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Introduction

Scientific data and results have to be accurate, precise and reliable and are subject to ever-increasing scrutiny by regulators in industry, the environment and medicine, in validation and also in research and development. Therefore, the choice of instrument to be used in particular circumstances is an important decision. Hence, analytical scientists today need a good working knowledge of the available techniques and equipment so that they can get the most out of analytical instruments and devices. Instrumentation is developing at such a rapid pace – getting smarter, smaller and faster – that it is difficult to keep up to date. This book attempts to bring together the key laboratory-based analytical techniques, hyphenated, field and portable instruments, process instrumentation in industry and trends in instrumentation such as miniaturisation. This should enable any analytical scientist to critically evaluate equipment, design suitable instrumentation for particular applications and use the most appropriate devices to solve problems and obtain results.

1.1 The Analytical Scientist

Analytical science is all around us. It pervades many disciplines such as chemistry, biology, physics, geology and engineering. It encompasses different types of analysis, such as chemical, physical, surface, materials, biomedical and environmental. Hence analytical scientists are found in all types of industrial and academic positions, from food and beverages to forensics to toxicology to pharmaceuticals to research.

A good analytical scientist must have a sound knowledge of experimental techniques in the laboratory as well as a strong theoretical knowledge of the fundamental science behind them – the analytical principles. This enables critical thinking and an understanding behind the techniques used, something that distinguishes the analytical scientist from other scientists. It is necessary to know how the equipment works, its range of applications and limitations and whether it is the best choice for the task at hand. The next step is to select the most appropriate technique for the job and analytical method development,

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which involves optimising the conditions for the analyte of interest. Analytical scientists also need to be able to critically evaluate a problem and decide on the best course of action taking into account time (people and sample turnaround), cost, availability of people and instruments, accuracy and knowledge of any knock-on effects or consequences that the result will have, and this is the analytical procedure. Hence, the analytical scientist must be proficient in all aspects of the process, from analytical principles to analytical methods and the final analytical procedure.

1.2 The Analytical Process

The analytical process is the science of taking measurements in an analytical and logical way. In practice, identifying or quantifying an analyte in a complex sample becomes an exercise in problem solving. To be efficient and effective, an analytical scientist must know the tools that are available to tackle a wide variety of different challenges. Armed also with a fundamental understanding of analytical methods, a scientist faced with a difficult analytical problem can apply the most suitable technique(s). This fundamental knowledge also makes it easier to identify when a particular problem cannot be solved by traditional methods, and gives an analyst the knowledge that is needed to develop creative approaches, hybrid instrumentation or new analytical methods. The analytical process is a logical sequence of steps that may take the form of the flowchart shown in Figure 1.1.

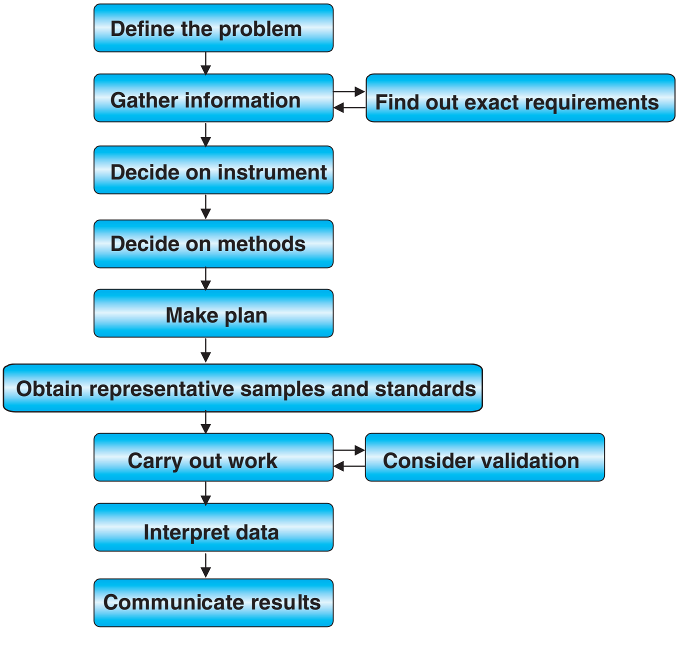

Figure 1.1 Steps in the analytical process.

When presented with a problem to solve, the analyst is likely to ask some of the following questions – What is it I am looking for? How much of it is likely to be there? Am I carrying out qualitative or quantitative analysis? What analytical technique should I use? How long will it take? How much will the assay(s) cost? The way an analysis is to be performed depends on experience, time, cost and the instrumentation available. Analysts must be able to evaluate available instruments in an open minded and critical way. Most assays used to be performed using classical methods of analysis such as gravimetry but a move towards instrumental methods began in the 1960s. Instrumental analysis is based on the measurement of a physical property of the sample and while they are generally more sensitive and selective than the older methods, they are sometimes less precise. Modern instruments are usually rapid, automated and capable of measuring more than one analyte at a time. Most instrumental methods of analysis are relative. Hence the equipment must be calibrated and the instrumental methods used on them must be validated to prove that they work reliably.

In answering the question 'what analytical technique should I use?', it must be selective for the compound of interest over the required concentration range, it must exhibit acceptable accuracy, precision and levels of sensitivity, it should be reliable, robust and easy to use, the frequency of measurement and speed of analysis must be suitable and the cost per sample should not be prohibitive. Looking at the big picture, the outlay is normally large when investing in instrumentation but in terms of the saving of time due to less labour-intensive analytical steps, in the long run such equipment usually works out to be econom.

1.3 Analytical Instrumentation

An instrument is a device that enables analytical measurements to be carried out automatically and objectively. Analytical instruments help analysts to work out composition, characterise samples, separate mixtures and yield useful results. Historically, instruments are often broken down into four component parts:

- Signal generator, e.g. radiation source
- Input transducer (detector)
- Electronic signal modifier, e.g. filter or amplifier
- Output transducer, e.g. computer.

As an example, for a UV–Vis spectrophotometer, the signal generator is the radiation source, the input transducer is the photodetector, the electronic signal modifier is a current-to-voltage converter and the output transducer is the digital display.

However, when comparing and contrasting instruments, especially across a multitude of disciplines, this and similar means of describing instruments can fall short. Another approach has been developed and reported by G. Rayson,[1] which allows a more unified description of instruments. This proposes that analytical instruments are comprised of five distinct modules:

- Source
- Sample

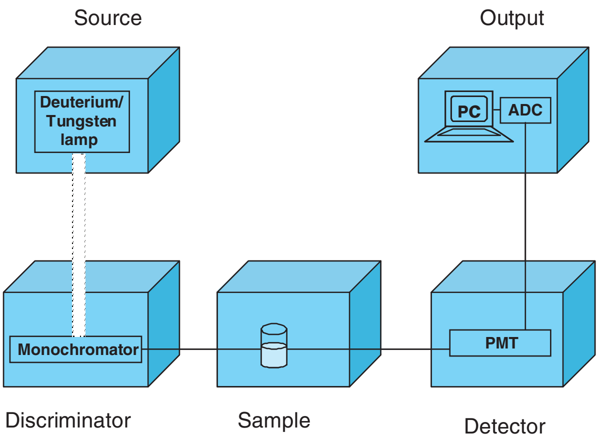

Figure 1.2 *Modules of an analytical instrument.*

- Discriminator
- Detector
- Output device

So, using this approach, for a UV–Vis spectrophotometer, the source is a deuterium/tungsten lamp, the sample is the cuvette or flowcell, the discriminator is the monochromator, the detector is a photomultiplier tube and the output device is a computer with an analogue-to-digital converter (Figure 1.2).

This means of describing instruments has been employed in this book, especially in the first section where many of the laboratory analytical instruments are discussed.

The demands on analytical instruments today are greater than ever before due to more challenging limits of sensitivity, smaller sample sizes, a wider range of applications and the growing list of new compounds that must be detected. It is fortunate, therefore, that modern instruments are improving all the time due to the availability of new technologies supporting their development. These include fibre optics, chemometrics, lasers, smaller components and more powerful computers.

1.4 Choosing the Right Instrument

To make a decision about which instrument is best to use for the job at hand, the analyst needs to know about the different types available.

Spectroscopic instruments are normally based on a compound's interaction with radiation, which yields information about its identity, quantity and/or its structure.

Separation instruments are usually based on chromatographic or electrophoretic separation of a mixture of compounds such that each can be identified and quantified. They are particularly powerful for complex samples. The detectors used in conjunction with separation techniques allow further identity information to be obtained and often structure to be elucidated.

Imaging instruments are based on close examination of the surface of a compound or material, which can allow identification, structural elucidation and an understanding of what is happening on a very small scale.

Electrochemical instruments are normally based on the changes in electrical energy that occur when a chemical reaction takes place, for example ion-selective electrodes (potentiometry) and voltammetric techniques. These can be measured in different ways and can give various qualitative and quantitative information about the reactants or products. In the case of conductivity measurements, changes in ionic content are monitored and, although nonspecific, can give useful data.

Thermoanalytical instruments allow the study of chemical and physical changes that occur with temperature, allowing the characterisation of materials and an understanding of their thermal events.

Diffraction instrumentation allows the structure of a compound at the atomic level to be understood.

As well as the general working knowledge of what instruments are capable of doing, the analyst also needs to understand the problem, the plan for solving it and the instruments that are available. If a number of techniques can be used, the analyst will need to know what the priorities are – is it sensitivity, is speed the most important factor or does expense play a role? This will help in deciding which equipment will best serve the purpose. Finally, experience is also a big factor. If an analyst is very familiar with a piece of equipment and with using it to analyse a variety of samples, it makes a big difference to the confidence in the results obtained. While, if newly trained in a technique, results may be given more tentatively. Hence, in choosing the right instrument, there are two sets of performance criteria to be considered.

The performance criteria affecting quality of the result include:

- Accuracy
- Precision (repeatability and reproducibility)
- Sensitivity (LOD and LOQ)
- Selectivity
- Linearity
- Dynamic range
- Stability.

The performance criteria for the economics include:

- Cost of purchase, installation and maintenance
- Analysis time
- Safety aspects
- Running costs – supplies, gases, consumables
- Training
- Sample throughput.

Overall, analytical instrumentation is as important as ever to the analytical scientist and to their analytical approach to problem solving. The range of equipment available today is enormous with manufacturers vying to make equipment smaller, better, more sensitive or less expensive than competitors in the market. Hence there is a need for analysts to have a strong understanding of the workings and capabilities of analytical instruments and devices so that wise decisions are made when purchasing or choosing to use one instrument over another.

Reference

1. Rayson, G. (2004) A unifying description of modern analytical instrumentation within a course on instrumental methods of analysis. *J Chem Ed*, **81 (12)**, 1767–71.