
- equipment is stable (use of appropriate stand)
- equipment can be reached easily when taking measurements.

The detail of the risk assessment will allow all risks to be managed successfully and with no incidents or accidents and no requirements for teacher intervention when the activity is undertaken so they will refer to both CLEAPSS which explains what is allowed to be done in school and HAZCARDS for information about the chemicals used and what to do if there is an accident must be consulted. At MB2 some risks might not be identified.

For MB3 detailed and critical consideration is given to the data and methods used to obtain them referring to sources of error and quality of data discussed and explained, including accuracy, repeatability and uncertainty, limitations of the method identified and suggestions for improvements justified whereas for MB2 some relevant comments without justification.

At MB2 there will some link between the description of the pattern/trend and the data from which it is derived. The explanation of the trend/pattern must have some science in it and be largely correct. MB3 demands the same but the scientific explanation should be in more depth, showing thorough understanding of the theory involved.

Any uncertainty as to the truth or otherwise of the pattern/trend observed should also be explored.

Learning Objective 2 - Be able to separate and identify the substances present in a mixture

Marking commentary on MB3 sample learner work

Independent selection of equipment from that provided. Measurements taken and recorded to good level of accuracy and precision. Recorded in appropriate format. Data processed - equation selected and Rf values-calculated to appropriate significant figure. HPLC discussed, with HPLC-MS, and use explained.

Why it was awarded MB3 not MB2

At MB2 some support might be given when undertaking the practical and processing the data whereas for MB3 no support is given and the appropriate types of chromatography to improve analysis of samples are explained and evaluated rather than just described. At MB3 candidates consider the appropriate accuracy and precision when recording measurements, this may result in candidates taking further measurements due to the quality of their initial measurements.

Learning Objective 3 - Be able to examine and record features of samples

Marking commentary on MB3 sample learner work

Independent selection of equipment to take measurements and microscope equipment set up correctly to obtain good detail. Magnification and calibration processed independently and accurately.

Scanning electron microscopy (SEM) and transmission electron microscopy explained.

Why it was awarded MB3 not MB2

At MB2 some support might be given when undertaking the practical and processing the data whereas for MB3 no support is given and the appropriate types of instrumental analysis to improve analysis of samples are explained and evaluated rather than just described.

At MB3 candidates consider the appropriate accuracy and precision when recording measurements and scale or scale bars are calculated correctly to appropriate numbers of significant figures rather than recording just what is observed. The detail of biological drawings will fully meet the expected conventions at MB3.

Learning Objective 4 - Be able to identify cations and anions in samples

Marking commentary on MB3 sample learner work

Equipment selected and all qualitative analysis carried out independently. Observations made in appropriate detail. Very good discussion of instrumental techniques to improve analysis.

Why it was awarded MB3 not MB2

At MB2 some support might be given when undertaking the practical and processing the data whereas for MB3 no support is given and the appropriate types of instrumental analysis to improve analysis of samples are explained and evaluated rather than just described.

The observations recorded will be in greater detail which will in turn demonstrate the greater depth of scientific knowledge.

Learning Objective 5 - Be able to determine the concentration of an acid or base using titration

Marking commentary on MB3 sample learner work

Independently selected indicator and equipment which was set up correctly. Measurements taken and recorded including use of correct units. Data processed accurately and independently and; outcomes correct to appropriate numbers of significant figures. Significant evaluation made linked to science and accuracy. Autotitrators explained.

Why it was awarded MB3 not MB2

At MB2 some support might be given when undertaking the practical and processing the data whereas for MB3 no support is given and the appropriate types of instrumental analysis to improve analysis of samples are explained and evaluated rather than just described.

Learning Objective 6 - Be able to determine the concentration of coloured substances in solution

Marking commentary on MB3 sample learner work

Standards made up independently. Calibration curve produced independently and used to find concentration of unknown. Comparison with spectrophotometer made.

Why it was awarded MB3 not MB2

At MB2 some support might be given when undertaking the practical and processing the data whereas for MB3 no support is given and the appropriate types of instrumental analysis to improve analysis of samples are explained and evaluated rather than just described.



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