

Teacher Guide - G628: Sampling, Testing and Processing

Although the title of this unit is 'Sampling, Testing and Processing', the main focus of this unit will be chemistry, with less emphasis on biology and physics.

Suggested Time Allocation:

- Based on 50-60 hours spent on this unit.
- It is suggested that some time is spent on reading and studying the articles from previous papers set for this unit and attempting the questions / using the mark schemes.
- A number of centres reserve the actual case study for the current series examination paper until closer to the date of the examination.
- The use of the examiners' reports is recommended as these encourage candidates to focus on areas of generally identified weakness. The main thrust of the questions is on the sampling, testing and processing aspects of the topic and less on the subject knowledge behind it.
- There is a need for practical work that is relevant to the title of this unit, as well as 'theory' connected with past paper material.

Some centres develop practical activities based on previous case study materials. Sampling may involve homogeneous or heterogeneous mixtures and the methods of separation related to these. Candidates should have experience of simple filtration, filtration under reduced pressure and the use of a centrifuge, where possible.

- A simple exercise would be an inhomogeneous mixture of powdered copper and black copper oxide (copper (II) oxide). Addition of dilute sulfuric acid, with warming, would remove the oxide as aqueous copper sulfate and leave the copper. Weighing a sample of the original mixture and then finding the mass of the dried copper metal residue would provide information about the percentage of copper (and copper (II) oxide) in the mixture.
- This experiment could be extended to involve colorimetry of the copper sulfate solution produced from the copper (II) oxide. A range of answers would be produced if the original sample was not well mixed and this could lead to discussion about the need for representative sampling. Discussion should include possible ways of improving the method and the advantages and disadvantages of different methods of separation.
- An even simpler experiment would be to (poorly) mix iron filings and sulfur, and explore the different ways of separating these two elements. This might include the use of a magnet or adding the mixture to water and washing away the less dense sulfur, leaving the iron.
- Another exercise involves hydrated sodium carbonate crystals (washing soda), $\text{Na}_2\text{CO}_3 \cdot x\text{H}_2\text{O}$, where the value of x is 10. On standing, this material loses water and the number of moles of water becomes less than 10. There are several ways to find the number of moles of water present in a sample.

- One way is to heat a known mass of washing soda to constant mass, and to find the mass of anhydrous sodium carbonate remaining. The percentage mass of solid remaining can be found and compared to given values calculated when x is, for example, 6, 7, 8, or 9.
- Another method might be to add a standard mass of washing soda to an excess of dilute hydrochloric acid and measure the volume of carbon dioxide evolved. The value of x could be read off from a prepared graph of volume of carbon dioxide evolved plotted against values of x .
- A third method might be the titration of a certain mass of washing soda against standard hydrochloric acid. The value of x could be read from a graph of volume of hydrochloric acid used against values of x . A prepared graph could avoid the problem of a titration calculation, if this was preferred.
- Candidates should be asked about each method – its advantages and disadvantages from the point of view of, for example - safety, ease of method, time taken, availability of apparatus and relative accuracy.
- Practical activities can be developed using simple physics experiments.
- For example, the volume of an irregularly shaped object such as a mineral sample is found by using a water displacement method, perhaps by use of a eureka can. If the mass is then found, the density of the mineral can be calculated. This experiment lends itself to discussions about accuracy and the correct use of significant figures.
- Another experiment involves using a bamboo cane and hanging masses at the end (or in the middle) and finding the amount by which the cane 'bends', perhaps until it snaps. The results can be graphed and used for the basis of discussion.
- Experiments with apples have been successfully used to find the acidity present. Variables that can be considered include ripeness, variety of eating apple, size/mass, type of apple (e.g. cooking or eating apples) and age. Students can use books or the internet to compare their answers to published values.

Section 3.9.4: Awareness of test methods

The specification now includes a section entitled 'Awareness of test methods' (3.9.4). With the move away from traditional 'wet' methods of chemical analysis towards instrumental methods, there is a need for candidates to have a basic awareness of some of the more common separation and analytical procedures.

Candidates should have a basic understanding of the use of the following techniques and an outline knowledge of how the results are interpreted. It is not intended that candidates should understand the theory underpinning these techniques in any detail.

Specific comments about individual techniques are given below.

Colorimetry

Many centres will be familiar with the use of colorimeters and it is felt that candidates should know:

- that a suitable filter should be used to give 'optimum' absorbance for the particular coloured solution used (no knowledge of complimentary colours or the colours produced by particular species will be required)
- that absorption/absorbance/'intensity' is directly proportional to concentration
- that a calibration graph is required and that this should be a straight line passing through the origin
- how to use the calibration graph to find the concentration of a test solution.

Chromatography

There are a number of different types of chromatography in use and candidates are required to be familiar with four of them. All rely on a static part (the stationary phase) and a moving part (the mobile phase). Candidates will not be asked questions on the theory of chromatography. Questions will concentrate on its application and the results of its use.

1. Thin layer chromatography (TLC)

Candidates will be familiar with paper chromatography and should view TLC as natural extension. Questions will be limited to the calculation and use of R_f values as an aid in the identification of the components of a mixture. This may include subsequent use of another solvent and/or the turning of chromatograms through 90° .

2. Gas-liquid chromatography (GLC)

This can be used to separate the components of gas mixtures or for those liquids that have an appreciable vapour pressure at the temperatures used. Candidates should be aware that peak areas can be used to find the percentage composition of mixtures and to be able to calculate these given appropriate information. Questions may be set that require a basic understanding and use of retention times.

3. Column chromatography (CC)

This is similar to GLC but uses a liquid mobile phase rather than a carrier gas as used in GLC. A typical column may have alumina or silica gel as the stationary phase contained in a glass tube. If the components of the mixture have different colours then visual

identification and separation is easy. However, if the separated components are colourless, they can be identified under ultraviolet light if they fluoresce.

4. High performance liquid chromatography (HPLC)

This is a variation of CC that uses a smaller particle size for the stationary phase and a high pressure to force the liquid through the column. This type of chromatography has many applications. For example, it can be used to find the caffeine and theobromine content of chocolate and in the detection and identification of compounds present in polluted water.

Infrared absorption spectroscopy

This technique can be used to detect various groups present in (organic) compounds. Covalent bonds between atoms absorb infrared radiation – the frequency absorbed depends on the bond itself and which other groups are present in the molecule. No knowledge of particular frequencies will be required but candidates will be expected to know that this technique can be used to identify certain bonds present in a molecule. The theory of infrared absorption spectroscopy is not required. Questions may be set that ask candidates to identify certain bonds present in simple molecules, given the infrared spectrum and a suitable data sheet showing bonds and their frequencies. A sample data sheet is shown below.

infrared spectroscopy characteristic absorption values	
bond	frequency / cm^{-1}
C – O	1000 – 1300
C = C	1620 – 1670
C = O	1650 – 1750
O – H	2500 – 3550
N – H	3300 – 3500

Mass spectrometry

Candidate will need to be aware of the terms *molecular ion* and *relative molecular mass*. They should be able to relate these two terms in a given mass spectrum and know that compounds give other peaks in their mass spectrum due to fragmentation. No knowledge of the fragmentation process is required and it is not expected that candidates should be able to recognise any structures from fragmentation values. Mass spectrometry can also be used to identify the isotopes present in a sample of an element and the percentage of those isotopes present. Any calculations based on this will be limited to a maximum of three isotopes in a sample of the element.