Unit 18: Practical Chemical Analysis

Unit code: K/502/5560

QCF Level 3: BTEC National

Credit value: 10

Guided learning hours: 60

Aim and purpose

The aim of this unit is to enable learners to understand and apply the principles behind a range of analytical techniques used in industrial and commercial laboratories, particularly titration, spectroscopy and chromatography and includes instrumental techniques. The way that analytical data is presented in industry is also covered.

Unit introduction

On average, 1 litre of seawater contains almost 11,000 mg of sodium, river water contains 6 mg and bottled waters can contain anything from 6 to 150 mg. Lead occurs naturally in soil at a level of approximately 10 ppm (parts per million), however uncontrolled industrial pollution can increase the lead content in soil to dangerous levels of more than 1,000 ppm. Such values are determined using analytical chemistry techniques and represent just a small area where aspects of analytical chemistry contribute to our lives. A wide range of chemical substances can be detected and analysed to measure environmental pollution. Even caffeine can be measured as a chemical marker for surface pollution by domestic wastewaters.

Chemical analysis also has many applications in manufacturing, particularly in product quality control, monitoring of production processes and drug development processes in the pharmaceutical industry. It is also a key component in healthcare (in the diagnosis of disease), forensic science (analysing substances found at crime scenes), and public health (testing drugs, food, air quality, water quality and monitoring industrial waste).

The analytical process encompasses a range of skills including sampling techniques, separation and isolation of components, estimating error limits, data manipulation and interpretation and communication of results. Increasingly, analytical procedures utilise complex electronic equipment and computer-aided interpretation of results.

In this unit spectroscopic and chromatographic methods are investigated, together with aspects of volumetric analysis necessary to produce accurate sample and reference solutions. The analytical process is contextualised by exploring the procedural features needed to assure quality of the data from an environmental science or other commercial or industrial analytical laboratory.

This unit gives learners a taste of what it is like to work in a science laboratory. It is suitable for all learners who are interested in a career in science.

Learning outcomes

On completion of this unit a learner should:

1 Be able to use standard solutions in quantitative analysis

2 Be able to analyse data from spectroscopic techniques to provide analytical information about chemical substances

3 Be able to use chromatographic techniques to analyse mixtures of chemical substances

4 Know how an industrial or commercial laboratory operates.

Unit content

1 Be able to use standard solutions in quantitative analysis

Concentration: calculations involving number of moles, volume and concentration; calculation of concentration from titrimetric analysis; calculations involved in producing a range of standard solutions from a given stock solution of known concentration

Standard solutions: properties of a primary standard; secondary standards; purity of substances used in preparation of standard solutions; preparation of solutions of fixed concentration; appropriate titrations to determine concentration or standardise given solutions; dilution of stock solutions to give a series of related standard solutions; errors associated with the process

2 Be able to analyse data from spectroscopic techniques to provide analytical information about chemical substances

Outputs: spectra; absorbance readings; emission readings

Spectroscopic techniques: ultraviolet/visible; infrared; atomic absorption and/or atomic emission; ^{1}H NMR; mass spectroscopy; method validation

Analytical information: ultraviolet/visible spectroscopy, Beer-Lambert law, measurement of absorbance, construction of calibration curves, addition of reagents to intensify/develop colour, measurement of concentration, eg nitrate, copper, nickel in water, aspirin in ethanol; infrared spectroscopy, absorption bands and correlation charts, identification of organic functional groups, origin and uses of the fingerprint region; applications of absorption and emission spectroscopy for determination of concentration of metal ions in solution; calibration curves; criteria for method selection; use in quantitative analysis, eg iron content of water samples, potassium and sodium in water; ^{1}H NMR spectroscopy, conditions for NMR activity; TMS as internal standard; correlation charts, integration traces; spin-spin splitting; assignment of peaks in ^{1}H NMR spectra to equivalent ^{1}H in simple molecules; mass spectrometry; measurement of relative molecular mass; molecular ion peak; simple fragmentation patterns; use of % elemental composition, derived empirical formula, infrared spectroscopy and ^{1}H NMR jointly to identify an organic compound

3 Be able to use chromatographic techniques to analyse mixtures of chemical substances

Chromatographic techniques: wet techniques, eg thin layer chromatography (TLC), paper chromatography, adsorption column chromatography, ion-exchange chromatography; instrumental techniques, eg gas chromatography (GC), high performance liquid chromatography (HPLC), ion-exchange chromatography, molecular exclusion chromatography; optimisation of separation

Analyse: basic instrumentation (where appropriate); variables, eg variation of mobile and stationary phases, oven temperature; qualitative analysis; Rf values; retention times; visualisation/detection of fractions; quantitative analysis, eg area under peaks, titration of column eluent.

Chemical substances: simple mixtures, eg extract of leaf pigments, glucose-maltose mixture, seven food dye mixture dissolved in water, alkanes by GC, caffeine and aspirin by HPLC; quantitative analysis, eg Cu^{2+} by ion-exchange, caffeine by HPLC

4 Know how an industrial or commercial laboratory operates

Laboratory type: any multifunctional laboratory, eg environmental analytical laboratory, other laboratories supplying regulatory information about safety and suitability of products, industrial quality control laboratories, forensic science, hospital clinical chemistry

Processes: range of analytical procedures; data recording and manipulation; data presentation; quality assurance; security; accreditation requirements for testing laboratories; documentation of standard operating procedures; good laboratory practice (for studies undertaken to generate data by which the hazards and risks to users, consumers and third parties, including the environment, can be assessed for pharmaceuticals, agrochemicals, veterinary medicines, industrial chemicals, cosmetics, food and feed additives and biocides)

Importance of health and safety: impact of appropriate legislation; risk assessment; safety audits

Assessment and grading criteria

In order to pass this unit, the evidence that the learner presents for assessment needs to demonstrate that they can meet all the learning outcomes for the unit. The assessment criteria for a pass grade describe the level of achievement required to pass this unit.

Assessment and grading criteria					
To achieve a pass grade the evidence must show that the learner is able to:		**To achieve a merit grade the evidence must show that, in addition to the pass criteria, the learner is able to:**		**To achieve a distinction grade the evidence must show that, in addition to the pass and merit criteria, the learner is able to:**	
P1	carry out practicals to prepare a range of solutions of known concentration for use in analytical techniques [EP3, 4, 5]	**M1**	explain the need for primary and secondary standards in analysis	**D1**	evaluate the reliability of the techniques used to guarantee the accuracy of concentrations of solutions of known concentration
P2	carry out the identification of unknown compounds by matching key features of a range of spectra with those of standard substances [RL2, 5]	**M2**	determine the structure of a simple organic compound from its % elemental composition, infrared spectrum, mass spectrum and ^{1}H NMR spectrum	**D2**	explain the process of determining the structure of a simple organic compound from its % elemental composition, infrared spectrum, mass spectrum and ^{1}H NMR spectrum
P3	carry out practicals to determine concentrations from spectroscopic techniques [EP3, 4, 5]	**M3**	describe the stages in developing a method for finding the concentration of a solution using a spectroscopic technique		
P4	carry out practicals to separate mixtures with chromatographic techniques [EP3, 4, 5]	**M4**	explain the principles involved in a chromatographic separation	**D3**	explain how the separation from a chromatographic technique may be optimised
P5	use data from instrumental techniques to determine the concentration of mixtures using chromatography [EP3, 4, 5]	**M5**	explain the stages in a method for finding the concentration of a solution by a chromatographic technique		
P6	identify the range of procedures used in carrying out analysis in an industrial or commercial analytical laboratory. [IE1, 2]	**M6**	describe how raw data from an industrial or commercial analytical procedure is transformed into the format required for the customer of the analysis.	**D4**	explain how the data from an industrial or commercial analysis is quality assured.

PLTS: This summary references where applicable, in the square brackets, the elements of the personal, learning and thinking skills applicable in the pass criteria. It identifies opportunities for learners to demonstrate effective application of the referenced elements of the skills.

Key	IE – independent enquirers	RL – reflective learners	SM – self-managers
	CT – creative thinkers	TW – team workers	EP – effective participators

Essential guidance for tutors

Delivery

This unit is delivered through a series of practical exercises. These should be set in the context of environmental analysis or other realistic vocational scenarios where possible. Learners need the opportunity to make and use primary and secondary standards. This will involve understanding the properties of standard substances and reference materials. Learners need to calculate concentrations of solutions and the quantities involved in making them and diluting them. They need to carry out appropriate titrimetric standardisation. The sources of error inherent in volumetric techniques should be discussed. This is not limited to the tolerance of the glassware. The need to ensure that materials are weighed accurately, eg by difference on an analytical balance, should be discussed, as should proper dissolution of substances before making to the mark and accurately making solutions to the mark. Thorough mixing is important.

The use of various regions of the electromagnetic spectrum in analysis should be considered, with learners doing appropriate practical work. It is expected that learners will have access to infrared and ultraviolet/visible spectrometers and atomic spectroscopic techniques such as flame emission, atomic absorption or inductively coupled plasma-optical emission spectroscopy (ICP-OES) spectrometers. This could be at the delivery centre or by arrangement with a local commercial laboratory. Use of mass and proton NMR spectra will also be considered. Where possible learners should visit higher educational institutions or commercial laboratories to observe these instruments in action.

Learners need to study and interpret spectra from infrared, ultraviolet, mass and proton NMR spectrometers. Learners should assign peaks in infrared spectra to the presence of functional groups, in particular O-H, C-H and C=O. Learners will study ultraviolet/visible spectra, identify wavelengths of maximum absorbance and relate this to the colourlessness/colour of the compounds. Learners should use % composition to determine empirical formula and determine molecular formula by consideration of mass spectra. Infrared, mass and proton NMR spectra should be used to elucidate structures of simple organic compounds. Tutors can teach how to develop and use methods for finding the concentration of analytes by ultraviolet/visible spectroscopy, using fixed wavelength and the Beer-Lambert law. Ideally learners should use visible spectrometers. However, they may be presented with spectra and develop methods for colorimeters if necessary, providing the peaks in the spectra are related to the filter giving maximum absorbance. Use of reagents, added to intensify colour (eg ammonia solution for determination of Cu^{2+} or molybdate determination of phosphate) should be covered. This is particularly important for centres that do not have a range of different types of spectrometer. Quantitative infrared spectroscopy may be used if available. Where possible, learners should collect and use quantitative data from atomic spectroscopic techniques, such as flame emission, atomic absorption and ICP, to determine the concentration of solutions. These applications use standard solutions at very low concentration. Learners should have the opportunity to prepare the solutions and use the instruments. Where use of the instruments is impossible learners should be shown them, given appropriate analytical data from the instruments and given the opportunity to find out about the principles of their operation. Learners should be familiarised with industrial uses of spectroscopic techniques.

Use of chromatographic techniques, particularly HPLC, in analysis is continuing to grow. Learners may have the opportunity to separate mixtures by paper chromatography, thin layer chromatography, column chromatography, such as ion exchange or packed alumina, gas chromatography and HPLC. Where the centre does not possess a gas chromatograph or HPLC chromatograph, learners could be taken to an appropriate laboratory to see these instruments in operation.

The concepts of mobile and stationary phases and sorption mechanisms (adsorption, partition, ion-exchange, exclusion) could be introduced for paper or thin layer techniques. Learners should be given the opportunity to investigate the effect of a variation of the composition of the mobile phase for a suitable technique, like paper or thin layer chromatography of plant pigments or food colours. Where possible, learners should be given the opportunity to make and pack columns of ion-exchange resin or alumina in order to understand the nature of the columns in GC and HPLC applications. Learners should have the opportunity to separate mixtures by paper, thin layer and gas chromatography. Additionally, mixtures may be separated by HPLC if convenient. Learners could see HPLC chromatograms showing separation of mixtures instead. Learners should be taught the nature of and the scope for variation of the composition of the stationary phase in GC and HPLC. The effects of variation of temperature in GC and mobile phase composition in HPLC should be studied. Although learners may not have direct access to GC or HPLC instrumentation, the techniques should still be taught and specimen chromatographs analysed.

GC and HPLC are used routinely in industry as quantitative techniques. Learners should use these techniques quantitatively or should be presented with chromatographs which will allow them to calculate the amounts of substances present in mixtures.

Throughout the practical work factors affecting accuracy and reliability of methods should be discussed. Learners should be acquainted with the range of standard procedures used in analytical laboratories to ensure the quality of the data. Because of the importance of health, safety and the environment in all laboratory contexts, the need to have appropriate risk assessment procedures and procedures to minimise environmental impact should also be covered here. Learners should be made aware that some laboratories work to Good Laboratory Practice (GLP) regulations. The data from these laboratories, eg public analysis laboratories and pharmaceutical laboratories, must be correct in order to ensure the safety of the public. Other laboratories, such as contract laboratories testing environmental samples or oil samples for other companies may be accredited by the United Kingdom Accreditation Service (UKAS). Companies using accredited laboratories must be sure that the results are correct. Even if data does not need to be legally defensible, it makes commercial sense for it to be correct. Laboratories not working to GLP or UKAS standards are likely to adopt similar techniques to ensure that data is as accurate as possible. Learners should understand the range of standard operating procedures adopted to quality assure data, eg use of appropriate analytical standard substances, calibration procedures, sampling procedures, recording and reporting procedures, statistical quality control procedures, and method and equipment validation procedures. Good links with and support from local laboratories are vital to learners' understanding of the importance of these procedures. One way to approach delivery is to ask, for example, 'if the balance is not calibrated what might happen?'; 'if a standard test method is not followed by everyone what might happen?'; 'if everyone records data in a different way what might happen?' and so on.

Outline learning plan

The outline learning plan has been included in this unit as guidance and can be used in conjunction with the programme of suggested assignments.

The outline learning plan demonstrates one way in planning the delivery and assessment of this unit.

Topic and suggested assignments/activities and/assessment
Introduction to unit and programme of assignments.
Calculations involving number of moles, concentration, volume.
Assignment 1 – Preparation of Standard Solutions (P1, (part) M1, D1) Standardisation of solutions using primary and secondary standards, eg use of potassium hydrogen phthalate to standardise sodium hydroxide solution followed by use of standardised sodium hydroxide solution to standardise an ethanoic acid solution.
Internet research by learners on the electromagnetic spectrum and the uses of different regions of the spectrum in spectroscopy.
Determination of empirical formula from % elemental composition.
Mass spectroscopy – instrumentation, mass/charge ratio, relative abundance, molecular ion peak, fragmentation patterns, identification of an unknown.
Infrared spectroscopy – running/studying spectra of simple functional group compounds – instrumentation, sample holders, identification of unknowns. Ultraviolet visible spectroscopy – running/studying spectra of simple organic and transition metal compounds – instrumentation, sample cells, identification of unknowns.
^{1}H NMR – instrumentation, TMS standards, chemical shift, correlation charts, integration, splitting pattern – assigning peaks to equivalent ^{1}H.
Assignment 2 – Identification of Unknown Compounds (P2, M2, part D2) Identification of unknown compounds by matching features of spectra. Simple structure elucidation by use of % elemental composition, mass spectroscopy, infrared spectroscopy and ^{1}H NMR. Presentation of the reasoning process.
Introduce the Beer-Lambert law and the instrumentation and cells used in ultraviolet/visible spectroscopy. Studying the appearance and meaning of examples of spectra.
Assignment 3 – Determination of Concentration by Spectroscopy (P1, (part) P3, M3, part D2) Covered in the next nine rows.
Preparation and use of standard solutions for determination of the concentration of a copper sulphate solution by visible spectroscopy, including scanning the spectrum and identification of a suitable fixed wavelength (or selection of a suitable filter for a colorimeter).
Preparation and use of standard solutions for determination of the concentration of a copper sulphate solution by visible spectroscopy, including scanning the spectrum and identification of a suitable fixed wavelength (or selection of a suitable filter for a colorimeter) – for a different ion. Could include addition of a colour reagent.
Preparation and use of standard solutions for determination of the concentration of nitrate in filtered river water by ultraviolet spectroscopy.
Analysis of the sources of error in ultraviolet/visible spectroscopy. Write about experiment.
Introduction to atomic absorption spectroscopy (or ICP-OES).
Preparation and use of standard solutions for the determination of the concentration of iron in a water sample by atomic absorption (or calculations and interpretation of data from such an experiment).
Analysis of sources of error in determination of iron in a water sample by atomic absorption. Write about the experiment.
Introduction to atomic emission spectroscopy.

Topic and suggested assignments/activities and/assessment
Preparation and use of standard solutions for the determination of the sodium content of bottled water by flame emission spectroscopy (or calculations and interpretation of data from such an experiment).
Analysis of sources of error in determination of finding sodium content of bottled water. Write about the experiment.
Introduction to chromatography – mobile/stationary phase.
Assignment 4 – Separation of Mixtures by Chromatography (P4, M4, part D3) Collation of practical work covered in the next seven rows.
Paper chromatography separation of plant pigments.
Paper chromatography of amino acids.
Separation of a mixture of sugars using paper chromatography and comparison to standards – calculation of R_f values; presentation of results.
Extraction and TLC of pigments extracted from leaves – calculation of R_f values; presentation of results – discussion of optimisation of the separation.
TLC halides – visualise spots with fluorescein.
Column chromatography – preparation of an alumina column in nitric acid – separation of a mixture of transition metal ions – evaluation of the problems – discussion of optimisation of separation.
Determination of the concentration of a copper (II) solution using a cation-exchange column in the H^+ form and titration with standardised sodium hydroxide.
Introduction to GC. Use of textbooks. Separation of mixture of alkanes. Effect of altering oven temperature explored – optimistion of separation discussed.
Introduction to HPLC. Use of textbooks. Effect of altering mobile and stationary phases discussed – optimisation of separation discussed.
Assignment 5 – Quantitative Chromatography (P5, M5) (part D3 here) Determination of the caffeine content of a drink using caffeine standards or interpreting given data for HPLC or GC involving standards. How may HPLC separations be optimised? Research from books. Write about optimisation.
Assignment 6 – Quality Assurance of Data and the Need for Standard Procedures (P6, M6, D4) Choose a straightforward analytical method – eg hypochlorite content of bleach, caffeine content of a drink or ethanoic acid in vinegar. Discuss how such a determination may be required industrially. Discuss procedure, highlighting the need for other standard procedures such as risk assessment, calibration, product sampling, matching sample numbers, reporting etc. Discuss who the customer for the analysis might be and the sort of report they may want. Identify quality procedures involved in analysis. Explain how raw data ends up as a report. Evaluate the quality assurance that would be needed.
Review and evaluation of unit.

Assessment

In general terms, pass learners will be able to carry out analytical procedures competently and accurately and know the type of standard procedures adopted in commercial analytical laboratories. Presentation of results may be done as part of a formal laboratory report or by completion of results sheets integral to the instruction sheets. Since this unit contains a great deal of content, it would be unfair to expect learners to present the results of all practical work in the form of standard laboratory reports.

Learners may have used volumetric technique as part of *Unit 22: Scientific Practical Techniques*. To achieve P1, learners should perform straightforward calculations to determine the amounts of substances necessary to make volumetric solutions of known concentration. Learners should make up concentrations, using good volumetric technique, and carry out accurate titrations to determine the concentration of the solutions accurately. Learners should work out a range of straightforward dilutions of a stock solution in order to prepare a range of solutions of known concentration for a spectroscopic application. This could be the determination of concentration of metal ions in solution by visible absorption, atomic emission or atomic absorption. Alternatively, learners could prepare a range of solutions of known concentration for use in a chromatographic application such as determination of caffeine in drinks by HPLC.

For M1, learners should explain why primary and secondary standard substances are needed in analysis. Learners must understand the difference between primary and secondary standards. They should explain why primary standard substances would not always be used in standardisation and why secondary standard substances are used. For example, sodium thiosulphate is a convenient titrant for many redox titration purposes but its concentration must be checked each time it is used because it is not stable over a period of time. For example, potassium hydrogen phthalate is used as a primary standard acid but, because it is a weak acid, would not be used to standardise a weak alkali, like ammonia solution. A secondary standard, a standardised strong acid would be used instead. (This may be done by explaining the consequence of not having standard substances.)

To achieve D1, learners should examine and evaluate volumetric techniques used to make accurate standard solutions for titration and for spectroscopy or chromatography. Although the tolerance of the glassware should be considered, learners should also identify common errors in volumetric technique and consider how these would be minimised. Learners may consider the linearity of spectroscopic or chromatography calibration plots, in relation to the accuracy of the dilution stages, for example. Learners should consider the importance of the accuracy of the stock solution and the need for pure standards and standardisation.

In the above suggested assessment of P1, M1 and D1, learners could make solutions using standard methods provided. This will involve titration to determine the accurate concentration of standard solutions. Learners should explain why standard solutions are needed and evaluate the key features of the techniques which ensure that the concentrations are accurate. This is unlikely to involve dilution from a stock solution. Learners also have to carry out the dilution involved in a Beer-Lambert law application to achieve P1 fully.

For P2, learners must be presented with infrared spectra and visible/ultraviolet for unidentified compounds. Learners should assign the main peaks in the spectra to the appropriate infrared-active functional groups, eg bond stretches for O-H, C-H, C=O, N-H, C=C etc. Substitution patterns in the fingerprint region may be matched for substituted aromatic compounds but this is not mandatory. Learners should use simple correlation charts in this activity. Once the main peaks have been assigned, the spectra should be compared with spectra of compounds whose identity is known in order to confirm the decisions made. Learners must also carry out matching exercises for ultraviolet/visible spectra but it is not necessary to assign the peaks to particular electronic transitions.

To achieve M2, learners should use data on % composition by mass to determine the empirical formula of a compound and use the molecular ion peak from the mass spectrum to determine the molecular formula. If learners have studied enough organic chemistry to allow them to suggest a range of possible structures and the number of possible isomers is small, they may be asked to suggest the possible structures. Alternatively, having worked out the molecular formula, they may be presented with a limited number of possible structures, say three, to distinguish between on the basis of the spectroscopic evidence. Learners will be expected to choose the correct structure by identifying key features of the given infrared spectrum by using a correlation chart and matching this with the possible structures. Learners need to identify the numbers of equivalent protons in the proposed structures and predict the number of expected peaks in the ^{1}H NMR spectra. They should identify the peaks which correspond to these from the ^{1}H NMR spectrum, with the aid of correlation charts for the $\tilde{\delta}$ values and the integration of the peaks. Splitting patterns may be considered if appropriate. Learners could be asked to use the fragment ion peaks to confirm the selection of the appropriate compound but this is not mandatory.

For D2, learners must explain the process of elucidation of the structure using the spectra in a logical way. If appropriate, learners may be presented with an M2 task and be given a sequence of steps to follow and then, to achieve D2, carry out another elucidation task without step-by-step guidance or simply explain the logic behind each step of the process in full. Learners could provide a commentary/annotation of why they think a particular compound matches the spectra while other isomers do not.

In the above, suggested assessment for P2, M2, D2, learners could be presented with infrared and ultraviolet spectra for substances or solutions of substances and be required to identify key features (such as O-H, C-H and C=O stretches and peaks for substituted aromatic compounds in the fingerprint region in the infrared spectra) and compare them with standard spectra in order to identify the unknown substances. Additionally, learners could be presented with the % composition of C, H and O in a substance, the mass, infrared and proton NMR spectra and asked to elucidate the structure of the compound, following a given method. For D2, the logic used throughout the process would have to be explained in full.

For P3, learners must obtain and use data to calculate the concentration of substances in solution using spectroscopic techniques. Learners should use at least three techniques. These could all be ultraviolet/visible techniques. In that case, it is essential that learners use one technique that involves addition of a colour reagent because that ensures breadth of coverage. Alternatively, learners could use ultraviolet/visible spectroscopy, atomic absorption (or ICP) and atomic emission spectroscopy. Calibration graphs for each technique may be plotted by spreadsheet or on paper.

For M3, learners could determine the wavelength of maximum absorbance for an ultraviolet/visible spectroscopy concentration determination and set the spectrometer on that fixed wavelength. If access to a spectrometer is limited, a colorimeter may be used. However, learners must be presented with an appropriate spectrum for the substance in solution and must be allowed to select the filter to be used and relate this to the spectrum of the substance in solution. Learners should explain how a suitable range of concentrations would be found in the accurate working range of absorbance (ideally up to an absorbance of about 1) of the spectrometer. They should be given the opportunity to test the suggested method but this is not mandatory. Learners could generate additional evidence for achievement of D2 through practical spectroscopy exercises.

For P4, learners should use a range of techniques to carry out qualitative separation of mixtures by chromatography. This should include at least three of the following: paper chromatography, thin layer chromatography, a column technique like packed alumina or ion-exchange, GC and HPLC. The results from the separation should be reported and processed appropriately. This should involve determination of Rf values for paper and thin layer chromatography and retention times for GC or HPLC. Comparisons with standard materials should be included where possible, eg spiking may be carried out for GC and HPLC and chromatograms of reference materials or published Rf values for paper and TLC.

For M4, learners should explain in detail the role of the stationary and mobile phases and the sorption mechanism for one of the applications studied. This may include discussion of what might happen if the conditions of the application, eg composition of mobile phase, nature of the stationary phase, temperature of the oven in GC, were altered. To achieve the related D3, learners should consider how the conditions would be varied until optimum separation was obtained. Learners need to define optimum separation and have a clear understanding of the variables in the application discussed.

For P5, learners should use at least two techniques quantitatively, eg GC and HPLC to measure concentration of mixtures. Learners could generate primary data or could interpret secondary data. For example, learners could be given GC chromatograms for solutions of known composition and be given a chromatogram of a substance, made in the same solvent. They would estimate the composition of that solution of the substance from the area under the peak. Quantitative HPLC is commonly used in a similar way to quantitative spectroscopy in that a series of standards of known composition is made and the integration (area under the peak) is determined. The integration is equivalent to the absorbance of the unknown.

To achieve M5, learners must explain the bases of the quantitative determinations.

For P6, learners should identify several types of standard procedures used in analysis in a particular industrial/ commercial context. Because of the importance of health, safety and environmental considerations these should be mentioned in addition to those procedures required to ensure the high quality of analytical data. Learners should outline in very general terms the need for these procedures or the consequences to results of not having them.

To achieve M6, learners must explain the stages in raw analytical data becoming a final report for a customer. This is best set in a particular context, eg analysis of river water requested by an environmental agency, sample of crude oil supplied from a ship to a refinery. To achieve D4, learners must explain why the range of procedures adopted in analysis is needed in detail. Learners should explain how the procedures ensure reliability, accuracy and traceability of results. Statistical control of analysis could be considered but this is not mandatory.

Programme of suggested assignments

The table below shows a programme of suggested assignments that cover the pass, merit and distinction criteria in the assessment and grading grid. This is for guidance and it is recommended that centres either write their own assignments or adapt any Edexcel assignments to meet local needs and resources.

Criteria covered	Assignment title	Scenario	Assessment method
P1, (part) M1, D1	Preparation of Standard Solutions	You are a technician who prepares solutions of accurately known concentration for other technicians to use with confidence in their analysis.	Make solutions using standard methods. Standardise the solutions by titration. Explain need for standard solutions. Explain key features of the techniques for ensuring accuracy.
P2, M2, D2	Identification of Unknown Compounds	You work for a chemical analysis laboratory which regularly identifies unknown substances found in rivers and streams.	Match spectra from substances found in the water sample with spectra from known substances using ultraviolet/ visible spectroscopy and infrared spectroscopy. Use % elemental composition, ^{1}H NMR, mass and infrared spectra to determine the identity of a compound, explaining the process in as much detail as possible.

Criteria covered	Assignment title	Scenario	Assessment method
P1, (part) P3, M3, D2	Determination of Concentration by Spectroscopy	You are an analytical technician working for a contract analytical laboratory. To be considered for promotion in a method development role within the spectroscopy laboratory, you have been asked to demonstrate carrying out a range of spectroscopic analysis techniques correctly and then to develop and prove the use of a method for finding the concentration of nickel in a solution.	Follow given methods to find the concentration of solutions of trace metals by atomic absorption and emission spectroscopy. Follow a given method to determine copper sulphate concentration by visible spectroscopy. This will involve dilution of a stock solution. Write and trial a method for finding the concentration of a nickel solution.
P4, M4, possibly D3	Separation of Mixtures by Chromatography	You have been asked to use suitable examples to write an introduction to chromatography for a new apprentice analyst.	Collate a portfolio of chromatograms and other results, from separations carried out (paper, thin layer, column) following given methods. Explain the principles of separation using chromatography with appropriate reference to work carried out.
P5, M5	Quantitative Chromatography	You work for a contract analysis laboratory and routinely measure concentrations by chromatography. Occasionally, you must develop methods for solutions of compounds not previously analysed by HPLC.	Gather data or use data provided for a quantitative application of GC and HPLC. Process the results to find the concentrations of the mixtures. For one application, eg HPLC of solutions using standards, explain how the method is developed. Explain in detail how one of the separations may be optimised.
P6, M6, D4	Quality Assurance of Data and the Need for Standard Procedures	You work for a contract analytical laboratory and have been asked to write a brochure, explaining to customers the procedures adopted to ensure that the required data supplied to them is correct.	List the procedures needed to ensure that data is correct, outlining why each is necessary. Describe how the analytical data in the laboratory is used to generate a report for the person requesting the analysis. Explain in detail how the standard procedures adopted ensure that the results of analysis are correct.

Links to National Occupational Standards, other BTEC units, other BTEC qualifications and other relevant units and qualifications

This unit forms part of the BTEC Applied Science sector suite. This unit has particular links with the following units in the BTEC Applied Science suite and the BTEC Environmental Sustainability suite:

Level 3
Working in the Science Industry
Scientific Practical Techniques
Chemistry for Biology Technicians
Chemical Laboratory Techniques
Industrial Applications of Organic Chemistry
Understanding Water Quality
Principles of Plant and Soil Science

Essential resources

Learners require access to a laboratory with a fume cupboard and a good supply of purified water. Because of the focus on instrumental techniques in spectroscopy and chromatography, learners need to be able to use these techniques at the centre or at a convenient industrial or higher education establishment. Learners need to be able to talk to industrial analysts to learn how the quality of data is assured. Learners require access to computers.

Employer engagement and vocational contexts

Where possible, learners should visit or have speakers from suitable laboratories (eg industrial, contract analytical). Work placements are especially useful. Learners must learn first hand from working analysts about how the procedures are followed to ensure accuracy and reliability of results. This will be particularly important for learners who are laboratory apprentices and who may also be studying an NVQ. If centres do not have a full range of analytical instrumentation, it is mandatory to visit higher education departments or local industry so that learners may have first-hand experience of using the instrumental techniques.

Indicative reading for learners

Textbooks

Barker J – *Mass Spectrometry* (Analytical Chemistry by Open Learning Series) (John Wiley & Sons, 1998) ISBN 9780471967620

Dean J R (editor) – *Atomic Absorption and Plasma Spectroscopy (Analytical Chemistry by Open Learning Series)* (John Wiley & Sons, 2000) ISBN 9780471972549

Downard K – *Mass Spectrometry: A Foundation Course* (Royal Society of Chemistry, 2004) ISBN 9780854046096

Faust B – *Modern Chemical Techniques: An Essential Reference for Students and Teachers* (Royal Society of Chemistry, 1997) ISBN 9780854042869

Fowlis I A – *Gas Chromatography (Analytical Chemistry by Open Learning Series)* (John Wiley & Sons, 1995) ISBN 9780471954682

Hanai T – *HPLC: A Practical Guide (RSC Chromatography Monographs)* (Royal Society of Chemistry, 1999) ISBN 9780854045150

Harris D – *Quantitative Chemical Analysis, 7th Edition* (WH Freeman, 2006) ISBN 9780716776949

Hill G and Holman J – *Chemistry in Context: Laboratory Manual and Student Guide (Chemistry in Context)* (Nelson Thornes Ltd, 2001) ISBN 9780174483076

Ho W F, Prichard E and Stuart B – *Practical Laboratory Skills Training Guides: High Performance Liquid Chromatography* (Royal Society of Chemistry, 2003) ISBN 9780854044832

Lajunen L H and Peramaki P – *Spectrochemical Analysis by Atomic Absorption and Emission, 2nd Edition* (Royal Society of Chemistry, 2005) ISBN 9780854046249

Levinson R – *More Modern Chemical Techniques* (Royal Society of Chemistry, 2002) ISBN 9780854049295

Lindsay S – *High Performance Liquid Chromatography (Analytical Chemistry by Open Learning Series)* (Wiley Blackwell, 1992) ISBN 9780471931157

Mueller-Harvey I and Baker RM – *Chemical Analysis in the Laboratory, A Basic Guide* (Royal Society of Chemistry, 2002) ISBN 9780854046461

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Details about GLP	www.mhra.gov.uk
Extensive library of organic spectra	riodb01.ibase.aist.go.jp/sdbs/cgi-bin/cre_index.cgi?lang=eng
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Delivery of personal, learning and thinking skills

The table below identifies the opportunities for personal, learning and thinking skills (PLTS) that have been included within the pass assessment criteria of this unit.

Skill	When learners are …
Independent enquirers	identifying the range of procedures used in carrying out analysis in an industrial or commercial analytical laboratory
Reflective learners	identifying unknown compounds by matching key features of a range of spectra with those of standard substances
Effective participators	preparing a range of solutions of known concentration for use in analytical techniques; determining concentrations from spectroscopic techniques; separating mixtures by chromatography; determining the concentration of mixtures with chromatographic techniques

Although PLTS are identified within this unit as an inherent part of the assessment criteria, there are further opportunities to develop a range of PLTS through various approaches to teaching and learning.

Skill	When learners are …
Independent enquirers	asking industrial analysts how they ensure that data is correct
Creative thinkers	devising a method for determining the concentration of a compound by ultraviolet/visible spectroscopy putting themselves into the role of an expert analyst in order to explain how data is assured
Reflective learners	analysing sources of errors in practical work.
Team workers	devising a set of questions which a group might ask an industrial analyst
Self-managers	completing assignment tasks by the deadlines given
Effective participators	making a positive contribution to question and answer revision sessions; carrying out analysis

Functional skills – Level 2

Skill	When learners are …
ICT – Using ICT	
Plan solutions to complex tasks by analysing the necessary stages	preparing for and carrying out practical work
Select, interact with and use ICT systems safely and securely for a complex task in non-routine and unfamiliar contexts	researching topics like the Beer-Lambert law keeping track of the content of useful websites and texts using a suitable ICT means of recording
Manage information storage to enable efficient retrieval	saving information from research as suitable files in appropriate folders
ICT – Finding and selecting information	
Use appropriate search techniques to locate and select relevant information	listing ICT-based reference sources and the strengths and weaknesses of these sources for tasks
Select information from a variety of sources to meet requirements of a complex task	identifying several sources of information and justifying the choice of particular sources, eg in the exploration of spectroscopy or chromatography tasks
ICT – Developing, presenting and communicating information	
Enter, develop and refine information using appropriate software to meet requirements of a complex task	collecting data for a formal laboratory report, including background theory on a topic, for example and account of the use of spectroscopic determinations of concentration recording numerical data as tables within text inserting block diagrams or photographs scanning initial planning documentation
Use appropriate software to meet the requirements of a complex data-handling task	gathering and using data in quantative chromatography
Use communications software to meet requirements of a complex task	emailing material, including attached files, to tutors and/or classmates using contact lists storing messages and replies in appropriate folders being observed doing the above
Combine and present information in ways that are fit for purpose and audience	collating information about the use of visible spectroscopy document presenting a report on ultraviolet/visible spectroscopy or an instructional account of method development or a promotional brochure for a potential customer explaining quality assurance
Evaluate the selection, use and effectiveness of ICT tools and facilities used to present information	explaining and justifying the methods chosen to present information and data

Skill	When learners are …
Mathematics – Representing	
Understand routine and non-routine problems in familiar and unfamiliar contexts and situations	carrying out mathematical tasks related to making up solutions and analysis of substances
Identify the situation or problems and identify the mathematical methods needed to solve them	recognising the quantities that must be calculated in order to proceed
Choose from a range of mathematics to find solutions	identifying the mathematical operations needed for calculating and measuring the numbers of moles needed to make up solutions of particular concentrates
Mathematics – Analysing	
Apply a range of mathematics to find solutions	identifying the mathematical operations needed for calculating and measuring the numbers of moles needed to make up solutions of particular concentrates
Use appropriate checking procedures and evaluate their effectiveness at each stage	calculating numbers of moles and hence mass of substance needed to make solutions of particular concentrations. Using that mass to work back to the concentration required calculating the concentration of an unknown substance from a Beer-Lambert application by using the formula from a straight line and by simply reading off the graphs for a hand drawn graph
Mathematics – Interpreting	
Interpret and communicate solutions to multistage practical problems in familiar and unfamiliar contexts and situations	presenting calculations of amounts needed to make up solutions for titrations and presenting the planning of the method for finding the concentration of a substance, say a nickel solution, where there is no given method
Draw conclusions and provide mathematical justifications	making conclusions about the concentrations of solutions, based on experiments and calculations of results justifying estimates of the possible error in analytical determinations
English – Speaking, Listening and Communication	
Make a range of contributions to discussions in a range of contexts, including those that are unfamiliar, and make effective presentations	taking part in classroom discussions, eg to analyse why standard procedures are used in industrial laboratories asking questions of experienced industrial analysts and listening to the responses discussing with other members of the group whether all laboratories should be adopting GLP

Skill	When learners are …
English – Reading	
Select, read, understand and compare texts and use them to gather information, ideas, arguments and opinions	reading information and collecting information from discussions about GLP and about UKAS accreditation recording views about whether all laboratories should use GLP
English – Writing	
Write a range of texts, including extended written documents, communicating information, ideas and opinions, effectively and persuasively	making a case for inclusion of elements of GLP in all laboratory work writing laboratory reports writing documents putting the case forward about why the results from a laboratory may be trusted.