Analytical Science

A course (in 15 Chapters), developed as an Open Educational Resource, designed for use at 2nd year England & Wales undergraduate level and as a CPD training resource

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Title	Chapter 1 – Introduction to Analytical Science
Classification	F180, Analytical Chemistry
Keywords	ukoer, sfsoer, oer, open educational resources, metadata, analytical science, cpd training resource, analytical chemistry, measurement science, analytical process model , skills for analytical science
Description	An introduction to the skills package required by the 21st century analytical scientist (chemist). Introduction to the concept of the 'Analytical Process' model and consideration of the 1st & 2nd units of this process.
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Language	English
File size	1.8 Mbytes
File format	Microsoft PowerPoint (1997 – 2003)

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Chapter 1 – Introduction to Analytical Science

Contents

Analytical science skill base

In 2000 the United Kingdom Analytical Partnership (UKAP), produced a detailed list of the skills required by the 21st century Analytical Scientist. The skills can be combined to form six skill sets which are:

- Practical analytical skills
- Knowledge skills
- Technical approach skills
- Interpretative skills
- Key skills
- Business skills

Because these are sets of skills, each of these include a number of individual skills. For instance, those related to 'Practical analytical skills' are shown on the next slide

Practical analytical skills

The practical skills considered to be important are shown below as individual sub-sets:

- Observation
- Manipulation
- Use of generic instrumentation
- Ability to follow written & verbal instructions
- Possess generic chemical practical skills

Each of these sub-sets also contains a number of individual skills. For instance ‘Manipulation’ skills include:

- Ability to weigh accurately, liquids and solids, using top-pan and analytical balances;
- Ability to transfer solids and liquids accurately, from one vessel to another;
- Ability to transfer liquids using glass and modern transfer pipettes;
- Ability to prepare standard solutions;
- Ability to dilute accurately to volume;
- Ability to handle very small sample weights and volumes.

The six skill sets are shown diagrammatically as Figure (1.1) on the next slide with explanation on the following slide .

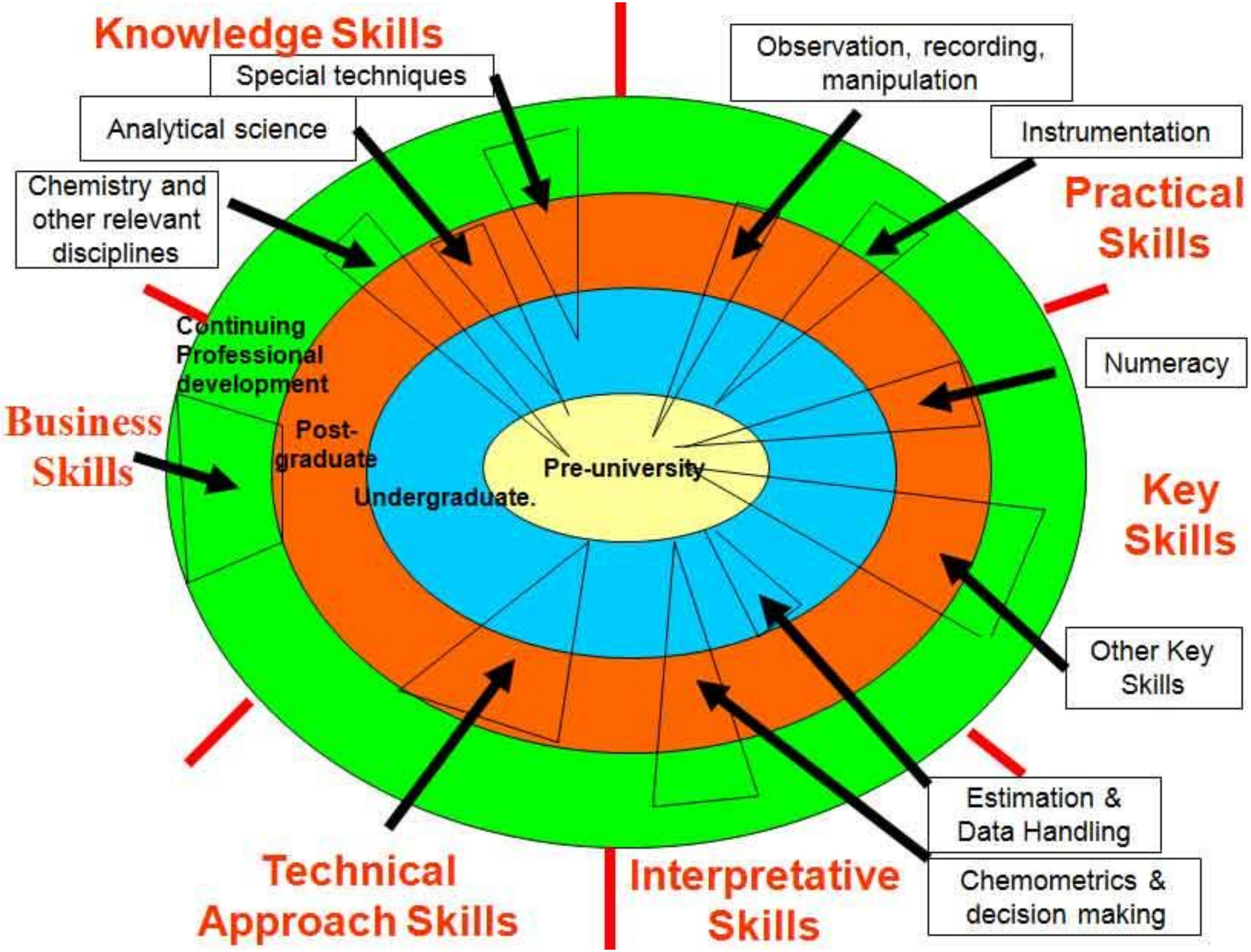

Figure 1.1 – Analytical Science skill sets

Explanation of previous slide

Each skill set has been apportioned a segment of the diagram, which consists of four concentric ellipses. Each ellipse represents a level of education and training, from stages of pre-university to continuing professional development (CPD), suggesting where the various skills should be acquired.

The ‘**Technical Approach Skills**’ and the ‘**Interpretative Skills**’ may be termed higher level skills and are most likely to be attained during post-graduate training and CPD levels.

Successful completion of this double module course, particularly if is combined with appropriate practical work, will enable you to acquire, or at least be exposed to, many of the important basic analytical skills associated with becoming a Professional Analytical Scientist.

Developing problem-solving skills

Analytical science is considered as a problem-solving science and each and every analysis may be considered as a problem that needs to be solved, some of which are of course far more complex than others. Across a wide range of industries, analytical measurements are frequently used to identify problems and thus help to bring about strategies for their solution.

The acquisition of problem-solving skills is of course not just a benefit in analytical science, but is considered as an asset across the industrial sector as a whole and is an essential component of all management training.

Generically, the solving of problems may be broken down into five strategies:

- Identifying the problem
- Representing the problem
- Selecting a strategy to solve the problem
- Implementing the strategy
- Evaluating solutions

A comparison can be made between these five generic strategies and the analytical process model, to be introduced in later in this Chapter and expanded upon in Chapters 2 – 5 in this teaching and learning programme.

Generic strategies	Process model
Identifying the problem Representing the problem	Consider the problem and decide on the objectives (Chapter 1)
Select a strategy to solve the problem	Select procedures to achieve these objectives (Chapter 1)
Implementing the strategy	Sampling (Chapter 2) Sample preparation (Chapter 3) Separation and/or concentration (Chapter 3) Measurement of target analytes (Chapter 4)
Evaluating solutions	Evaluation of the data – have the objectives been met? (Chapter 5)

Table 1.17 - comparison of generic problem-solving strategies with the analytical process model

Thus, an appreciation of the processes involved in carrying out an analysis can help with the development of problem-solving skills.

To be a problem-solver in analytical science thus requires a number of skill sets:

- Generic problem-solving skills acquired through an appreciation of the analytical process;
- Broad range of technical analytical skills, gained through study and application of the subject;
- Key skills – particularly team-working and communication skills.

In most industrial environments, complex problems are unlikely to be solved by individuals working in isolation. It is more likely that problems will be solved **by teams of scientists**, some of whom have expert technical knowledge of specific areas of analytical science - for instance, NMR and mass spectroscopy.

The role of the analytical project manager, is to be able to identify the specific technical areas where measurements and information might prove useful in helping to solve the problem. **Some general understanding/appreciation of what information, specific techniques can provide, is thus imperative**. A diagram showing the decision-making process in analytical problem-solving is shown as figure (1.2) on the next slide.

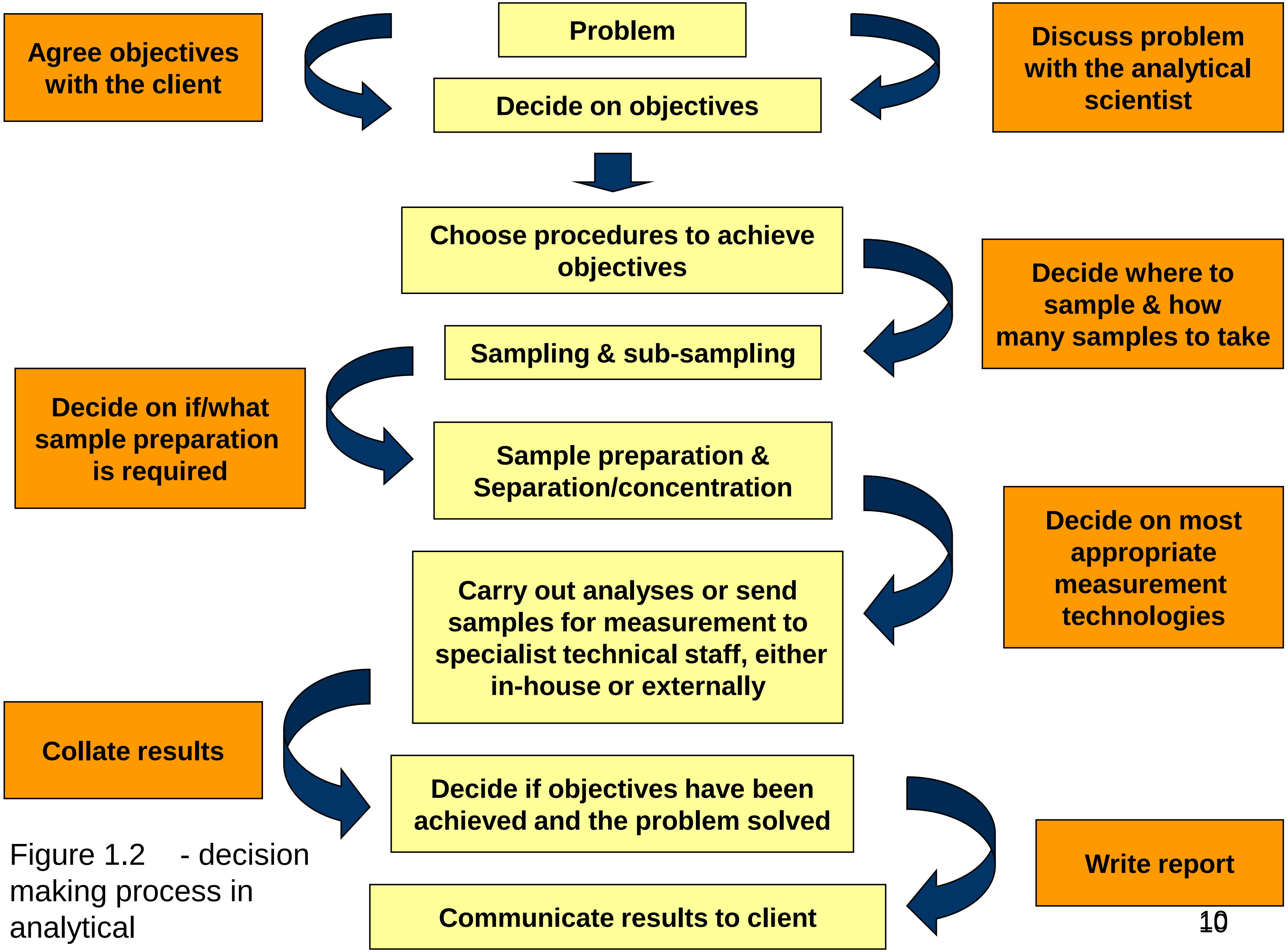

Figure 1.2 - decision making process in analytical problem-solving

Introduction to Analytical Science

Analytical Science is recognised as a key measurement technology, of critical importance to the needs of the UK's chemical and pharmaceutical industries, and to sectors such as food, medicine, the environment, forensics etc. It is truly an interdisciplinary science involving fundamentals of both natural and life sciences, together with aspects of engineering, computing and mathematics.

Analytical science is first and foremost a problem-solving science with analyses being performed in order to provide information of both a **qualitative** and **quantitative** nature. The complexity of the methods and technologies used to generate this information depends upon a number of factors including:

- the complexity of the sample to be analysed – which could be, for instance the number of components that the sample contains;
- the quantities present of the components (**target analytes**) to be measured - **macro**, **micro trace** or **ultra-trace**;
- the number of samples that need to be analysed. Automated methods are generally used when a large number of similar analyses need to be carried out.

Note: those terms shown in blue are defined on the next slides and in the 'Glossary of Terms' which accompanies this teaching and learning programme.

Qualitative analysis – identifies what chemicals, substances, ions, atoms or molecular functional groups, are present in a sample that is to be analysed.

Although some **qualitative** information can be obtained by simple chemical tests [eg: the addition of silver ions (Ag^+) to a solution containing chloride ions (Cl^-), will produce a white precipitate of silver chloride (AgCl), thereby identifying the presence of chloride ions in the sample], most **qualitative** analysis is carried out by subjecting the sample to complex instrumental analysis. Instrumental analytical techniques which can provide **qualitative** information include:

X-ray diffraction;
Nuclear magnetic resonance (NMR) spectroscopy;
Mass spectrometry;
Chromatographic techniques

Note: you will learn more about most of these techniques in later chapters of this teaching and learning programme

Identification of the crystals shown in figure (1.3) would constitute a qualitative analysis

Figure (1.3) – natural crystalline mineral

Quantitative analysis – measures the quantity of a **substance**, **compound**, **ion**, **atom** or **molecule grouping** which is present in the sample presented for analysis. Some examples of these measurements are shown in figure (1.4)

Measurement of ionic components in a sample of bottled mineral water

Measurement of the unsaturated fat content (molecular grouping) in margarines and spreads

Measurement of the atomic copper to zinc ratio of brass

Measurement of the meat and fat contents (substances) of sausages

Figure (1.4) – examples of quantitative measurements

Measurement of the active Ingredient (compound) In pharmaceuticals

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Continued on the next slide

Although some **quantitative** analysis is still performed by using classical chemical methods (eg: volumetric analysis), most **quantitative** measurement is now carried out by using sophisticated instrumental techniques based upon principles of chromatography, spectroscopy and electrochemistry. However it is important to remember that the basis of all quantitative measurement is still a calibrated analytical balance as illustrated in figure (1.5)

Figure (1.5) - Analytical balance with a set of calibrated weights

Note: although chromatographic techniques are strictly methods of separation, by combining them with detectors based for instance upon spectroscopy and electrochemistry, they are able to produce accurate qualitative and **quantitative** information.

The generic term **target analyte**, refers to that part of the sample which is being directly targeted for measurement.

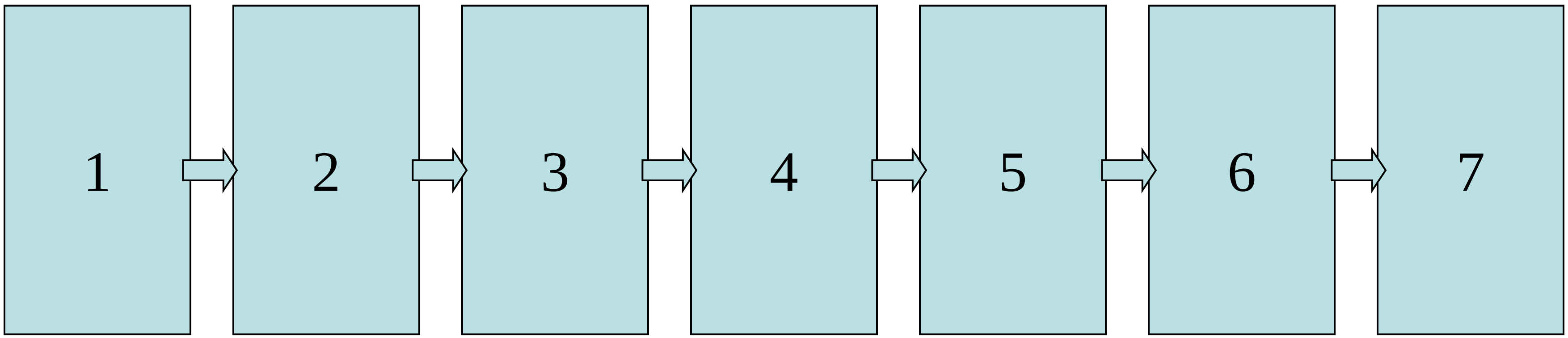
The terms **macro**, **micro**, **trace** and **ultra-trace**, refer to the levels of target analytes expected to be present in a sample matrix

Macro	Levels between 1 – 100%
Micro	Levels between 0.01 – 1%
Trace	Levels between 0.00001 – 0.01% (0.1-100 ppm)
Ultra-trace	Levels below 0.00001% (less than 0.1 ppm)

Note: units for expressing quantities of analytes present in samples will be covered later in Chapter 4 of this teaching and learning programme

The ‘Analytical Process’ model

Any analysis may be considered as consisting of a maximum of seven unit processes. These are shown diagrammatically and descriptively below:



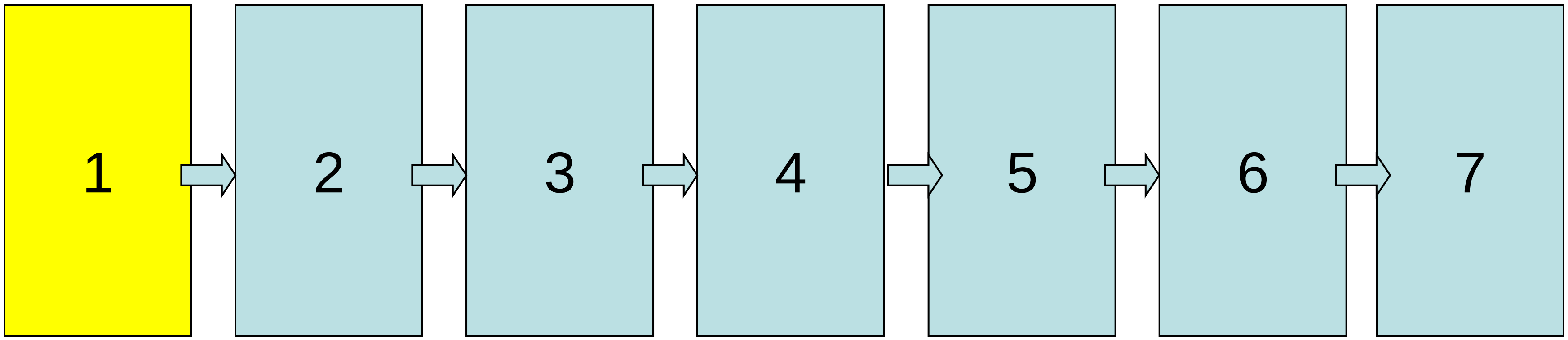
Unit 1. Consider the problem and decide on the objectives
Unit 2. Select procedure to achieve objectives
Unit 3. Sampling
Unit 4. Sample preparation
Unit 5. Separation and/or concentration
Unit 6. Measurement of target analytes
Unit 7. Evaluation of the data, have the objectives been met?

It was indicated at the start of this teaching and learning programme, that analyses are performed in order to solve problems and of course, some problems are far more complex than others.

However, by using the process model, it is possible to identify the salient features of any analysis and thereby decide what needs to be done in order to solve the problem posed. It may not always be necessary to physically carry out all of the seven unit processes, either because of the simplicity of the measurement (information) required or the specificity of the measurement technique being used.

It is important to remember, that to obtain any analytical information costs money and thus analysis should only be contemplated, when the specific objectives for carrying out the work have been established.

Process unit 1 – consideration of the problem and decision on the objectives



It is the responsibility of the Analytical Scientist in conjunction with the client or sample provider, to agree the overall objectives for the analysis. Within a large industrial organisation, a chemical plant for instance, the Analytical Scientist will often be proactive in designing process analysis (measurement) systems which may help to improve the quality of a product and/or reduce the cost of production of that produce. Let us consider some examples of what some of the objectives could be. These are shown on the next 2 slides with suggestions as to how the objective be attained in Process unit 2 (slides 21 - 25)

Example 1 – Acidity of a metal plating bath

Metal plating is normally carried out from baths which have an acid strength within certain specification limits. The objective of the analysis therefore is to see that the acidity level is maintained within these specification limits.

Example 2 – production of a discoloured pharmaceutical product

A routine production process suddenly begins to produce a discoloured product. The problem is thus the colouring of the product and the objective to identify and possibly quantify what is causing the problem and finally suggest how it might be avoided in the future.

Example 3 - Quality control of a pharmaceutical production process

The objectives here are to ensure that the product complies to BP specification. The components of the product are known and analytical methods will be chosen such that the process can be accurately controlled to produce the product within that agreed specification.

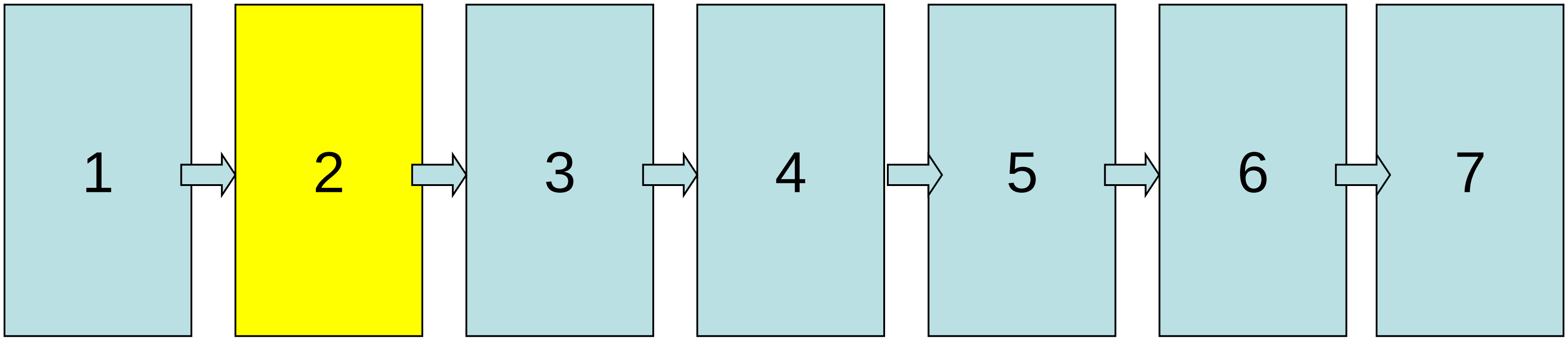
Note: BP is short for British Pharmacopoeia

Example 4 - Dead fish floating in a lake

Figure 1.6 – photograph of dead fish

Dead fish are seen to be floating in a lake. It is possible that this may be due to a chemical pollution incident. The problem is thus the dead fish and the objective is to identify the cause of the problem (what is causing the fish to die) and if possible, from where and how it arose.

Process unit 2 – choice of procedures to achieve objectives



Although there are many methods and procedures the analytical scientist can utilise, to provide the necessary analytical information to achieve the agreed objective(s), the choice of method and analytical technologies chosen should really focus on:

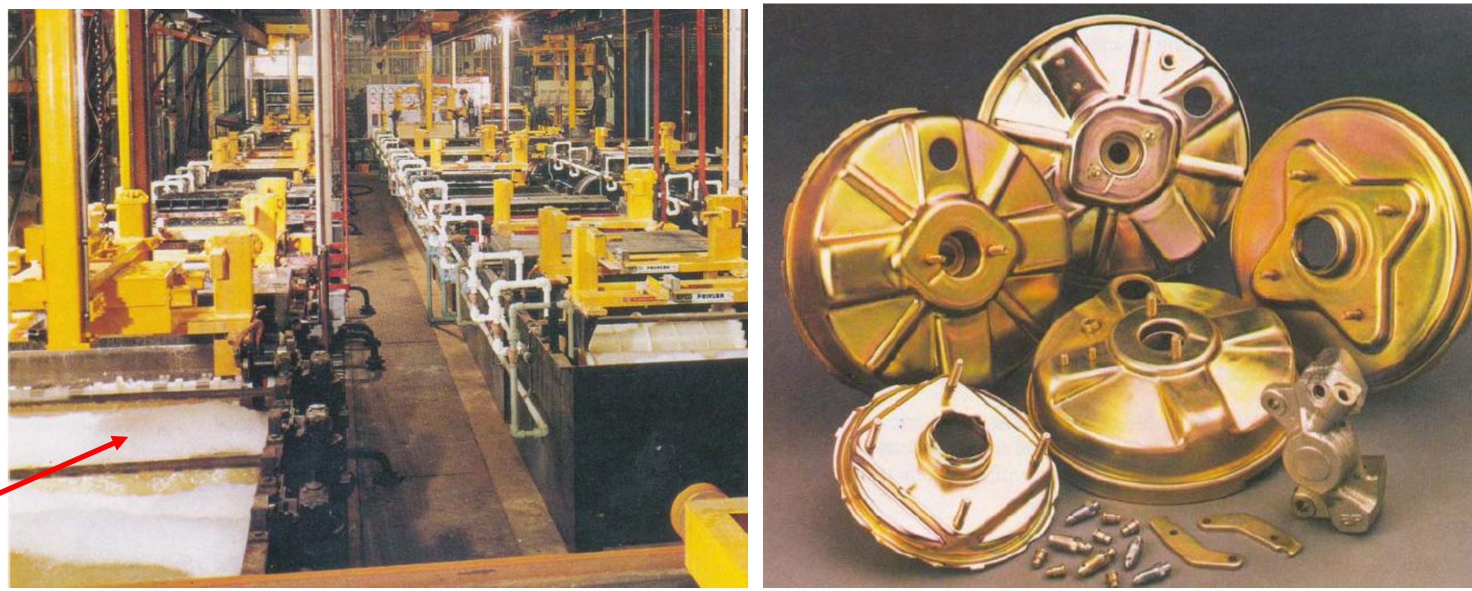
- is the analysis qualitative, quantitative or possible both;
- expected level(s) of analyte(s) from macro to ultra-micro;
- precision and accuracy required;
- the sample matrix and likely interferences present in the sample;
- the number of samples to be analysed;
- is the equipment required available ‘in-house’ or will the analysis have to be outsourced (sent to an outside contractor)?

Let us look again at the four examples used to illustrate Process unit 1

Example 1 – Acidity of a metal plating bath

Figure 1.7 – metal plating bath and Zn plated components

Plating solution To be tested



This should be a straightforward routine measurement, probably involving an acid/base titration. As the acidity is only likely to change slowly over a period of time, samples of the bath for analysis should only really need to be taken about once or twice a day. It may even be possible to train the process operatives to carry out the analysis and to report results which come outside some accepted specification limits.

The two images shown in figure 1.7 are shown by permission of the Institute of Metal Finishing

Example 2 – production of a discoloured pharmaceutical product

This is a potentially a difficult problem to solve. The level of the compound probably causing the discolouration is likely to be very low and thus the procedure to be adopted is likely to include the following stages:

- To concentrate and then isolate a sample of the discoloured material;
- To identify the nature of the discolouration probably by using spectroscopic procedures such as infra-red, nuclear magnetic resonance or mass spectroscopy.
- In conjunction with the production and research chemists, to predict how the discolouration arose;
- To develop analytical methods to monitor the future production of this by-product.

Example 3 - Quality control of a pharmaceutical production process

Pharmaceutical products have to be controlled generally within tight specification limits and thus methods developed to monitor the production must be capable of achieving the accuracy and precision demanded of the product. This will generally mean that samples for analysis will be taken at regularly specified intervals and then analysed using properly validated procedures. Measurement of target analytes will generally involve the use of chromatographic and/or spectroscopic procedures.

Note: method validation is covered in Chapter 15 of this teaching and learning package

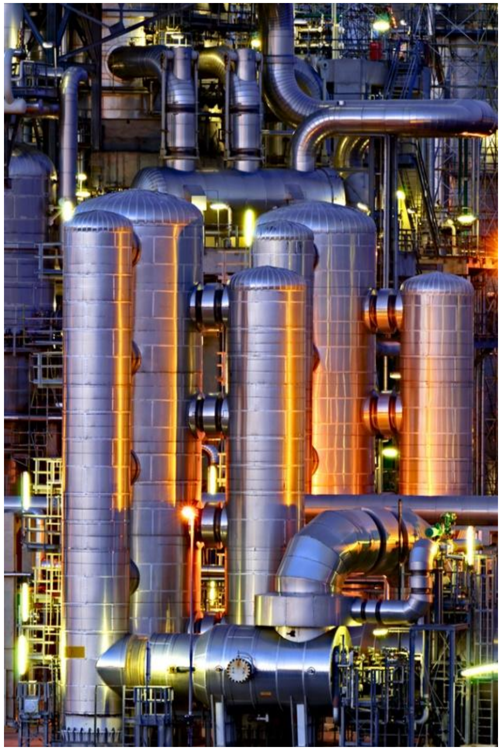


Figure 1.8 – pharmaceutical production

Example 4 - Dead fish floating in a lake

Although it may be thought initially that the fish have died (figure 1.4) due to chemical pollution, this may not necessarily be the case – viral disease, lack of oxygen in the water or simply a rise in temperature can all cause fish to die. So in this instance it will be necessary to work in conjunction with environmental specialists and bio-scientists in order to identify the cause of the problem.

If the problem is thought to be pollution related, then analysis of the dead fish may indicate whether the cause was organic or inorganic. Analysis of water samples may also be of help in attempting to identify the pollutants, however if the pollution was a ‘one-off’ occurrence, then the water samples analysed may not be representative of the water that caused the fish to die. Given that the levels of pollutants are likely to be low, a range of trace analytical techniques will need to be employed. If the pollutant can be identified then it will be necessary to look geographically at the flow of the river and industries and sewage treatment plants that are allowed to discharge into the water course.

Reflection

The previous two stages in the analytical process have involved identifying the problem and deciding on the best way that it can be solved. The remaining five stages all have practical components which will need to be addressed individually. Thus following each introduction to the process stage, there will be a more detailed coverage of topics which can be considered under each of the process headings.

Question 1.1 Distinguish between the terms ‘Qualitative’ and ‘Quantitative’ analysis. Explain by giving specific examples, how quantitative measurement may be related to a variety of chemical, molecular and substance species.
Question 1.2 Explain what is meant by the ‘Analytical Process Model’ and indicate how this may be used to help to develop problem-solving skills.
Question 1.3 List and explain the decisions that need to be taken into account when selecting methods and procedures to solve analytical problems and achieve the objectives for carrying out the analysis.

Outline answer to question number 1.1

The answer to this question may be found on slides 11 - 15.

Qualitative analysis identifies what chemicals, substances, ions, atoms or molecules are present in the sample to be analysed. Quantitative analysis measures the quantities or amounts of these chemical species. Some measurement techniques provide both qualitative and quantitative information, for instance chromatographic techniques, but in many quantitative procedures the analyte is assumed to be present in the sample and if not found is given a less than value, based upon the limit of detection.

The term ‘Chemical’, refers to a whole molecular species, for instance the analysis of caffeine in tea and coffee; the term ‘molecular’ generally relates to the measurement of parts of a molecule, for instance levels of unsaturation in processed fats; the term ‘substances’ in this context can be used to refer to groups or mixtures of chemical species that require to be analysed in ‘real-life’ situations. For instance, the meat and fat contents of processed meats and meat products such as sausages.

Outline answer to question number 1.2

The answer to this question may be found on slides 16 & 17, 7 - 10
Analytical science is often referred to as a ‘problem-solving’ science and the process model is a means of formally breaking down any analytical procedure into a discrete number of steps. The first two of these steps relate to the reason for wanting to carry out the analysis and what is to be achieved. The remaining five steps are all practical, starting with the taking of a representative sample and finishing with the evaluation of the data. In many instances in 21st century analytical procedures, some of the steps are carried out automatically as part of the measurement process. For instance, a modern automated gas chromatograph can perform a sample preparation, will separate out the analyte from other compounds present, measure the target analyte, perform a quantitative calculation based upon calibration data and carry out a statistical evaluation of the resultant data.

The separate stages (steps) within the process model are very similar to the generic strategies often used by management experts to develop problem-solving skills, and thus an awareness of the process model is of benefit in the acquisition of these important skills.

Outline answer to question number 1.3

The answer to this question may be found on slides 18 - 26

Given the variety of analytical techniques that are available to the analytical scientist, the choice of procedures to achieve a given objective are indeed vast.
Questions that need to be considered are:

- Is the analysis qualitative or quantitative?
- What are the expected levels of target analytes?
- What is the estimated level of measurement uncertainty that will be allowable?
- How many samples are to be analysed?
- Do we have the equipment or the expertise to carry out the task or will the task need to be outsourced?
- How quickly are the results required?

Many of the above questions will have financial implications. Remember that all analyses cost money to carry out and thus where possible, the cheapest option for performing the analytical task, that gives results consistent with the agreed requirements and specifications, should be chosen. For example, for the quality control of a commercial bleach product that has a wide specification limit in terms of the sodium hypochlorite content, a procedure offering accuracy and precision no better than $\pm$ 5% may be chosen. On the other hand for the quality control of a pharmaceutical product, where the specifications limits for the level of target analyte are narrow, a more accurate and precise method of analysis will be required.