Unit title: Chemical Laboratory Techniques

Unit code: **H/601/0355**

QCF level: **4**

Credit value: **15**

Aim

This unit gives learners the opportunity to practise and become proficient in a range of practical skills and data analysis commonly used in analytical and preparative chemistry.

Unit abstract

Science learners need to acquire a breadth and depth of practical skills in order to become proficient at experimental work across the range of disciplines embraced by the subject of chemistry. In this unit learners will become familiar with titrimetric, spectroscopic and chromatographic techniques in addition to techniques needed to prepare pure samples of compounds. They will also develop the ability to present experimental results in a variety of formats and to write different styles of report. Learners will also learn how to assess the risks associated with particular practical techniques.

On completion of the unit, learners should have developed the flexibility to use unfamiliar techniques by following given instructions and be able to report on and assess the reliability of the techniques.

Learning outcomes

On successful completion of this unit a learner will:

1 Be able to use a range of techniques in the synthesis of substances

2 Be able to use spectroscopic techniques

3 Be able to use chromatographic techniques

4 Be able to use titrimetric techniques.

Unit content

1 Be able to use a range of techniques in the synthesis of substances

Minimising risks: hazards associated with chemicals e.g. flammable, toxic, harmful; other hazards e.g. high temperatures, use of glass equipment; risk minimisation e.g. use of alternative substances, reduction of quantities, selection of method of heating, selection of location, use of fume cupboard, wearing gloves, lab coat, safety glasses, methods for handling hot objects

Preparative techniques: common procedures e.g. vacuum filtration, recrystallisation, simple distillation, fractional distillation, vacuum distillation, steam distillation, rotary evaporation, solvent extraction, drying

Substances: solid organic compounds e.g. DNP and semicarbizone derivatives, aspirin, paracetamol, antifebrin; a liquid organic compound e.g. ethyl ethanoate, cyclohexene, heptene; inorganic compounds e.g. complexes of copper or nickel, tin (IV) iodide, organometallic compounds such as 1,1 - diacetylferrocene

Tests to determine purity: melting points; boiling points e.g. Siwoloboff's method and simple distillation; spectroscopic techniques e.g. infrared spectroscopy, ultraviolet/visible spectroscopy; chromatographic techniques e.g. thin layer chromatography

Yields: theoretical and percentage yields

Report: formal laboratory report; other methods of reporting e.g. completion of a proforma, preparation of a PowerPoint presentation, making a poster, writing an article

2 Be able to use spectroscopic techniques

Guidelines: format e.g. instruction sheets, verbal instructions, instruction manuals

Spectroscopic techniques: infrared (IR) spectroscopy; ultraviolet (UV) spectroscopy; visible (Vis) spectroscopy; other techniques e.g. flame emission, atomic absorption (AA), nuclear magnetic resonance spectroscopy (NMR), mass spectroscopy (MS), x-ray fluorescence (XRF)

Analyses: use of spectra to determine purity; use of Beer-Lambert Law to determine concentrations of solutions

Appropriate degree of accuracy: in quantitative determinations e.g. comparison with reference value with given tolerance, use of class results/statistical treatments to establish appropriate tolerance

Present: format e.g. poster, documented verbal account including use of diagrams, part of a report, separate written account, PowerPoint presentation slides, verbal presentation

Principles behind techniques: component representation using block diagrams e.g. source of radiation, means of wavelength selection, nature of sample, sample container, detection method, scanning, fixed wavelength applications; Beer-Lambert Law; range of standard solutions; calibration graph; methods of calculation of unknown concentrations

Report: formal laboratory report; other methods of reporting e.g. completion of a pro forma, preparation of a PowerPoint presentation, making a poster, writing an article

3 Be able to use chromatographic techniques

Assess the risks: formal risk assessment for thin layer chromatography (TLC) involving a liquid mobile phase and a locating agent; formal risk assessment for an instrumental technique

Chromatographic separations: TLC; column chromatography; other techniques as available e.g. gas chromatography (GC), high performance liquid chromatography (HPLC), electrophoresis, ion-exchange; use of locating agents e.g. iodine, ninhydrin, cerium sulfate

Quantitative techniques: interpretation of results from GC; HPLC; integration of peak area; composition of a mixture or concentration of a solution

Present: format e.g. poster, documented verbal account including use of diagrams, part of a report, separate written account, PowerPoint presentation slides, verbal presentation

Principles: mobile phase e.g. solvent, carrier gas; stationary phase e.g. water within paper, silica, viscous liquid on GC capillary/support; sorption mechanism e.g. adsorption, partition, ion-exchange; column e.g. GC, HPLC, ion-exchange; layer e.g. paper and thin layer; detection of components e.g. colour of components, locating agent, flame ionisation detector (FID), absorption of ultraviolet light; calculation of R_f values; retention time; features of specific techniques e.g. oven in GC, pump and degassing of solvents in HPLC; block diagrams of instrumental techniques

Report: formal laboratory report; other methods of reporting e.g. completion of a pro forma, preparation of a PowerPoint presentation, making a poster, writing an article

4 Be able to use titrimetric techniques

Quantitative methods: balances e.g. top pan, analytical; volumetric equipment e.g. automated pipettes, syringes, burettes, volumetric flasks; use of primary standard solutions; dilution techniques

Range of titrimetric methods: acid-base; redox e.g. use of potassium manganate (VII), thiosulfate/iodine; complexometric e.g. use of ethylene diamine tetra-acetic acid (EDTA) to determine Cu^{2+} concentration; precipitation e.g. titration of chloride with silver nitrate; potentiometry eg use of a pH electrode; indicators; forward titrations; backward titrations

Identify the risks: formal risk assessment for a titrimetric procedure; identify aspects of given procedures which minimise the inherent risks

Report: formal laboratory report; other methods of reporting e.g. completion of a pro forma, preparation of a PowerPoint presentation, making a poster, writing an article

Learning outcomes and assessment criteria

Learning outcomes On successful completion of this unit a learner will:	**Assessment criteria for pass** The learner can:
LO1 Be able to use a range of techniques in the synthesis of substances	1.1 assess the risks inherent in syntheses 1.2 demonstrate competence in a range of preparative techniques, using safe practices 1.3 perform appropriate tests to determine the purity of synthesised substances 1.4 determine the yield of compounds prepared 1.5 report on the syntheses
LO2 Be able to use spectroscopic techniques	2.1 prepare and calibrate instruments for use following given guidelines 2.2 perform analyses using spectroscopic techniques to an appropriate degree of accuracy, using safe practices 2.3 explain the principles behind the techniques used 2.4 report on the analyses
LO3 Be able to use chromatographic techniques	3.1 assess the risks and carry out chromatographic separations, using safe practices 3.2 use results from chromatographic techniques quantitatively 3.3 explain the principles of chromatography 3.4 report on analyses that use chromatography
LO4 Be able to use titrimetric techniques	4.1 identify the risks associated with titrimetric procedures 4.2 routinely and accurately use equipment to prepare solutions using quantitative methods 4.3 use a range of titrimetric methods to determine the concentrations of solutions, using safe practices 4.4 report on the titrations

Guidance

Links

This unit has particular links with the following units within this qualification:

- *Unit reference number R/601/0352: Organic Chemistry*
- *Unit reference number R/601/0349: Inorganic Chemistry*
- *Unit reference number Y/601/0353: Physical Chemistry*
- *Unit reference number J/601/0364: Physical Chemistry of Spectroscopy, Surfaces and Chemical and Phase Equilibria*
- *Unit reference number M/601/0410: Analytical Chemistry.*
- *Unit reference number M/601/0360: Inorganic Chemistry of Crystal Structures and Transition Metal Complexes*
- *Unit reference number A/601/0362: Organic Chemistry of Aromatic and Carbonyl Compounds.*

Essential requirements

Delivery

The unit must be delivered through a well-planned programme of practical work. Learners must learn about chemical and non-chemical hazards, and be able to identify how given procedures minimise the risks associated with hazards. Learners must learn how to undertake a risk assessment of a procedure and use the correct risk and safety terminology.

The centre should select the most appropriate synthetic techniques to use. Learners must prepare two organic solids. One could be a derivative, such as a dinitrophenyl hydrazone or semi-carbizone derivative of an aldehyde or ketone. The other should be a compound other than a derivative. Learners must prepare one organic liquid and two inorganic solids. These could be transition metal complexes or organometallic compounds. In selecting the syntheses, learners must have the opportunity to use TLC to follow the progress of one of the syntheses. Learners should use chromatographic, spectroscopic and titrimetric methods to estimate the purity of the compounds made, in addition to melting point and boiling point measurements. Yield must be measured for each compound prepared.

Learners must be able to use scanning infrared and ultraviolet/visible spectroscopy and relate the spectra to the structure and bonding of the compounds. Learners should also use fixed wavelength applications, based on the Beer-Lambert Law to determine concentrations. Learners need access to Excel spreadsheets to construct calibration graphs and use the equation of the calibration graphs to calculate the unknown concentrations.

Learners must be able to use column chromatography and thin layer chromatography.

Learners require access to infrared and ultraviolet visible spectrometers, gas chromatographs and high performance liquid chromatographs. Where centres do not have these instruments visits should be arranged for learners to use the spectroscopic techniques and see the chromatographic techniques in action. Learners should be given chromatograms to interpret. Learners must be able to use, or observe the use of, as many spectroscopic techniques as possible. The operation of instruments in relation to block diagrams should be explained.

Learners must learn how to identify components of chromatograms from GC and HPLC in relation to retention time. The integration of the area under the peaks should be introduced as a quantitative measure.

Assessment

Producing a formal report for each synthetic or analytical procedure is likely to be too time consuming. A limited number of formal reports should be produced (see below). Learners should present the results from their work in a variety of ways e.g. using formal presentations, completion of pro formas, construction of posters and articles. Emphasis must be on recording and reporting all results, calculations and conclusions in an appropriate format.

In identifying how the risks inherent in syntheses may be minimised, learners must identify the chemical and non-chemical hazards for at least two syntheses. Learners need to record ways of minimising the risks from these hazards which they could do by producing a formal risk assessment for the two syntheses.

Learners must undertake five preparatory techniques i.e., two organic solids, one organic liquid and two inorganic or organometallic compounds.

Learners must measure melting points of solid compounds prepared and boiling points of the liquids prepared and interpret the results from these tests as part of the reporting of results. Depending on the facilities of the centre, learners should perform additional tests to determine the purity of substances prepared and report on the results. As part of the reporting of results, there must be a calculation of the % yield of substance prepared, based on the number of moles expected and actually produced. Learners must produce one formal report on a synthesis carried out.

Analyses using spectroscopic techniques could be qualitative or quantitative. Learners could carry out qualitative analysis in relation to synthetic techniques. Qualitative spectroscopy may simply involve identification of the functional groups present in a sample and matching spectra of unknown compounds with those of known compounds. Quantitative spectroscopy involves use of the Beer-Lambert Law. Learners could either use a given method or develop a method where standards of known concentration are made and used to find the concentration of a sample at a fixed wavelength. A colorimeter could be used for this if the centre does not have regular access to a visible spectrometer.

Learners must produce one formal report for a spectroscopic technique. This must include a description of the underlying principles and a consideration of the reliability of results. Each technique used must be reported using an appropriate format, for example by a pro forma or producing a report. Where a pro forma is used for calculations and consideration of the reliability of results, learners must produce an additional account of the principles of the technique. It is up to the centre to ensure that learners carry this out in a meaningful way.

Learners must be observed carrying out competent separations using column chromatography and thin layer chromatography. Since many such separations involve mobile phases and possibly locating agents with significant chemical hazards, learners must carry out at least one recorded formal risk assessment for such a procedure and one risk assessment for an instrumental procedure.

Learners could carry out GC and HPLC quantitatively or be given results to interpret. They must produce at least one formal report for a chromatographic technique including the principles of the technique. The results and conclusions from the other techniques may be presented as a report, pro forma, presentation or other document.

There must be a record of the titrimetric procedures that learners have carried out. When reporting on titrations, learners must consider the reliability of the results obtained. They must produce at least one formal report on a titrimetric technique and one formal risk assessment for a titrimetric procedure. Learners need to show evidence of considering the risks associated with other procedures.

Resources

Access to practical laboratory facilities, technical support, library facilities and IT resources are essential.

Royal Society of Chemistry online access to sites such as: Chemistry Hazards in Industry and the Analytical Cookbook database.

Employer engagement and vocational contexts

Where learners work in the chemical industry, discussion about the use of techniques should be encouraged. For full time learners visits should be arranged to local industry and to local higher education institutions to see the techniques in action. Guidance on assessment may be contextualised with reference to techniques used routinely in local industry.

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