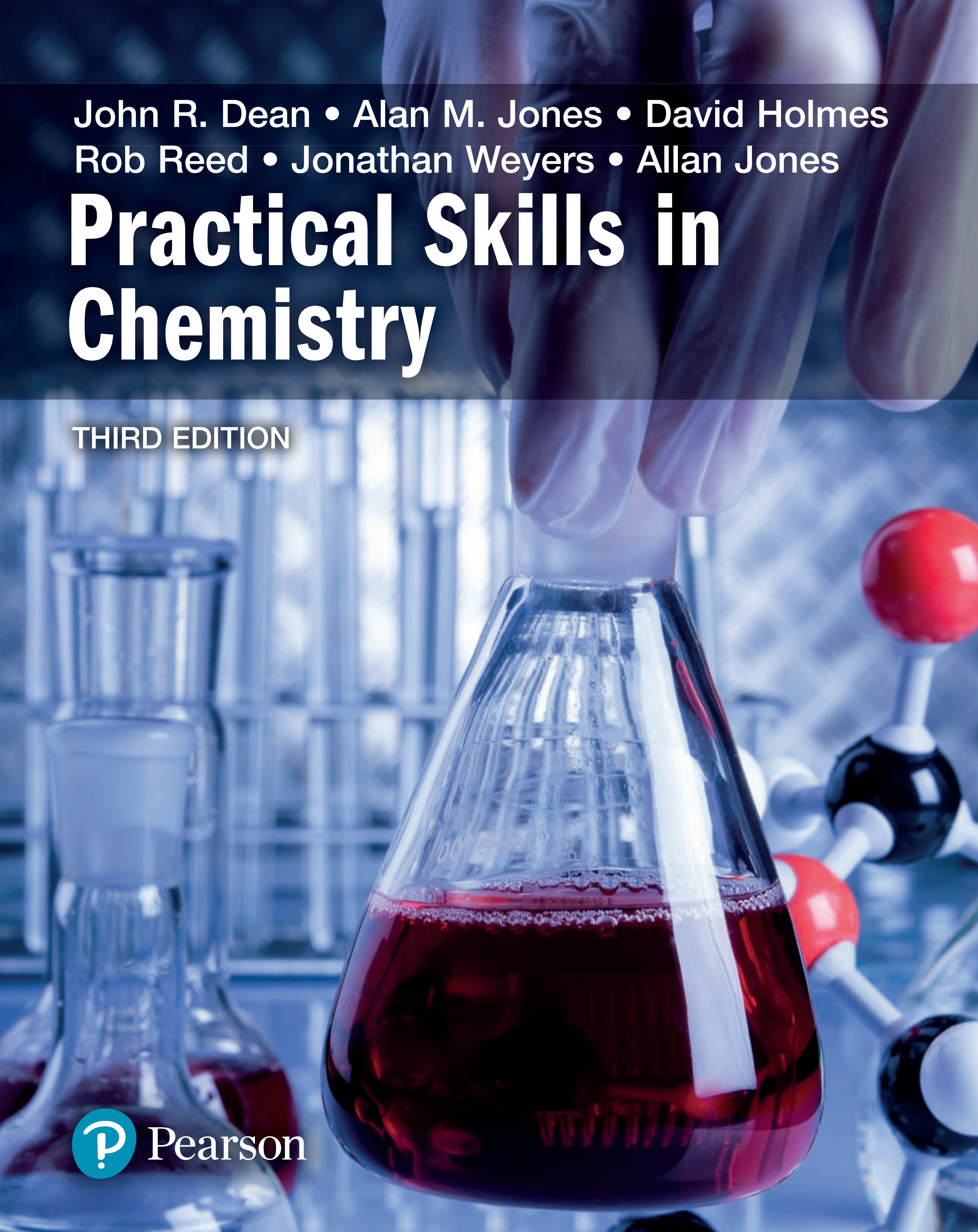
John R. Dean • Alan M. Jones • David Holmes
Rob Reed • Jonathan Weyers • Allan Jones

Practical Skills in Chemistry

THIRD EDITION



Pearson

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List of boxes

Preface to the third edition

'Chemistry can be defined as the science that studies systematically the composition properties, and reactivity of matter at the atomic and molecular level. Since matter is everything that can be touched, made visible, smelt or tasted, it follows that the scope of chemistry as a subject is very broad'.

QAA for HE Subject Benchmark Statement for Chemistry (2014).

Practical skills form the cornerstone of chemistry. However, the diversity of skills required in the laboratory means that a student's experience may be limited. While some techniques do require specific skills, many of them are transferable generic skills that are required throughout the subject area.

The time constraints of the modern curriculum often preclude or minimise laboratory time. It is the aim of this book to provide general guidance for use in and out of practical sessions and also to cover a range of techniques from the basic to the more advanced.

In creating the third edition of *Practical Skills in Chemistry*, we have maintained the approach of the previous editions, with the aim of providing support to students taking chemistry based courses in a concise and user friendly manner. Key points, definitions, illustrations, 'how to' boxes, checklists, worked examples, tips and hints are included where appropriate. However, we have also used this opportunity of the new edition to restructure the layout, to literally start at the beginning of the laboratory process and progress to the end, with the dissemination of results.

In updating and thoroughly revising the book to include a 'taste' of the latest developments in methodology, we have considered carefully the Quality Assurance Agency UK Subject Benchmarking statements for Chemistry, reviewed and updated in 2014, and have attempted to cover all of the generic skills, along with the practical aspects of the subject specific topics in chemistry. We have been mindful of two of the QAA's aims for chemistry degree (under- and post-graduate) programmes in the context of practical skills. Specifically, 'to develop in students a range of practical skills so that they can understand and assess risks and work safely and competently in the laboratory' [for undergraduate students] and 'to provide students with the ability to plan and carry out experiments independently and assess the significance of outcomes' [for postgraduate students].

To students who buy this book, we hope you will find it useful in the laboratory during your practical classes and in your project work – this is not a book to be left on the bookshelf.

We would like to take this opportunity to thank our wives and families for their continued support, and to recognise the following colleagues and friends who have provided assistance, comment and food for thought at various points during the production of all editions: Gary Askwith, Dave Bannister, Jon Bookham, Samantha Bowerbank, Susan Carlile, Michelle Carlin, Jim Creighton, Sarah Cresswell, Martin Davies, Mike Deary, Les Dix, Marcus Durrant, Jackie Eager, Gordon Forrest, Derek Holmes, Ed Ludkin, Dave Osborne, Justin Perry, Lee Rounds, Jane Shaw, Tony Simpson, Dave Wealleans and Ian Winship. We would also like to thank the staff of Pearson Education for the friendly support over the years, and would wish to acknowledge Richelle Zakrewski, Rufus Cornow, Pat Bond, Owen Knight, Simon Lake, Alex Seabrook and Pauline Gillett.

As with previous editions, we would be grateful to hear of any errors you might notice, so that these can be put right at the earliest opportunity.

JOHN R. DEAN (john.dean@northumbria.ac.uk)

ALAN M. JONES (alanjones559@btinternet.com)

DAVE HOLMES (david.holmes@northumbria.ac.uk)

ROB REED (r.reed@cqu.edu.au)

ALLAN M. JONES (a.m.jones@dundee.ac.uk)

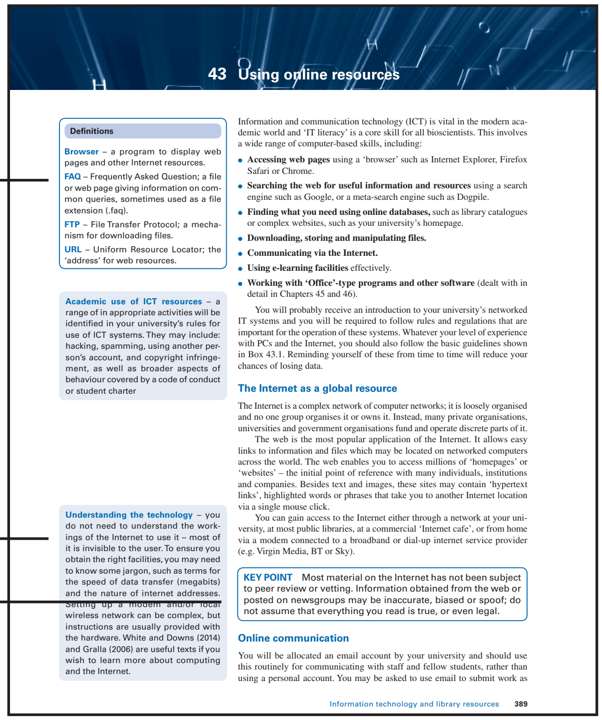
JONATHON WEYERS (j.d.b.weyers@dundee.ac.uk)

Guided tour

Definitions of key terms and concepts are highlighted in the text margin.

Tips and Hints provide useful hints and practical advice, and are highlighted in the text margin.

Key Points highlight critical features of methodology.



43 Using online resources

Definitions

Browser – a program to display web pages and other Internet resources.

FAQ – Frequently Asked Question; a file or web page giving information on common queries, sometimes used as a file extension (.faq).

FTP – File Transfer Protocol; a mechanism for downloading files.

URL – Uniform Resource Locator; the 'address' for web resources.

Academic use of ICT resources – a range of in appropriate activities will be identified in your university's rules for use of ICT systems. They may include: hacking, spamming, using another person's account, and copyright infringement, as well as broader aspects of behaviour covered by a code of conduct or student charter

Understanding the technology – you do not need to understand the workings of the Internet to use it – most of it is invisible to the user. To ensure you obtain the right facilities, you may need to know some jargon, such as terms for the speed of data transfer (megabits) and the nature of internet addresses. Setting up a modem and/or local wireless network can be complex, but instructions are usually provided with the hardware. White and Downs (2014) and Gralla (2006) are useful texts if you wish to learn more about computing and the Internet.

Information and communication technology (ICT) is vital in the modern academic world and 'IT literacy' is a core skill for all bioscientists. This involves a wide range of computer-based skills, including:

- **Accessing web pages** using a 'browser' such as Internet Explorer, Firefox Safari or Chrome.
- **Searching the web for useful information and resources** using a search engine such as Google, or a meta-search engine such as Dogpile.
- **Finding what you need using online databases,** such as library catalogues or complex websites, such as your university's homepage.
- **Downloading, storing and manipulating files.**
- **Communicating via the Internet.**
- **Using e-learning facilities** effectively.
- **Working with 'Office'-type programs and other software** (dealt with in detail in Chapters 45 and 46).

You will probably receive an introduction to your university's networked IT systems and you will be required to follow rules and regulations that are important for the operation of these systems. Whatever your level of experience with PCs and the Internet, you should also follow the basic guidelines shown in Box 43.1. Reminding yourself of these from time to time will reduce your chances of losing data.

The Internet as a global resource

The Internet is a complex network of computer networks; it is loosely organised and no one group organises it or owns it. Instead, many private organisations, universities and government organisations fund and operate discrete parts of it.

The web is the most popular application of the Internet. It allows easy links to information and files which may be located on networked computers across the world. The web enables you to access millions of 'homepages' or 'websites' – the initial point of reference with many individuals, institutions and companies. Besides text and images, these sites may contain 'hypertext links', highlighted words or phrases that take you to another Internet location via a single mouse click.

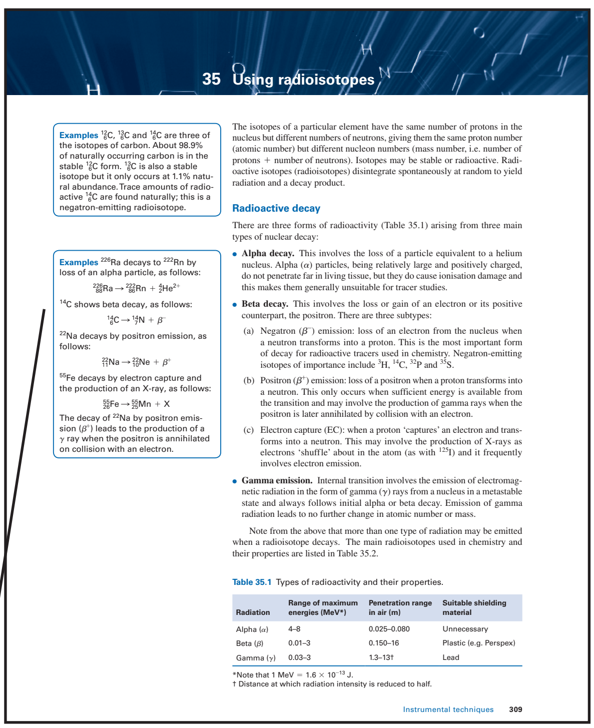
You can gain access to the Internet either through a network at your university, at most public libraries, at a commercial 'Internet cafe', or from home via a modem connected to a broadband or dial-up internet service provider (e.g. Virgin Media, BT or Sky).

KEY POINT Most material on the Internet has not been subject to peer review or vetting. Information obtained from the web or posted on newsgroups may be inaccurate, biased or spoof; do not assume that everything you read is true, or even legal.

Online communication

You will be allocated an email account by your university and should use this routinely for communicating with staff and fellow students, rather than using a personal account. You may be asked to use email to submit work as

Information technology and library resources 389



35 Using radioisotopes

Examples $^{12}_{6}C$, $^{13}_{6}C$ and $^{14}_{6}C$ are three of the isotopes of carbon. About 98.9% of naturally occurring carbon is in the stable $^{12}_{6}C$ form. $^{13}_{6}C$ is also a stable isotope but it only occurs at 1.1% natural abundance. Trace amounts of radioactive $^{14}_{6}C$ are found naturally; this is a negatron-emitting radioisotope.

Examples ^{226}Ra decays to ^{222}Rn by loss of an alpha particle, as follows:

$$^{226}_{88}Ra \rightarrow {}^{222}_{86}Rn + {}^{4}_{2}He^{2+}$$

^{14}C shows beta decay, as follows:

$$^{14}_{6}C \rightarrow {}^{14}_{7}N + \beta^-$$

^{22}Na decays by positron emission, as follows:

$$^{22}_{11}Na \rightarrow {}^{22}_{10}Ne + \beta^+$$

^{55}Fe decays by electron capture and the production of an X-ray, as follows:

$$^{55}_{26}Fe \rightarrow {}^{55}_{25}Mn + X$$

The decay of ^{22}Na by positron emission (β^+) leads to the production of a γ ray when the positron is annihilated on collision with an electron.

The isotopes of a particular element have the same number of protons in the nucleus but different numbers of neutrons, giving them the same proton number (atomic number) but different nucleon numbers (mass number, i.e. number of protons + number of neutrons). Isotopes may be stable or radioactive. Radioactive isotopes (radioisotopes) disintegrate spontaneously at random to yield radiation and a decay product.

Radioactive decay

There are three forms of radioactivity (Table 35.1) arising from three main types of nuclear decay:

- **Alpha decay.** This involves the loss of a particle equivalent to a helium nucleus. Alpha (α) particles, being relatively large and positively charged, do not penetrate far in living tissue, but they do cause ionisation damage and this makes them generally unsuitable for tracer studies.
- **Beta decay.** This involves the loss or gain of an electron or its positive counterpart, the positron. There are three subtypes:
  - (a) Negatron (β^-) emission: loss of an electron from the nucleus when a neutron transforms into a proton. This is the most important form of decay for radioactive tracers used in chemistry. Negatron-emitting isotopes of importance include ^{3}H, ^{14}C, ^{32}P and ^{35}S.
  - (b) Positron (β^+) emission: loss of a positron when a proton transforms into a neutron. This only occurs when sufficient energy is available from the transition and may involve the production of gamma rays when the positron is later annihilated by collision with an electron.
  - (c) Electron capture (EC): when a proton 'captures' an electron and transforms into a neutron. This may involve the production of X-rays as electrons 'shuffle' about in the atom (as with ^{125}I) and it frequently involves electron emission.
- **Gamma emission.** Internal transition involves the emission of electromagnetic radiation in the form of gamma (γ) rays from a nucleus in a metastable state and always follows initial alpha or beta decay. Emission of gamma radiation leads to no further change in atomic number or mass.

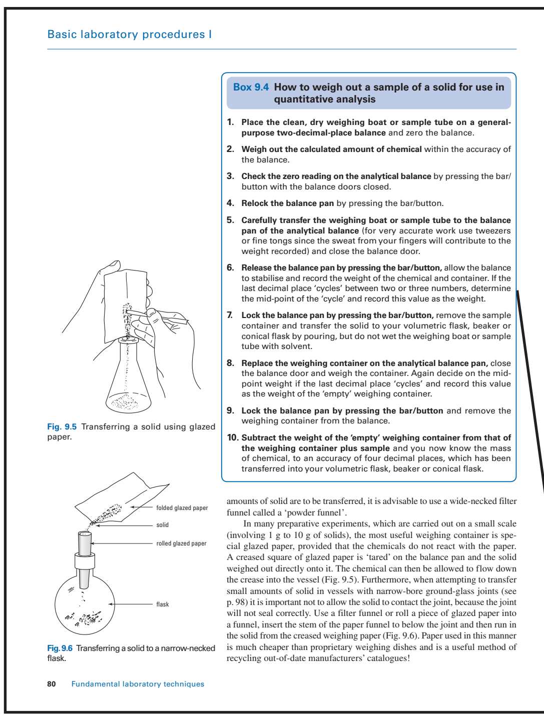
Note from the above that more than one type of radiation may be emitted when a radioisotope decays. The main radioisotopes used in chemistry and their properties are listed in Table 35.2.

Table 35.1 Types of radioactivity and their properties.

Radiation	Range of maximum energies (MeV*)	Penetration range in air (m)	Suitable shielding material
Alpha (α)	4–8	0.025–0.080	Unnecessary
Beta (β)	0.01–3	0.150–16	Plastic (e.g. Perspex)
Gamma (γ)	0.03–3	1.3–13†	Lead

*Note that 1 MeV = 1.6×10^{-13} J.
† Distance at which radiation intensity is reduced to half.

Instrumental techniques 309



Basic laboratory procedures I

Box 9.4 How to weigh out a sample of a solid for use in quantitative analysis

1. **Place the clean, dry weighing boat or sample tube on a general-purpose two-decimal-place balance** and zero the balance.
2. **Weigh out the calculated amount of chemical** within the accuracy of the balance.
3. **Check the zero reading on the analytical balance** by pressing the bar/button with the balance doors closed.
4. **Relock the balance pan** by pressing the bar/button.
5. **Carefully transfer the weighing boat or sample tube to the balance pan of the analytical balance** (for very accurate work use tweezers or fine tongs since the sweat from your fingers will contribute to the weight recorded) and close the balance door.
6. **Release the balance pan by pressing the bar/button,** allow the balance to stabilise and record the weight of the chemical and container. If the last decimal place 'cycles' between two or three numbers, determine the mid-point of the 'cycle' and record this value as the weight.
7. **Lock the balance pan by pressing the bar/button,** remove the sample container and transfer the solid to your volumetric flask, beaker or conical flask by pouring, but do not wet the weighing boat or sample tube with solvent.
8. **Replace the weighing container on the analytical balance pan,** close the balance door and weigh the container. Again decide on the mid-point weight if the last decimal place 'cycles' and record this value as the weight of the 'empty' weighing container.
9. **Lock the balance pan by pressing the bar/button** and remove the weighing container from the balance.
10. **Subtract the weight of the 'empty' weighing container from that of the weighing container plus sample** and you now know the mass of chemical, to an accuracy of four decimal places, which has been transferred into your volumetric flask, beaker or conical flask.

Fig. 9.5 Transferring a solid using glazed paper.

Fig. 9.6 Transferring a solid to a narrow-necked flask.

amounts of solid are to be transferred, it is advisable to use a wide-necked filter funnel called a 'powder funnel'.

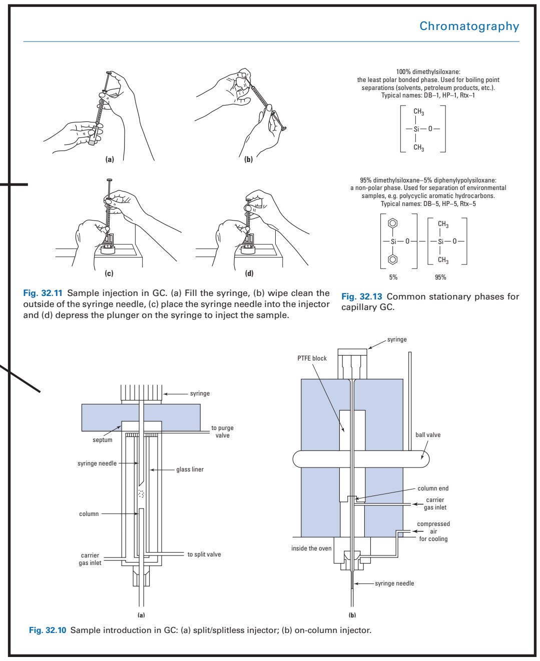
In many preparative experiments, which are carried out on a small scale (involving 1 g to 10 g of solids), the most useful weighing container is special glazed paper, provided that the chemicals do not react with the paper. A creased square of glazed paper is 'tared' on the balance pan and the solid weighed out directly onto it. The chemical can then be allowed to flow down the crease into the vessel (Fig. 9.5). Furthermore, when attempting to transfer small amounts of solid in vessels with narrow-bore ground-glass joints (see p. 98) it is important not to allow the solid to contact the joint, because the joint will not seal correctly. Use a filter funnel or roll a piece of glazed paper into a funnel, insert the stem of the paper funnel to below the joint and then run in the solid from the creased weighing paper (Fig. 9.6). Paper used in this manner is much cheaper than proprietary weighing dishes and is a useful method of recycling out-of-date manufacturers' catalogues!

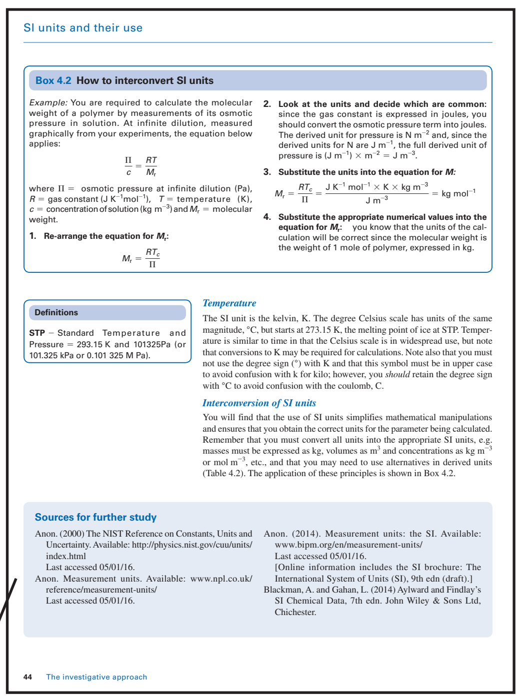
80 Fundamental laboratory techniques

Examples are included in the margin to illustrate important points without interrupting the flow of the main text.

Worked examples and **'How to' boxes** set out the essential procedures in a step-by-step manner.

Figures are used to illustrate key points, techniques and equipment.





SI units and their use

Box 4.2 How to interconvert SI units

Example: You are required to calculate the molecular weight of a polymer by measurements of its osmotic pressure in solution. At infinite dilution, measured graphically from your experiments, the equation below applies:

$$\frac{\Pi}{c} = \frac{RT}{M_r}$$

where $\Pi =$ osmotic pressure at infinite dilution (Pa), $R =$ gas constant (J $K^{-1}mol^{-1}$), $T =$ temperature (K), $c =$ concentration of solution (kg m^{-3}) and $M_r =$ molecular weight.

1. **Re-arrange the equation for M_r:**

$$M_r = \frac{RT_c}{\Pi}$$

2. **Look at the units and decide which are common:** since the gas constant is expressed in joules, you should convert the osmotic pressure term into joules. The derived unit for pressure is N m^{-2} and, since the derived units for N are J m^{-1}, the full derived unit of pressure is (J m^{-1}) $\times$ m^{-2} = J m^{-3}.
3. **Substitute the units into the equation for *M*:**

$$M_r = \frac{RT_c}{\Pi} = \frac{\text{J K}^{-1}\text{ mol}^{-1} \times \text{K} \times \text{kg m}^{-3}}{\text{J m}^{-3}} = \text{kg mol}^{-1}$$

4. **Substitute the appropriate numerical values into the equation for M_r:** you know that the units of the calculation will be correct since the molecular weight is the weight of 1 mole of polymer, expressed in kg.

Definitions

STP – Standard Temperature and Pressure = 293.15 K and 101325Pa (or 101.325 kPa or 0.101 325 M Pa).

Temperature

The SI unit is the kelvin, K. The degree Celsius scale has units of the same magnitude, °C, but starts at 273.15 K, the melting point of ice at STP. Temperature is similar to time in that the Celsius scale is in widespread use, but note that conversions to K may be required for calculations. Note also that you must not use the degree sign (°) with K and that this symbol must be in upper case to avoid confusion with k for kilo; however, you *should* retain the degree sign with °C to avoid confusion with the coulomb, C.

Interconversion of SI units

You will find that the use of SI units simplifies mathematical manipulations and ensures that you obtain the correct units for the parameter being calculated. Remember that you must convert all units into the appropriate SI units, e.g. masses must be expressed as kg, volumes as m^3 and concentrations as kg m^{-3} or mol m^{-3}, etc., and that you may need to use alternatives in derived units (Table 4.2). The application of these principles is shown in Box 4.2.

Sources for further study

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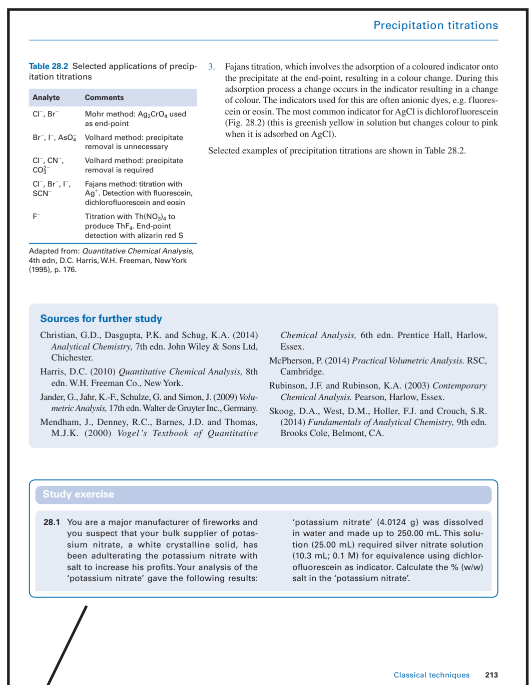
44 The investigative approach



Precipitation titrations

Table 28.2 Selected applications of precipitation titrations

Analyte	Comments
Cl^-, Br^-	Mohr method: Ag_2CrO_4 used as end-point
Br^-, I^-, AsO_4^-	Volhard method: precipitate removal is unnecessary
Cl^-, CN^-, CO_3^{2-}	Volhard method: precipitate removal is required
Cl^-, Br^-, I^-, SCN^-	Fajans method: titration with Ag^+. Detection with fluorescein, dichlorofluorescein and eosin
F^-	Titration with $Th(NO_3)_4$ to produce ThF_4. End-point detection with alizarin red S

Adapted from: *Quantitative Chemical Analysis,* 4th edn, D.C. Harris, W.H. Freeman, New York (1995), p. 176.

3. Fajans titration, which involves the adsorption of a coloured indicator onto the precipitate at the end-point, resulting in a colour change. During this adsorption process a change occurs in the indicator resulting in a change of colour. The indicators used for this are often anionic dyes, e.g. fluorescein or eosin. The most common indicator for AgCl is dichlorofluorescein (Fig. 28.2) (this is greenish yellow in solution but changes colour to pink when it is adsorbed on AgCl).

Selected examples of precipitation titrations are shown in Table 28.2.

Sources for further study

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Study exercise

28.1 You are a major manufacturer of fireworks and you suspect that your bulk supplier of potassium nitrate, a white crystalline solid, has been adulterating the potassium nitrate with salt to increase his profits. Your analysis of the 'potassium nitrate' gave the following results: 'potassium nitrate' (4.0124 g) was dissolved in water and made up to 250.00 mL. This solution (25.00 mL) required silver nitrate solution (10.3 mL; 0.1 M) for equivalence using dichlorofluorescein as indicator. Calculate the % (w/w) salt in the 'potassium nitrate'.

Classical techniques 213

Sources for further study – every chapter is supported by a section giving printed and electronic sources for further study.

Study exercises are included in every chapter to reinforce learning with problems and practical exercises.

For the student

This book aims to provide guidance and support over the broad range of undergraduate courses, as well as some postgraduate courses, including laboratory classes, project work, lectures, tutorials, seminars and examinations, as outlined below:

Chapters 1–7 (The investigative approach)

Introduce the initial key aspects of all laboratory work. Specifically, the essentials of all practical work, health and safety aspects (Risk Assessment and COSHH), making measurements, SI Units and their use, scientific method and design of experiments, making notes of practical exercises and project work.

Chapters 8–12 (Fundamental laboratory techniques)

Cover all aspects of laboratory procedures including working with liquids, solution chemistry and pH and buffer solutions.

Chapters 13–28 (Laboratory techniques)

Introduce all the basic laboratory techniques for use in chemistry. Their contents range from basic techniques used in synthetic chemistry (e.g. melting point, recrystallisation, solvent extraction, distillation, reflux and evaporation) through to more advanced areas (e.g. inert atmosphere techniques and combinatorial chemistry). In addition, classical techniques for qualitative inorganic analysis are covered as well as quantitative approaches (including gravimetry, molecular formulae and titrimetry techniques).

Chapters 29–40 (Instrumental techniques)

Cover essential relevant analytical instrumental techniques from the analysis of molecules (basic spectroscopy), elemental analysis (atomic spectroscopy, X-ray fluorescence spectroscopy), to separation techniques (chromatography and electrophoresis), to electrochemistry, use of radioisotopes and structural techniques (infrared spectroscopy, nuclear magnetic resonance spectrometry, mass spectrometry, X-ray diffraction and thermal analysis).

Chapters 41–57 (IT, internet and data analysis)

Cover all aspects of data, from finding useful and relevant information to solving a problem to useful references on 'how to' perform statistical tests.

Chapters 58–65 (Study and examination skills)

Focus on the specific skills that will allow you to work effectively to achieve optimum success during your course and beyond.

Chapters 66–71 (Communicating information)

Are key to success in chemistry; these chapters provide the essential components that you need to consolidate or improve upon to succeed.

Study exercises

Provide a valuable resource to allow you to practice and revise key aspects of selected chapters. Answers are provided at the back of the book. For numerical exercises, the working out is also provided, as well as the final answer. In some cases, the answer is in the form of tips to allow you to investigate further or provide the direction for a suitable answer.

We hope that you find this book a useful resource throughout your chosen course, and beyond.

Acknowledgements

We are grateful to the following for permission to reproduce copyright material:

EPSRC funded National Chemical Database Service hosted by the Royal Society of Chemistry; MassBank: a public repository for sharing mass spectral data for life sciences; Northumbria University for the Risk Assessment, COSHH (short) and COSHH (extended) forms; and, Sigma Aldrich Ltd. for Hazards Statements, Precautionary Statements, Pictograms – hazard codes and MSDS for phenol (as an example).

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Figures

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Tables

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List of abbreviations

A	absorbance
AAS	atomic absorption spectroscopy
AC	affinity chromatography
ACN	acetonitrile
ACS	American Chemical Society
AES	atomic emission spectroscopy
ANOVA	analysis of variance
AO	atomic orbital
APCI	atmospheric pressure chemical ionisation
A_r	relative atomic mass
ASE	accelerated solvent extraction
ATP	adenosine triphosphate
ATR	Attentuated Total Reflection
b.pt.	boiling point
CCD	central composite design
CCP	cubic close packed
CE	capillary electrophoresis
CEC	capillary electrochromatography
CGE	capillary gel electrophoresis
CI	chemical ionisation
COSHH	control of substances hazardous to health
COSY	Correlation Spectroscopy
CoV	coefficient of variation
CRM	certified reference material
CW	continuous wave
CZE	capillary zone electrophoresis
dp	decimal point
DAD	diode array detection
DCM	dichloromethane
DEPT	distortionless enhancement by polarisation transfer
DNA	deoxyribonucleic acid
dpm	disintegrations per minute
DSC	differential scanning colorimetry
DTA	differential thermal analysis
DVB	divinylbenzene
ECD	electron capture detector
EDTA	ethylenediaminetetraacetic acid
EI	electron impact (ionisation)
EIE	easily ionisable element
EF	empirical formula
EMR	electromagnetic radiation
en	ethylenediamine
EOF	electroosmotic flow
ESI	electrospray ionisation
F	Faraday constant
FAAS	flame atomic absorption spectroscopy
FID	flame ionisation detector
FT	Fourier transform
FT–IR	Fourier transform–infrared spectroscopy
GC	gas chromatography
GC–MS	gas chromatography–mass spectrometry
GFC	gel filtration chromatography
GPC	gel permeation chromatography
h	Planck constant
HASAW	hazards at work
HCB	hexachloro-1,3-butadiene
HCL	hollow cathode lamp
HCP	hexagonal close packed
HEPES	*N*-(2-hydroxyethyl)-*N*′-piperazine ethane sulphonic acid
HIC	hydrophobic interaction chromatography
HPLC	high performance liquid chromatography
HS	headspace
HRMS	high resolution mass spectrum
HTML	hypertext markup language
ICP	inductively coupled plasma
ICP–MS	inductively coupled plasma–mass spectrometry
IEC	ion exchange chromatography
IEF	isoelectric focusing
IR	infrared (radiation)
ISE	ion selective electrode
IUPAC	International Union of Pure and Applied Chemistry
K_a	acid dissociation constant
kg	kilogram
K_{ow}	octanol–water partition coefficient
K_s	solubility product
K_w	ion product of water
LC–MS	liquid chromatography–mass spectrometry
LGC	Laboratory of the Government Chemist
LOD	limit of detection
LOQ	limit of quantitation
LRMS	low resolution mass spectrum

m.pt.	melting point
MAE	microwave assisted extraction
MDL	minimum detectable level
MEKC	micellar electrokinetic chromatography
MEL	maximum exposure limit
MF	molecular formula
MO	molecular orbital
M_r	relative molecular mass
MS	mass spectrometry
NH	null hypothesis
NIST	National Institute of Standards and Technology
NMR	nuclear magnetic resonance
NP–HPLC	normal phase high performance liquid chromatography
ODS	octadecylsilane
OEL	occupational exposure standard
PAGE	polyacrylamide gel electrophoresis
PCA	principal component analysis
pdf	portable document format
PDMS	polydimethylsiloxane
PEEK	poly(etheretherketone)
PFA	perfluoroalkoxyvinylether
PFA	perfluoroalkoxy fluorocarbon
PFE	pressurised fluid extraction
pH	$\log_{10}$ proton concentration (activity)
PLOT	porous layer open tubular (column)
PMT	photomultiplier tube
ppb	parts per billion (10^9)
PPE	personal protection equipment
ppm	parts per million (10^6)
PTFE	polytetrafluoroethylene
QA	quality assurance
R	universal gas constant
RA	relative abundance
R_f	relative frontal mobility
RNA	ribonucleic acid
RP–HPLC	reversed phase high performance liquid chromatography
rpm	revolutions per minute
RSC	Royal Society of Chemistry
RSD	relative standard deviation
SAX	strong anion exchange
SCOT	support coated open tubular (column)
SCX	strong cation exchange
SDS	sodium dodecyl sulphate
SE	standard error (of the sample mean)
SEM	scanning electron microscopy
SFE	supercritical fluid extraction
SI	Système Internationale D'Unités
SPE	solid phase extraction
SPME	solid phase microextraction
STP	standard temperature and pressure
TCA	trichloroacetic acid
TCD	thermal conductivity detector
TG	thermogravimetry
TLC	thin layer chromatography
TMS	tetramethylsilane
TOF-MS	time-of-flight mass spectrometry
TRIS	tris(hydroxymethyl)aminomethane or 2-amino-2-hydroxymethyl-1,3-propanediol
UKAS	United Kingdom Accreditation Services
URL	uniform resource locator
USEPA	United States Environmental Protection Agency
UV	ultraviolet
WCOT	wall-coated open tubular (column)
www	World Wide Web
z	net charge on an ion

The investigative approach

1 Essentials of practical work

Developing practical skills – these will include:

- designing experiments
- observing and measuring
- recording data
- analysing and interpreting data
- reporting/presenting.

All knowledge and theory in science has originated from practical observation and experimentation: this is equally true for chemical disciplines as diverse as analysis and synthesis. Laboratory work is an essential part of all chemistry courses and often accounts for a significant proportion of the assessment marks. The skills and abilities developed in practical classes will continue to be useful throughout your course and beyond, some within science and others in any career you choose (see Chapter 58).

SAFETY NOTE Mobile phones – these should never be used in a lab class, as there is a risk of contamination from hazardous substances. Always switch off your mobile phone before entering a laboratory.

Being prepared

KEY POINT You will get the most out of laboratory work if you prepare well. Do not go into a practical session assuming that everything will be provided, without any input on your part.

The main points to remember are:

- **Read any handouts in advance:** make sure you understand the purpose of the practical and the particular skills involved. Does the practical relate to, or expand upon, a current topic in your lectures? Is there any additional preparatory reading that will help?
- **Take along appropriate textbooks,** to explain aspects in the practical.
- **Consider what safety hazards might be involved,** and any precautions you might need to take, before you begin (p. 6).
- **Listen carefully to any introductory guidance** and note any important points: adjust your schedule/handout, as necessary.
- **During the practical session, organise your bench space** – make sure your lab book is adjacent to, but not within, your working area. You will often find it easiest to keep clean items of glassware, etc., on one side of your working space, with used equipment on the other side.
- **All chemical waste (solid or liquid)** should be disposed of in the appropriate containers provided (consult the demonstrator or lecturer-in-charge).
- **Write up your work as soon as possible** and submit it on time or you may lose marks.
- **Catch up on any work you have missed** as soon as possible – preferably before the next practical session.

Using textbooks in the lab – take this book (or photocopies of relevant pages) along to the relevant classes, so that you can make full use of the information during the practical sessions.

SAFETY NOTE If in doubt over any part of the practical procedure – ASK! There is no such thing as a silly question in the laboratory.

Basic requirements

Recording practical results

Presenting results – while you don't need to be a graphic designer to produce work of a satisfactory standard, presentation and layout are important and you will lose marks for poorly presented work.

An A4 loose-leaf ring binder offers flexibility, since you can insert laboratory handouts, and lined and graph paper, at appropriate points. The danger of losing one or more pages from a loose-leaf system is the main drawback. Bound books avoid this problem, although those containing alternating lined/graph or lined/blank pages tend to be wasteful – it is often better to paste sheets of graph paper into a bound book, as required.

Presenting results – layout and presentation of work are important. Ensure that the information presented is legible. You will lose marks for poorly presented work. Chapter 6 gives further practical advice.

All experimental observations and data should be recorded in a notebook in ink at the time they are made because it is easy to forget when you are busy.

A good-quality HB pencil or propelling pencil is recommended for making diagrams, etc. as mistakes are easily corrected with a vinyl eraser. Buy a black, spirit-based (permanent) marker to label experimental glassware, sample tubes, etc. Fibre-tipped fine line drawing/lettering pens are useful for preparing final versions of graphs and diagrams for assessment purposes. Use a clear ruler (with an undamaged edge) for graph drawing, so that you can see data points/ information below the ruler as you draw.

Using calculators for numerical problems – Chapter 6 gives further advice.

Calculators

These range from basic machines with no pre-programmed functions and only one memory, to sophisticated programmable minicomputers with many memories. Note: Many university departments specify a particular make and model of calculator for use in examinations. It is important that you purchase and become familiar with the use of this calculator. The following may be helpful when using a calculator:

Using calculators – take particular care when using the exponential key 'EXP' or 'EE'. Pressing this key produces $10^{\text{something}}$. For example, if you want to enter 2×10^{-4}, the order entry is 2, EXP, −, 4 not 2, ×, 10, EXP, −, 4.

Using inexpensive calculators – many unsophisticated calculators have a restricted display for exponential numbers and do not show the 'power of 10', e.g. displaying 2.4×10^{-5} as 2.4^{-05}, or 2.4E–05, or even 2.4–05.

- **Power sources.** Choose a battery-powered machine, rather than a mains-operated or solar-powered type. You will need one with basic mathematical/ scientific operations including powers, logarithms (p. 472), roots and parentheses (brackets), together with statistical functions such as sample means and standard deviations (Chapter 53).
- **Mode of operation.** Calculators fall into two distinct groups. The older system used by, for example, Hewlett Packard calculators is known as the reverse Polish notation: to calculate the sum of two numbers, the sequence is 2 [enter] 4 + and the answer 6 is displayed. The more usual method of calculating this equation is as 2 + 4 =, which is the system used by the majority of modern calculators. Most newcomers find the latter approach to be more straightforward. Spend some time finding out how a calculator operates, e.g. does it have true algebraic logic ($\sqrt{}$ then number, rather than number then $\sqrt{}$)? How does it deal with scientific notation (p. 471)?
- **Display.** Some calculators will display an entire mathematical operation (e.g. '2 + 4 = 6'), while others simply display the last number/operation. The former type may offer advantages in tracing errors.
- **Complexity.** In the early stages, it is usually better to avoid the more complex machines, full of impressive-looking, but often unused preprogrammed functions – go for more memory, parentheses or statistical functions rather than engineering or mathematical constants. Programmable calculators may be worth considering for more advanced studies. However, it is important to note that such calculators are often unacceptable for exams.

Presenting more advanced practical work

Presenting graphs and diagrams – ensure these are large enough to be easily read: a common error is to present graphs or diagrams that are too small, with poorly chosen scales (see p. 453).

In some practical reports and in project work, you may need to use more sophisticated presentation equipment. Word processing may be essential and computer-based graphics packages can be useful. Choose easily read fonts such as Arial or Times New Roman for project work and posters and consider the layout and content carefully (p. 601). Alternatively, you could use fine line drawing pens plus dry-transfer lettering and symbols, such as those made by Letraset®, although this approach is usually more time consuming and less flexible than computer-based system, e.g. using Microsoft *Excel*.

Printing on acetates – standard overhead transparencies are not suitable for use in laser printers or photocopiers: you need to make sure that you use the correct type.

The use of Microsoft PowerPoint® as a presentation package is common place. It is common to find a computer and presenter available for student use. Advice on content and presentation is given in Chapter 68.

Source for further study

Bennett, S.W. and O'Neale, K. (1999) *Progressive Development of Practical Skills in Chemistry. A guide to early-undergraduate experimental work.* Royal Society of Chemistry, Cambridge.

Overton, T., Johnson, S. and Scott, J. (2015) *Study and Communication Skills for the Chemical Sciences*, 2nd edn., Oxford University Press, Oxford.

Study exercises

1.1 Consider the value of practical work. Spend a few minutes thinking about the purpose of practical work within a specific part of your course (e.g. a particular first year module) and then write a list of the six most important points. Compare your list with the generic list we have provided on p. 602, which is based on our experience as lecturers – does it differ much from your list, which is drawn up from a student perspective?

1.2 Make a list of items required for a particular practical experiment. This exercise is likely to be most useful if you can relate it to an appropriate practical session on your course. However, we have given a model list for a recrystallisation of an impure compound from water as an example.

1.3 Check your calculator skills. Carry out the following mathematical operations, using either a hand-held calculator or a PC with appropriate 'calculator' software.

(a) $5 \times (2 + 6)$
(b) $[8.3 \div (6.4 - 1.9)] \times 24$ (to 4 significant figures)
(c) $(1 \div 32) \times (5 \div 8)$ (to 3 significant figures)
(d) $1.2 \times 10^5 + 4.0 \times 10^4$ in scientific notation (see p. 471)
(e) $3.4 \times 10^{-2} - 2.7 \times 10^{-3}$ in 'normal' notation (i.e. conventional notation, not scientific format) and to 3 decimal places.

(See also numerical exercises in Chapter 51)

2 Health and safety

Health and Safety Legislation – in the UK, the **Health and Safety at Work Act 1974** provides the main legal framework for health and safety. The **Control of Substances Hazardous to Health (COSHH) Regulations 2002** impose specific legal requirements for risk assessment wherever hazardous chemicals or biological agents are used, with Approved Codes of Practice for the control of hazardous substances, carcinogens and biological agents, including pathogenic microbes.

Health and safety law requires institutions to provide a working environment that is safe and without risk to health. Where appropriate, training and information on safe working practices must be provided. Students and staff must take reasonable care to ensure the health and safety of themselves and of others, and must not misuse any safety equipment.

KEY POINT All practical work must be carried out with safety in mind, to minimise the risk of harm to yourself and to others – safety is everyone's responsibility by law.

Risk assessment

A risk assessment is a systematic approach to hazard identification and control. It is essential to consider what aspects of a laboratory activity can cause injury to people and then to introduce control measures that will reduce the risk of injury to an acceptable level. Important aspects to consider are:

- Substance hazards
- How the substance is to be used
- How it can be controlled
- Who is exposed
- How much exposure
- The duration of exposure

Definitions

Hazard – the potential of a substance to cause harm.

Risk – the likelihood that a substance will harm you and the severity of harm in the actual circumstances of use.

KEY POINT It is important to distinguish between the ***HAZARD*** of a substance and the ***RISK*** resulting from exposure.

The risk assessment process

The five step process requires you to:

1. **Identify the hazards and risk:** One way to do this is by using 'PEME,' i.e. People, Equipment, Materials and Environment.
    a. **'People' hazards** can cover a range of issues including the individual themselves and the systems that people have to use. In this 'people' context consider the following terms: training, capabilities/restrictions, supervision, communication, adequate numbers and human error.
    b. **'Equipment' hazards** relate to the equipment to be used; it will also consider related aspects of the equipment including repair, maintenance, handling, storage, cleaning and operation of the equipment.
    c. **'Materials' hazards** cover any liquid, solid or gas associated with the task. This aspect also covers any by-products or waste generated by the activity.

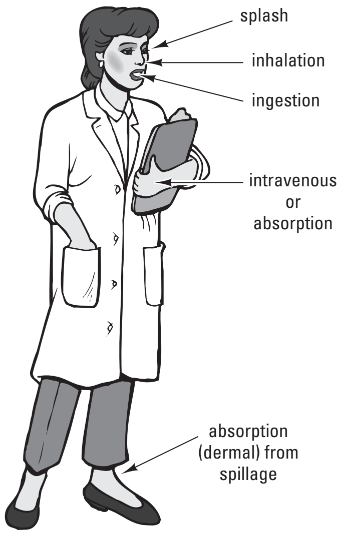

Fig. 2.1 Major routes of entry of harmful substances into the body.

SAFETY NOTE

Protective clothing is worn as a first barrier to spillage of chemicals on to your body.

Lab coats are for protection of you and your clothing.

Eye protection special spectacles with side pieces to protect you from your own mistakes and those of your colleagues. If you wear spectacles, eye protection with prescription lenses and side pieces is available from your optician, an expensive but worthwhile investment. Otherwise goggles can be worn over spectacles.

Contact lenses should not be worn in the laboratory. Chemicals can get under the lens and damage the eye before the lens can be removed. It is often very difficult to remove the contact lens from the eye after a chemical splash.

Shoes should cover the feet: no open-toed sandals, for example.

Long hair should be tied back and hats (e.g. baseball caps) should not be worn.

   d. **'Environment' hazards** relate to the surrounds you are working in. Examples include poor lighting, heating and ventilation, poor access and egress, tripping/slipping hazards, restricted space/visibility and other activities taking place nearby.

2. **Identify who can be harmed and how: Who –** Although a task may seem to be well managed, if control measures fail then a whole range of people could be injured, e.g. co-lab workers in the area or people visiting the area. Your risk assessment should consider all those people who could potentially be harmed if the control measures fail. **How** – the five routes of chemical exposure (Fig. 2.1) are: ***inhalation*** – breathing in small particles or chemical vapours is the most common exposure pathway; ***dermal*** – some chemicals can be absorbed into the body; ***ingestion*** – inadvertent hand-to-mouth transmission; ***intravenously*** – improper use of needles/glass pipettes and their disposal can lead to inadvertent exposure; ***eye contact*** – rubbing your eyes after chemical exposure with your hands (with or without gloves).

3. **Identify the current controls and decide if more is required**

   a. **Identify the control measures currently in place** for each hazard you have identified i.e. physical controls (i.e. local exhaust ventilation); procedural controls (i.e. a safe working procedure for the task); and behavioural controls (i.e. adequate supervision and monitoring of behaviour).

   b. **Identify the risks and decide on precautions – a risk matrix analysis.** A risk analysis is a qualitative estimate of risk associated with each applicable risk; it assumes that the planned or existing controls are in place. Box 2.1 shows you how to undertake a risk matrix analysis. The risk matrix evaluates the risk by allocating a numeric risk level and the tolerability of the hazard.

4. **Record your findings –** you will need to record your assessments. You will need to:

   a. state clearly what task/activity the risk assessment covers

   b. ensure that the hazards and controls are clearly listed

   c. consider all those people who could potentially be harmed

   d. ensure that the appropriate member of staff signs off the assessment (e.g. technical demonstrator; lecturer-in-charge; project supervisor)

   e. make sure the completed risk assessments are readily available to those who might need them (e.g. module tutor).

5. **Review as necessary.** Risk assessments should be reviewed on a regular basis. The period of review should reflect the hazards: the greater the hazards the more frequent the review. The risk assessments should also be reviewed, if for example, the experiment is modified in any way.

Box 2.1 How to perform a risk matrix analysis

A risk matrix analysis allows you to prioritise the likelihood and severity of risk to an individual from the hazard identified.

1. Using the form in Fig. 2.2 conduct a COSHH assessment of the chemical to be used in a practical laboratory class. If the Signal word is DANGER then the extended COSHH form should be used (Fig. 2.3).
2. First consult the **Material Safety Data Sheet** (MSDS) supplied; all manufacturers of hazardous chemicals are required to provide one of these sheets for all products which they sell.
3. Consult the **Hazard pictograms** (Fig. 2.4) for visible relevant information. In addition, **H (hazard) statements** (Table 2.1) and **P (precautionary) statements** (Table 2.2) are available on MSDS sheets and/or at: http://www.sigmaaldrich.com/help-welcome/hazard-and-precautionary-statements.html. Enter the compound name in the *search* facility, then *click* 'MSDS' at the appropriate product line.
4. Assess the 'likelihood' of harm coming to pass given the amount/nature of substance used, the environment / manner it is used in; *in the absence of any specific control measures* you should indicate the highest likelihood among the various risks (Table 2.3).
5. Assess the 'severity'; this should be substance-specific rather than activity-specific. This should relate directly to the information provided on the MSDS sheet (provided by the manufacturer); use the highest severity assessment among the various risks (Table 2.3).
6. Then, calculate the risk rating using the **risk matrix** (Table 2.4). The risk is calculated by multiplying the likelihood by the severity before any control measures additional to Good Laboratory Practice (GLP) / Personal Protective Equipment (PPE) – laboratory coat and safety glasses are factored in. This calculation of risk should quote the highest risk associated with the substance (i.e. what is the most dangerous feature of the substance).
7. You are aiming to reduce the likelihood to as close to 1 as you can get (e.g. by performing the experiment in a fume cupboard).

All manufacturers of hazardous chemicals are required to provide a Material Safety Data Sheet, or MSDS. The MSDS will contain the following information:

- Manufacturer
- Name of Chemical
- Chemical Components
- Hazards Associated with the Product
- First Aid Measures
- Fire Fighting Measures
- Handling and Storage
- Accidental Release Procedures
- Exposure Control and Personal Protection
- Physical and Chemical Properties
- Stability and Reactivity
- Toxicological and Ecological Information
- Disposal Practices
- Other miscellaneous information

An example MSDS sheet for phenol is shown in Fig. 2.5. In addition, an example of a completed COSHH form for phenol is shown in Fig. 2.6. In addition, as the Signal word is Danger an extended COSHH form (Fig. 2.3) would be required to be completed.

Hazard statements – There are 72 individual and 17 combined Hazard statements (Table 2.1). Each one of them is assigned a unique alphanumerical code which consists of one letter and three numbers as follows:

- the letter 'H' (for 'hazard statement');
- a number designating the type of hazard: '2' for physical hazards; '3' for health hazards; and '4' for environmental hazards; and finally,
- two numbers corresponding to the sequential numbering of hazards arising from the intrinsic properties of the substance or mixture, i.e. explosive properties (codes from 200 to 210), flammability (codes from 220 to 230), etc.

Precautionary statements – There are 116 individual and 33 combined Precautionary statements (Table 2.2). These are assigned a unique alphanumerical code which consists of one letter and three numbers as follows:

- the letter "P" (for 'precautionary statement');

- one number designating the type of precautionary statement: '1' for general precautionary statements; '2' for prevention precautionary statements; '3' for response precautionary statements; '4' for storage precautionary statements; and, '5' for disposal precautionary statements; and finally
- two numbers (corresponding to the sequential numbering of precautionary statements).

Experiment title: ....

Print name of assessor: .... **Signature of assessor:** .... **Date:** ....

Substance	H Statement	Hazard Key hazard(s) associated with the substance	Signal Word?	Likelihood	Severity	Risk (before additional control measures)	Specific Risk Control Measures	Controlled Risk

Substance	P Statement	Storage	Emergency Procedures (in event of spillage, fire etc.) Give specific detail:	Disposal

Fig. 2.2 Control of Substances Hazardous to Health (COSHH) form