Setting standards
in analytical science

Introduction to Valid Analytical Measurements

valid analytical measurement

Outline

- Reasons for analysis
- The need for valid measurements
- The VAM programme
- Ways to ensure measurements are fit for purpose

Chemical analysis is used for many reasons and affects all aspects of our lives.

The slide shows examples of some of the analyses that are carried out at LGC. *For further information about LGC visit www.lgc.co.uk.*

Veterinary drug residues: The Veterinary Medicines Directorate runs a national surveillance programme to comply with EU legislation aimed at protecting consumers from harmful residues of veterinary drugs. Animal organs and tissues and animal feedstuffs are analysed for a range of drugs such as antibacterials, steroids and hormones.

Foodstuffs: Foodstuffs are analysed for nutrients (e.g. carbohydrates, fats, vitamins) and contaminants and also to check authenticity (e.g. detection of non-durum wheat in pasta).

Environmental samples: Soil and water samples are analysed for a range of inorganic and organic contaminants.

Bioanalysis and toxicology: Blood samples are analysed to check for the presence of drugs of abuse and to determine the level of alcohol present in drink driving cases.

Consumer safety: This involves the testing of consumer products such as cosmetics, toys and childcare articles to ensure their chemical safety. Analytes include metals, plasticisers, colourants and flame retardants.

Pesticide residues:The levels of pesticides in a range of foodstuffs, animal feeds and human and animal tissues are determined on behalf of the Pesticides Safety Directorate. The results are used to check compliance with legislation which aims to protect consumers from harmful residue levels. 'Organic' produce is also tested to ensure there are no pesticides present.

Reasons for analysis (1)

- Comparison with a regulatory limit
  - possible legal action
    - e.g. amount of cadmium released from ceramic ware
- Comparison with manufacturing control limits
  - rejection of unsatisfactory batches
    - e.g. amount of active ingredient in a tablet
- Forensic case
  - conviction
    - e.g. blood alcohol level

Measurements are always made for a reason. When an analyst carries out tests on a sample they are trying to answer a question for the customer. It is important that the analyst understands why they are carrying out a particular analysis and what the results will be used for. The reason for the analysis will, amongst other things, influence the choice of test method used. The method performance requirements for an analysis which may lead to legal action are likely to be different from those for a quick screening test which is used to decide whether more rigorous additional analysis is required.

The following slide gives some further examples of when chemical analysis is required. The customers of analytical laboratories are many and varied. They include government departments and organisations (e.g. DEFRA, Food Standards Agency, Environment Agency), local authorities, Customs and Excise, police forces, pharmaceutical companies, food manufacturers, chemical manufacturers and water companies.

Reasons for analysis (2)

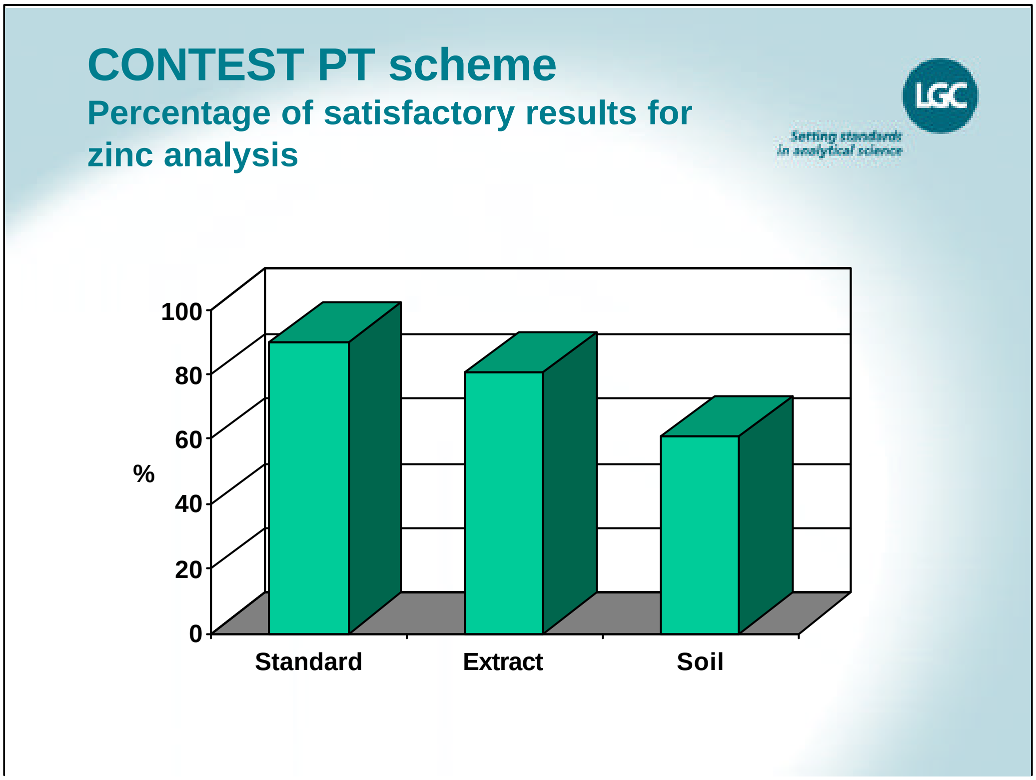
- Part of a survey
  - to determine if legislation is required to control a problem
    - e.g. plasticizer release from PVC teethers
- Long term monitoring
  - legislation or changes in practices required
    - e.g. levels of metals in foodstuffs
- Screening test to decide if further analysis is required
  - more sophisticated analysis used to confirm ‘positives’
    - e.g. drugs of abuse in urine

Evidence of problems

- Evidence suggested a large number of analytical measurements were not ‘fit for purpose’
- Poor quality data represent a major cost and risk to business and society
- Requirement for harmonisation of chemical measurements in Europe and worldwide

Over the years, data from interlaboratory studies have shown that laboratories don’t always produce results that agree. There is much historical evidence that many of the measurements made by laboratories are not fit for purpose.

Some examples are shown on the following slides.



In this study shown on the slide, laboratories participating in a proficiency testing scheme for soil analysis (the Contest PT Scheme) were asked to analyse 3 different samples using a method of their choice.

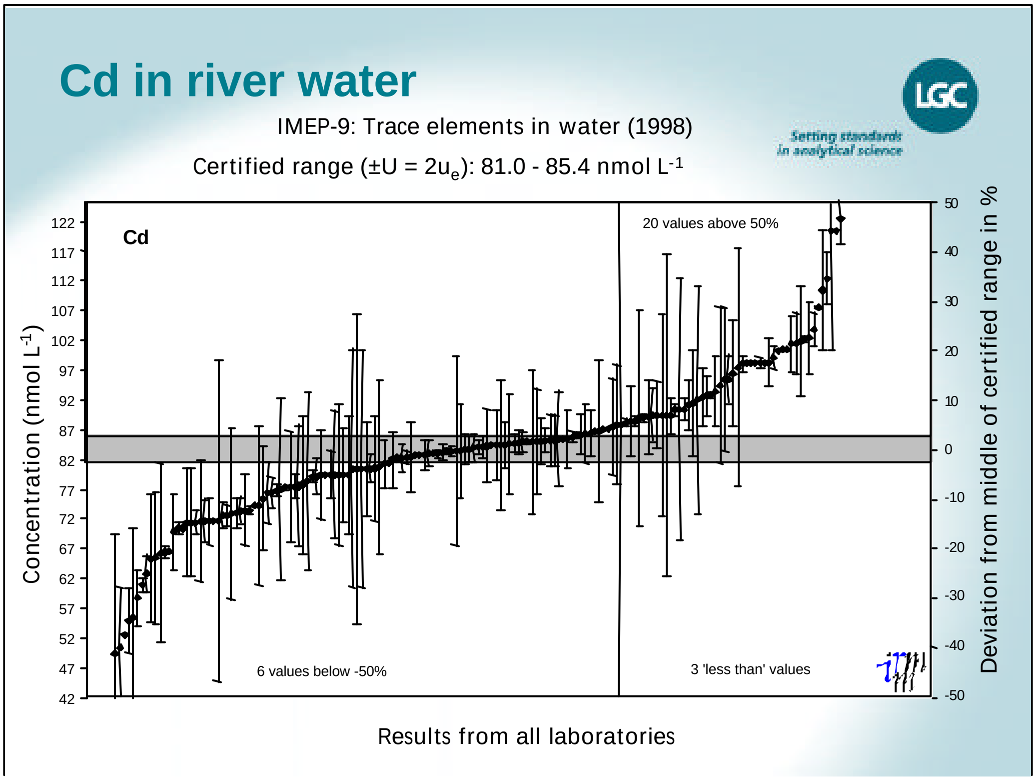
The first sample was simply a standard solution of zinc. The second sample was an extract from a soil sample. Finally the laboratories were asked to analyses a portion of a soil sample.

The majority of laboratories (90%) managed to get the correct result for the standard solution.

80% of laboratories successfully analysed the extract solution. Compared to the standard solution the extract solution was more complex as it contained other components extracted from the soil along with the zinc. These additional components might interfere with the measurement of the zinc.

In analysing the standard solution and the extract solution only the measurement capabilities of the laboratories were being evaluated. Analysis of the soil sample tests the whole analytical method including sample pre-treatment and extraction of the zinc from the soil. This proved much more challenging for laboratories. Only 61% of laboratories got an acceptable result for the soil sample.

The study showed that as the amount of sample preparation the laboratories had to do increased and the complexity of the sample increased, the more variable the results became.



IMEP is the International Measurement Evaluation Programme which is organised by the Institute for Reference Materials and Measurements(IRMM).

IMEP is a series of international interlaboratory comparison exercises which aims to study the degree of equivalence and the quality of chemical measurements by comparing participant's measurement results with external certified reference values. The results from many of the studies clearly point to a lack of agreement of measurement results.

The slide shows one set of results (for cadmium) from IMEP-9 which studied the measurement of trace elements in river water. It can be seen that there is a large spread of results. The shaded area represents the target value plus its uncertainty.

The vertical bars for each result represent the laboratory's estimate of the uncertainty associated with the value that it reported.

Further examples can be found on the IMEP website at www.irmm.jrc.be/imep/.

Need for quality
Consequences of getting it wrong

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- Forensic science - wrongful conviction
- Trade - substandard goods
- Health - drinking water contamination
- Environment - homes built on contaminated land
- New materials go undiscovered
- Impurities go unnoticed

When discussing examples of where analytical chemistry is used, it is also useful to think about the consequences of a laboratory producing unreliable results. This helps to get across the importance of taking steps to ensure the quality of analytical results.

The slide shows some of the possible consequences of analytical results that are not fit for purpose.

There is always a cost associated with poor quality analytical data. If the problem is spotted, the laboratory will have to repeat the analyses. It is costly for laboratories to have to repeat a large number of tests. It is therefore in the laboratory's interest to ensure that it gets results 'right first time'.

In a manufacturing company, erroneous data could lead to the unnecessary destruction of batches of product (or the release of sub-standard product to the market).

Unreliable data can lead to costly legal disputes, particularly if different laboratories can't agree on the answer for a particular sample.

A laboratory that produces data that do not satisfy its customers is bound to attract bad publicity which is likely to result in lost customers.

Aims of the Valid Analytical Measurement (VAM) programme

At DTI funded programme which aims to:

- Improve the quality of analytical measurements made in the UK
- Facilitate mutual recognition of analytical data across international boundaries
- Develop a robust and transparent infrastructure aimed at achieving international comparability and traceability of chemical and biochemical measurements

Measured anywhere accepted everywhere

Evidence of poor analytical results led the government to develop a programme aimed at improving the quality of analytical measurements.

The VAM programme is one of a portfolio of programmes supporting the development of the UK's National Measurement System (NMS). The NMS is the technical and organisational infrastructure that ensures a consistent and internationally recognised basis for measurement in the UK. In simple terms, this means enabling organisations in the UK to make valid measurements that are fit for purpose.

The programme covers the field of 'analytical' measurements, which are carried out widely by industry, for example, to assure the composition of manufactured products, in process control, and in research and development.

The VAM programme helps organisations in the UK to carry out analytical measurements competently and accurately. The programme enables the UK to demonstrate the comparability of analytical measurements with those of its trading partners and provides working laboratories with the 'tools' needed to implement best practice and demonstrate the reliability and integrity of their results.

Measurements: Essential and easy?

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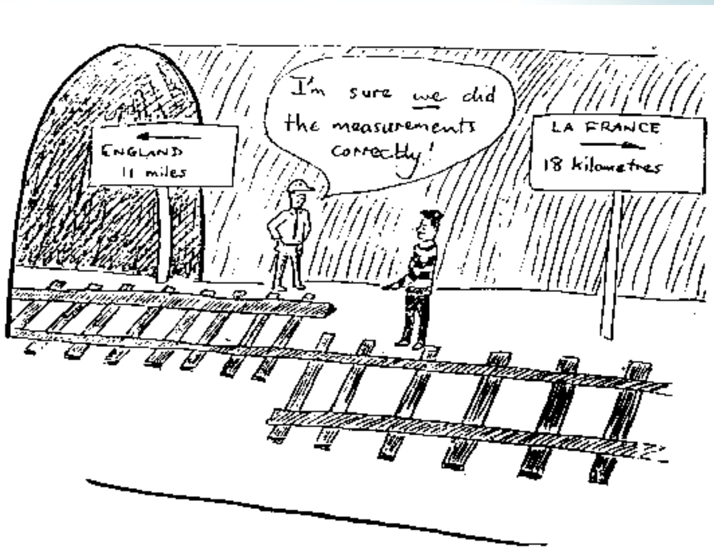

Making measurements might appear to be easy. In fact, modern instrumentation allows analysts to generate a large amount of data relatively quickly. However, as we have seen, getting the right result from a measurement is not always straightforward!

This slide can be used to generate a discussion about what can go wrong in a processes where measurements are critical. In the example shown, the outcome is obviously not fit for purpose.

Issues:

- Units of measurement: The analyst needs to be sure that measurements are made and results are reported using the correct units of measurement. NASA's Mars Climate Orbiter was lost in 1999 due to a fault in its navigation software caused by one development team using imperial units whilst another was using metric (SI) units.
- Language: Sometimes there is a 'language barrier' between the customer and the analyst. There is range of terminology relating to measurement science which may not be understood by the customer. The analyst needs to make sure that the customer's requirements have been understood.
- Specifying the problem: Agreeing the analytical requirement is an essential part of the measurement process. It is no good if the analyst makes a very accurate measurement but measures the wrong thing. In this example perhaps the requirement wasn't clearly defined.

How can laboratories prevent things from going wrong and ensure that they produce results that meet their customers' requirements?

The VAM principles

- Analytical measurements should be made to satisfy an agreed requirement
- Analytical measurements should be made using methods and equipment which have been tested to ensure they are fit for purpose
- Staff making analytical measurements should be both qualified and competent to undertake the task
- There should be a regular and independent assessment of the technical performance of a laboratory
- Analytical measurements made in one location should be consistent with those made elsewhere
- Organisations making analytical measurements should have well defined QC and QA procedures

The six VAM principles provide a framework to enable organisations to deliver reliable results first time, every time, and achieve bottom line improvements through increased operational efficiency and reduction in risk. Laboratories that adopt the VAM principles provide customers and users of data with increased confidence that results of analytical measurements are valid and fit for purpose.

The VAM principles are formalised in international quality standards such as ISO/IEC 17025, ‘General requirements for the competence of testing and calibration laboratories’.

Workshop 1

Setting standards in analytical science

- A potential client has come to you with a request for analysis that involves examining a field for contamination by diesel oil
- Consider the issues you would discuss with the client before agreeing to take on the work in your laboratory

Working in small groups students should agree on a list of the issues that they would discuss with a customer before agreeing to take on the work.

A list of the key issues is given in the document ‘Workshop 1 answers’.

Acknowledgement

- This lecture material was produced by LGC under contract with the Department of Trade and Industry as part of the National Measurement System Valid Analytical Measurement (VAM) programme
- For further information on the VAM programme visit www.vam.org.uk

Additional VAM resources

The following resources, produced under the VAM programme, may also be of interest.

Introduction to Measurement Terminology, E. Prichard, LGC, 2004, ISBN 0 948926 21 X

Introducing Measurement Uncertainty, V. Barwick, E. Prichard, LGC, 2003, ISBN 0 948926 19 8

Preparation of calibration curves: A guide to best practice, V. Barwick, LGC/VAM/2003/032

In-house method validation: A guide for chemical laboratories, LGC, 2003, ISBN 0 948926 18 X

Analytical Measurement Terminology, E. Prichard, RSC, 2001, ISBN: 0 85404 443 4

VAMSTAT II: Statistics training for valid analytical measurements, LGC, 2001

help@postgrad_studies.ok, E. Prichard, LGC/VAM/2000/098

QA training resources web area, http://www.vam.org.uk/training/training_qaofmeasurement.asp

For further information, visit the 'Publications' area of the VAM website.