Unit 2: Practical Scientific Procedures and Techniques

Level: 3
Unit type: Internal
Guided learning hours: 90

Unit in brief

Learners will be introduced to quantitative laboratory techniques, calibration, chromatography, calorimetry and laboratory safety, which are relevant to the chemical and life science industries.

Unit introduction

This unit introduces you to standard laboratory equipment and techniques, including titration, colorimetry, calorimetry, chromatography, calibration procedures and laboratory safety. Through the practical tasks in the unit, you will develop proficiency in the quantitative analytical techniques of titration and colorimetry, including learning to calculate the concentration of solutions. You will use measurement of temperature to study cooling curves and be introduced to paper and thin-layer chromatography (TLC). You will also have the opportunity to calibrate equipment and will be encouraged to be aware of the safety aspects of given laboratory procedures and techniques.

While you develop your practical competence, the discussion and analysis of group results will allow you to understand your progress in relation to that of others and also to gain an understanding of the reliability, repeatability and reproducibility of various procedures and techniques. You will have the opportunity to use problem-solving skills when you undertake calorimetry work. There is scope throughout the unit to reflect on the skills you have gained and how you may develop further. The fundamental knowledge, practical skills, transferable skills – for example, organisation, self-assessment and problem-solving, and the ability to interpret data – all developed in this unit will give you confidence when you undertake the more complex practical techniques involved in higher education science courses such as biochemistry, chemistry, forensic science and environmental science.

The experience you gain will be invaluable when you begin your career as a trainee laboratory technician in industries such as contract analysis, oil, biopharmaceuticals, water treatment, and polymers. Employers in these industries will appreciate your ability to follow written scientific procedures and your desire to ensure accuracy by using techniques correctly and by checking that equipment – for example, pipettes, balances, pH meters and thermometers – is calibrated correctly and that appropriate standard calibration documentation has been completed.

Learning aims

In this unit you will:

A Undertake titration and colorimetry to determine the concentration of solutions
B Undertake calorimetry to study cooling curves
C Undertake chromatographic techniques to identify components in mixtures
D Review personal development of scientific skills for laboratory work.
### Summary of unit

<table>
<thead>
<tr>
<th>Learning aim</th>
<th>Key content areas</th>
<th>Recommended assessment approach</th>
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</thead>
<tbody>
<tr>
<td><strong>A</strong> Undertake titration and colorimetry to determine the concentration of solutions</td>
<td><strong>A1</strong> Laboratory equipment and its calibration</td>
<td>Pro formas of results for checking the calibration of a pipette and balance(s) and calibration of a pH meter.</td>
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<td></td>
<td><strong>A2</strong> Preparation and standardisation of solutions using titration</td>
<td>A report on the use of Na&lt;sub&gt;2&lt;/sub&gt;CO&lt;sub&gt;3&lt;/sub&gt; to standardise HCl, used in turn to standardise NaOH. pH curve from the titration plus a differential plot.</td>
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<td><strong>A3</strong> Colorimetry</td>
<td>Results, calculations and calibration graph for the determination of the concentration of a coloured solution using colorimetry.</td>
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<td>Explanations of how the accuracy, precision and safety of the quantitative techniques may be optimised.</td>
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<td>Observation checklist, completed by the teacher, including safety.</td>
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<tr>
<td><strong>B</strong> Undertake calorimetry to study cooling curves</td>
<td><strong>B1</strong> Thermometers</td>
<td>Results from checking the calibration of at least two types of thermometer.</td>
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<td><strong>B2</strong> Cooling curves</td>
<td>A table of time/temperature data and a graph of temperature against time for a substance cooling.</td>
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<td>Calculations of the rate of cooling at points on the graph.</td>
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<td>An analysis of how the rate of cooling is related to intermolecular forces and the state of the substance.</td>
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<td>A report evaluating the accuracy of the cooling curve experiment.</td>
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<td>An observation report with a checklist, completed by the teacher, including safety.</td>
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<tr>
<td><strong>C</strong> Undertake chromatographic techniques to identify components in mixtures</td>
<td><strong>C1</strong> Chromatographic techniques</td>
<td>Results from the paper chromatography and TLC of extracted plant pigments from paper chromatography of amino acids.</td>
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<td><strong>C2</strong> Application of chromatography</td>
<td>An explanation of the principles behind the chromatographic separations.</td>
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<td><strong>C3</strong> Interpretation of a chromatogram</td>
<td>Suggestions for improvements to the chromatographic procedures carried out and full justification of these suggestions.</td>
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<td>An observation report with a checklist, completed by the teacher, including safety.</td>
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<tr>
<td><strong>D</strong> Review personal development of scientific skills for laboratory work</td>
<td><strong>D1</strong> Personal responsibility</td>
<td>A presentation or report that focuses on the evaluation of learners’ performance and skill development across all scientific procedures and techniques carried out in learning aims A, B and C.</td>
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<td><strong>D2</strong> Interpersonal skills</td>
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<td><strong>D3</strong> Professional practice</td>
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Content

Learning aim A: Undertake titration and colorimetry to determine the concentration of solutions

A1 Laboratory equipment and its calibration

Equipment and glassware used in titration and colorimetry and the importance and processes involved in calibration of measuring equipment.

- Use of pH meters and probes:
  - calibration according to the manufacturer’s instructions.
- Use of balances and weighing:
  - electronic balances – rough balances (two decimal places), analytical balances (four decimal places)
  - checking calibration with certified weights
  - measurement of mass using increasingly accurate balances
  - suitable containers for weighing liquids and solids
  - density of water at different temperatures.
- Safe use of volumetric glassware:
  - bulb, graduated, automated and teat pipettes
  - burettes
  - glass and plastic filter funnels
  - volumetric flasks
  - accurate dilution
  - use of water as a standard for calibrating volumetric glassware.

A2 Preparation and standardisation of solutions using titration

Processes involved in the preparation and standardisation of solutions using titration.

- Accurate determination of the end-point of titrations from:
  - the colour change of a suitable indicator
  - plots of pH versus volume
  - $\Delta$\(\text{pH/\text{volume}}\) versus volume.
- Calculation of concentrations:
  - use of molecular mass from periodic table.
- Use of primary and secondary titrimetric standards.

A3 Colorimetry

Understanding and practical application of colorimetry techniques.

- Selection and use of a colorimeter or visible spectrometer – selection of filter (colorimeter) or fixed wavelength (spectrometer).
- Measurement and use of absorbance readings.
- Use of Beer-Lambert law to determine the concentration of a transition metal ion solution.
- Accurate dilution of stock solutions to prepare a range of calibration standards with absorbance in the range 0 to 1.
- Use of blank solutions.
- Calibration plot.
- Determination of unknown solution concentration from reading from graph (graph paper) or from the equation of a linear trend line through the origin (Microsoft Excel).
Learning aim B: Undertake calorimetry to study cooling curves

B1 Thermometers
Types of thermometer, appropriate use and practical application of measurements of heat.
- The relationship between temperature and heat energy.
- Types of thermometer and how they are used to gain accurate readings:
  - electronic thermometers/temperature probes
  - liquid-filled thermometers.
- Checking the calibration of thermometers by using ice and boiling water.
- Accuracy of thermometers and temperature probes at different temperatures.

B2 Cooling curves
Construction and interpretation of cooling curves:
- temperature as a function of time
- rate of cooling from the gradient of the tangent to the cooling curve
- determination of melting point from the shape of a curve for a substance freezing
- super cooling
- shape of the curve and rate of cooling in relation to intermolecular forces and the state (solid or liquid) of the substance.

Learning aim C: Undertake chromatographic techniques to identify components in mixtures

C1 Chromatographic techniques
Theory, equipment and procedures used in chromatography.
- Terminology:
  - mobile and stationary phases
  - adsorption.
- Principles of paper chromatography.
- Principles of thin-layer chromatography (TLC):
  - nature of a TLC plate – glass, metal or plastic sheet with solid adsorbent layer.
- Use of capillary tubes to apply mixtures to paper or TLC plates.
- Choice of developing solvent and vessel.
- Preparative methods for samples:
  - solvent extraction
  - filtration
  - concentration by evaporation.
- The use of locating agents.

C2 Application of chromatography
- Separation of components of a mixture, to include plant pigments extracted from leaves/herbs with propanone (paper chromatography and TLC).
- Identification of unknown mixtures and pure substances using chromatography, to include amino acids (paper chromatography).
- Awareness of other types of chromatography – e.g. gas chromatography, ion-exchange chromatography – and that procedures and chromatogram interpretations are very different.
C3 Interpretation of a chromatogram

- Polarity of molecules/intermolecular forces in relation to solubility in the mobile phase.
- Polarity of molecules/intermolecular forces in relation to retention of molecules in the stationary phase.
- Size of molecules in relation to solubility and mobility.
- Calculation of Rf value.
- Interpretation of chromatograms in terms of the number of substances present and the Rf values of components.
- Awareness of common problems in technique resulting in difficulty interpreting a chromatogram, e.g. overloading samples, disturbing plate/paper during development or contamination of plate/paper.

Learning aim D: Review personal development of scientific skills for laboratory work

D1 Personal responsibility
Understanding of the personal responsibilities that must be accepted for successful work in science.
- Work to appropriate standards and protocols.
- Application of safe working practices.
- Accept responsibility for the quality of own work.
- Take responsibility for completing tasks and procedures as well as using judgements within defined parameters.

D2 Interpersonal skills
Understanding and development of skills for effective and efficient working with others:
- Communication and co-operation in the scientific working environment
- Give and receive constructive feedback
- Behaviour for safe and efficient working in science.

D3 Professional practice
Understanding and personal development of standard practices applicable to working as a professional scientist:
- Recognise problems and apply appropriate scientific methods to identify causes and achieve solutions
- Identify, organise and use resources effectively to complete tasks
- Maintain and enhance competence.
# Assessment criteria

<table>
<thead>
<tr>
<th>Pass</th>
<th>Merit</th>
<th>Distinction</th>
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<tbody>
<tr>
<td><strong>Learning aim A: Undertake titration and colorimetry to determine the concentration of solutions</strong></td>
<td></td>
<td>A.D1 Evaluate the accuracy of procedures and techniques used in titration and colorimetry in relation to outcomes and suggest improvements.</td>
</tr>
<tr>
<td>A.P1 Correctly prepare and standardise solutions for titration and colorimetry.</td>
<td>A.M1 Demonstrate skilful application of procedures and techniques in titration and colorimetry to accurately determine the concentration of solutions.</td>
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<tr>
<td>A.P2 Investigate the concentration of unknown solutions, using procedures and techniques in titration and colorimetry.</td>
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<tr>
<td><strong>Learning aim B: Undertake calorimetry to study cooling curves</strong></td>
<td></td>
<td>B.D2 Evaluate the accuracy of practical work in calorimetry in relation to the analysis of the cooling curve.</td>
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<tr>
<td>B.P3 Correctly obtain data using different equipment to construct cooling curves.</td>
<td>B.M2 Analyse the rate of cooling of substances from your data using cooling curves to draw valid conclusions.</td>
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<tr>
<td>B.P4 Correctly determine the rate of cooling of substances using cooling curves.</td>
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<tr>
<td><strong>Learning aim C: Undertake chromatographic techniques to identify components in mixtures</strong></td>
<td></td>
<td>C.D3 Evaluate the chromatographic techniques used in relation to outcomes and suggest improvements.</td>
</tr>
<tr>
<td>C.P5 Correctly use chromatographic techniques to produce chromatograms.</td>
<td>C.M3 Analyse own chromatograms and relate the factors that affect the separation of mixtures to the quality of results obtained.</td>
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<tr>
<td>C.P6 Explain the use of chromatographic techniques to separate mixtures.</td>
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<tr>
<td><strong>Learning aim D: Review personal development of scientific skills for laboratory work</strong></td>
<td></td>
<td>D.D4 Evaluate scientific skills developed in terms of potential for future progression.</td>
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<tr>
<td>D.P7 Summarise key personal competencies developed in relation to scientific skills undertaken.</td>
<td>D.M4 Analyse skills developed and suggest improvements to own practice.</td>
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**Essential information for assignments**

The recommended structure of assessment is shown in the unit summary along with suitable forms of evidence. *Section 6* gives information on setting assignments and there is further information on our website.

There is a maximum number of four summative assignments for this unit. The relationship of the learning aims and criteria is:

- Learning aim: A (A.P1, A.P2, A.M1, A.D1)
- Learning aim: B (B.P3, B.P4, B.M2, B.D2)
- Learning aim: C (C.P5, C.P6, C.M3, C.D3)
- Learning aim: D (D.P7, D.M4, D.D4)
Further information for teachers and assessors

Resource requirements

For this unit, learners must have access to:

- a well-equipped laboratory with a fume cupboard
- accurate balances
- a range of volumetric glassware
- pH meters, thermometers and temperature probes (access to data-logging software is useful but not essential)
- colorimeter or visible spectrometer
- chromatography paper, TLC slides
- a range of suitable chemicals, dependent on specific practical work that centres choose to utilise.

Essential information for assessment decisions

Learning aim A

For distinction standard, learners will interpret outcomes of their quantitative analytical procedures and techniques to make sound judgements on the accuracy of them. They will place the accuracy of their results in the context of those obtained by other learners in a meaningful and quantitative way. Learners will be able to coherently discuss problems/issues with the quantitative procedures and techniques used and develop a strong rationale for suggestions made to improve accuracy and precision in order to obtain reliable and valid outcomes (or for justifying the appropriate steps already taken should no problems be identified).

Learners will provide sound discussion of inherent hazards and risks associated with the analytical techniques and procedures, for example justifying why certain aspects are carried out in a particular way on safety grounds.

For merit standard, learners will undertake quantitative analytical procedures and techniques with minimal supervision, and perform to a high degree of accuracy and precision in order to obtain reliable and valid outcomes, with consideration for health and safety. Learners will demonstrate skill and fluency in a number of areas, such as: calibrating pipettes transferring solids, measuring volumes, mixing solutions, carrying out titrations and making the dilutions for colorimetry standards. They will be fully prepared in terms of equipment, reference material and consumables before attempting each step.

For pass standard, learners will follow instructions to safely undertake titration and colorimetry, although they may need to refer frequently to the instructions. These must be performed correctly to obtain reliable and valid outcomes. Learners will correctly carry out calculations of concentration.

For titration, learners will check the calibration of equipment used to ensure the validity of outcomes obtained (for example the calibration of a pipette, balances and a pH meter using buffer solutions). It is expected that learners will be assessed making a solution by weighing a solid, making the solution to volume and shaking to ensure that it mixes thoroughly. They could use a primary standard acid/base in a titration to standardise sodium hydroxide/hydrochloric acid prepared by the learner. Learners must also safely and correctly calibrate and use a colorimeter or visible spectrometer to determine the concentration of a coloured solution.
Learning aim B

For distinction standard, learners will interpret outcomes of their calorimetry to make sound judgements on accuracy. Learners will be able to use appropriate mathematical terminology (for example rapid increase, decrease, approximately constant, etc.) to describe the patterns and trends in the shapes of cooling curves. They will be able to use the cooling curve of a substance to evaluate how close their values for the melting points are to literature and to class values, explaining where specific errors or problems with the given method or equipment may have led to inaccuracy. Learners could, for example, discuss the way in which the substance was cooled and the resulting changes to the curve. Learners will explain why it may be necessary to make changes to procedures in order to reduce levels of uncertainty.

For merit standard, learners will demonstrate selection of an appropriate amount of solid; selection of a suitable vessel for heating the solid, setting up the equipment to enable heating and cooling of the vessel in an appropriate way and monitoring temperature as a function of time in a safe way.

Learners will demonstrate numerical skills in graph plotting when constructing their cooling curve. These must include selecting the most appropriate scale, using appropriate labels including units, and drawing a smooth, best-fit curve through the points. By drawing tangents at appropriate points and finding their gradients, learners will correctly determine the rate of cooling near the start, end and where the rate appears to have changed dramatically in between. They will draw valid conclusions linking the rate of cooling to what is happening at a molecular level in terms of the positions and velocity of molecules and the forces between them. They will be able to explain which part of the graph corresponds to, for example, the melting point (freezing temperature).

For pass standard, learners will safely check the calibration of a given thermometer, following instructions. This could be done by using ice and boiling water. Learners will also explore the accuracy of the temperature measurements obtained from thermometers and other equipment by comparing their readings in water that is being heated. Learners will use a table of their own design for recording their readings. Learners will demonstrate key practical competencies in calorimetry, including being able to set up a vessel containing a solid, heating it to above its melting point, cooling it and measuring its temperature as a function of time, following a standard procedure.

Learners will plot graphs for a substance undergoing freezing. Learners might not select the most appropriate scale but will label axes correctly and draw a smooth curve through the points. They will accurately determine the rate of cooling near the start, demonstrating the ability to draw a tangent to the curve and find its gradient.

Learning aim C

For distinction standard, learners will articulate strong links between outcomes and techniques used in order to give a rationale for specific improvements that could be made to the chromatographic techniques. They will articulate what would happen if a particular change were to be made. They will demonstrate awareness that some chromatograms may show the spots rising at an angle or have spots that are too big or smeared out rather than being distinct.

For merit standard, learners will demonstrate safe working practices and a high level of proficiency when carrying out paper- and thin-layer chromatography (TLC) with minimal supervision. They will produce chromatograms showing clear separation of spots, repeating the separations if they are not satisfied with the quality of the separation obtained. Learners will also comment on the suitability of the techniques for separation.

Learners will use appropriately calculated Rf values and consider factors that influence separation to justify conclusions drawn about the identification of components in a mixture (for example the polarity of the components of the mixtures and the polarity of the solvents and effect of the size of a molecule on its mobility).
For pass standard, learners will follow instructions, demonstrating safe working practices and a good level of ability when carrying out paper and TLC. Learners will comment on the suitability of the techniques for separation and the chromatogram produced for each technique (TLC and paper chromatography). At this standard, the chromatograms may not produce spots showing an optimum degree of separation (for example the spots may be too large and lacking in distinction). They will determine Rf values using paper chromatograms, using these to correctly identify components in a mixture.

Learning aim D

For distinction standard, learners will draw upon all areas of practical work carried out to critically reflect on strengths and weaknesses of their own performance and skill development drawing on feedback, for example from peers, teachers and industry. Drawing on others’ feedback is crucial for developing balanced progression goals.

For merit standard, learners will need to make judgements on their skill development and level in relation to their peer group. They will need to recognise the improvements that need to be made and how they will take steps to achieve them.

For pass standard, learners will identify areas of scientific skills developed in relation to the learning aims. They should to draw on scientific skills developed across other units to illustrate the transferability of skills.

Links to other units

This unit links to:
- Unit 1: Principles and Applications of Science I
- Unit 3: Science Investigation Skills
- Unit 4: Laboratory Techniques and their Application
- Unit 19: Practical Chemical Analysis.

This unit also links to a wide range of optional units available across the qualification.

Employer involvement

Centres may involve employers in the delivery of this unit if there are local opportunities. It would be beneficial for an industry representative to explain the importance of the routine calibration of equipment in ensuring the reliability of results. A visit to a local laboratory would reinforce the importance of calibration of equipment and health and safety. Even if the local organisations that use science only operate on a small scale, their representatives will be able to reinforce the importance of the transferable skills this unit develops.