

Commentary

Do we need quality assurance and quality control of analytical measurements in R&D laboratories?

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Introduction

Analysis is of critical importance to a wide range of subject areas, including those relevant to this new journal, i.e. food, agriculture and environment. There is no place within the context of good scientific research for the view that 'analysis is routine, easy and boring, i.e. not worth spending time or money on'. Gillespie¹ put this simply and succinctly: 'rubbish in, rubbish out'. Proper care during the entire analytical process, i.e. from the collection of samples to the analysis in the laboratory, can have far reaching consequences. To mention just one example, the incorporation of a new NIST Standard Reference Material for sulfur content as a tool to improve quality control (QC) during analysis significantly reduced measurement uncertainties, achieved considerable savings for manufacturers, and decreased S emissions to the environment².

Inter-Laboratory Variation

The need for comparable, useful data became obvious with the beginning of 'scientific agriculture' in the 19th century³. Different methods led to widely varying results and a great deal of confusion. The president of the Association of Official Agricultural Chemists (AOAC), now called the 'Association of Official Analytical Chemists', stated in 1896 'The matter of the analysis of foods and feedstuffs, as shown by the experience of the association, is one of the most difficult questions connected with the work of this organisation'. Numerous inter-laboratory comparisons have demonstrated that the analysis of biological samples tends to be more difficult to standardise between laboratories^{3,4}. This applies especially to some less well-defined, but nutritionally or environmentally important parameters, such as fibre, starch or digestibility of foods and animal feeds or 'available nutrients' in soils for which unacceptably large coefficients of variation have been reported⁵⁻⁸. However, other types of analyses are no exception. A report compiled in 1985 on the quality of data relating to Pb and Cd analysis in food laboratories concluded that results were inaccurate and the validity of the data was in doubt⁹. The Global Environmental Monitoring Scheme of the WHO tested the performance of EU laboratories that contribute data on food contamination¹⁰. This involved 5 proficiency tests and involved 136 laboratories in 21 countries using their own preferred methods for the analysis of trace metals (Pb, Cd, Hg in milk powder), pesticides (organochlorine, organophosphorus, pyrethroid in spinach powder), nitrate in spinach powder and aflatoxins in nut-based animal feeds. Only 60% reported accurate results for trace metals, 41% for pesticides, 43% for nitrate, 88% for aflatoxins and 53% for patulin. Recently, a major UK food manufacturer discovered that only very few laboratories achieved satisfactory results for dioxin analysis (pers. information). Problems with DDT analysis were discovered when only 71% of participants achieved satisfactory z-scores in an international proficiency testing scheme for foods and animal feeds⁴. Similarly, a feed check sample programme operated by the American Association of Feed Control Officials

(AAFCO) observed large variation in reported vitamin A results. This variation was investigated further¹¹ and resulted in several recommendations to reduce errors of Vitamin A in animal feed and pet food analysis. This shows that considerable effort is required in order to achieve reliable data.

Part of the problem seems to be that reports claiming a useful new technique are often based on tests using recently spiked materials, despite the fact that extractions from recently spiked samples are much easier and not as rigorous as a test for a robust method than extractions from aged, contaminated matrices. Whilst spiking experiments are suitable for method development, they do not represent a 'real recovery test' for a robust and reliable method as recoveries from weathered soils for example may recover only 50% of the analyte compared to spiked soils^{12,13}. It is surprising, therefore, how many scientific publications do not use CRMs to validate methods or for QC purposes¹⁴. On the other hand, it has been pointed out¹⁵ that even the incorporation of a performance evaluation standard (PES), as required by many EPA methods, can give a false sense of confidence and may not be sufficient to detect some matrix interferences, as the PES sample may have a different matrix to the real samples being tested. The authors¹⁵ commented that matrix-enhanced GC degradation might cast doubt on the quality of some data relating to DDT degradation in the environment.

Benefits of Quality Assurance Programmes

It has been observed¹⁶ that "most experimentation dealing with analytical methodology in biological sciences has been conducted within a single laboratory. Method validation by other laboratories was considered not only unnecessary but also detrimental because, in the words of one commentator, 'the results are too variable'. Within the last two decades, however, it has become increasingly apparent that a collaborative inter-laboratory study is the only way to estimate the variability characteristics of methods" and to meet the increasing demand for high quality data. The results of the UK Food Analysis Performance Assessment Scheme (FAPAS)

have been summarised from 1990-1996⁴ as follows: for pig feeds (moisture, ash, oil, protein, fibre, Cu), only 76% of laboratories achieved satisfactory results and for nutritional analysis 80% were satisfactory. However, once laboratories were participating in proficiency tests on a regular basis the average percentage for accurate results increased⁴.

How to Obtain Valid Data?

Researchers need to ensure the correct use of blanks, standards, certified reference materials (CRMs) and understand the concepts of traceability in analysis, the purpose of proficiency testing schemes and laboratory accreditation^{17, 18}. A case has been made for rigorous Quality Control procedures during routine analysis (although I would prefer to call this 'systematic analysis') and for Quality Assessment in research and development. The UK Department for Trade and Industry launched the Valid Analytical Measurement (VAM, see website in Appendix) initiative in 1994 which incorporates six principles:

1. Analytical measurements should be made to satisfy agreed requirements.
2. Analytical measurements should be made using methods and equipment, which have been tested to ensure they are fit for their purpose.
3. Staff making analytical measurements should be both qualified and competent to undertake the task.
4. There should be a regular independent assessment of the technical performance of a laboratory.
5. Analytical measurements made in one location should be consistent with those elsewhere.
6. Organisations making analytical measurements should have well defined quality control and quality assurance procedures.

These principles require properly validated methods that provide information on the performance of an analytical technique, such as accuracy and precision, ruggedness, operating range, selectivity and limits of detection. It is essential when reporting a measured value to also give its uncertainty. Otherwise, it is not possible for users of the data to know what confidence to place in the data. All Quality Assurance protocols should incorporate certified reference materials (CRMs) to ensure the traceability of measurements. This can be achieved through the use of CRMs, which can be used for:

- Calibration and verification of measurements during systematic analysis
- Internal quality control and quality assurance schemes
- Verification of the correct application of standardised methods
- Development and validation of new methods.

Significant progress has been achieved through international efforts in producing CRMs for elemental composition, pesticides and pollutants in a range of environmental and food matrices (see Appendix for CRM suppliers). Furthermore, a thorough description on how to produce in-house reference materials has been published¹⁹ which are essential if no suitable (i.e. matrix matched) CRMs are available. It is considered good practice to include both CRMs and in-house reference materials into all analytical procedures whenever possible. Independent assessment of a laboratory can be achieved by participating in national and international proficiency testing schemes (PTS). The true concentration of an analyte can be determined by addition of a known amount of analyte to a base material (given the provisos mentioned above) or better still through the use of CRMs with consensus values which have been produced by a

group of analysts (see Appendix for proficiency testing schemes). Independent approval of a laboratory's quality assurance arrangements can be obtained by accreditation to a recognised quality standard, such as ISO 90025 or ISO/IEC 17025. Guidelines are available for the statistical evaluation of analytical tests and laboratory performance⁵. Horwitz' group found that the within-laboratory variation was approximately one-half to two-thirds of the between-laboratory variation and can be used as a 'bench mark for judging previously unevaluated methods'⁵.

Conclusion - The Need to Report QC Procedures in Publications

A survey¹⁴ of a range of scientific journals found that many authors do not use CRMs appropriately. Jorhem²⁰ of the National Food Administration in Sweden strongly argued the case that all results presented in published papers should be traceable. Authors can demonstrate this by reporting their results obtained in proficiency testing schemes and from relevant CRMs. This will ensure that research, which has been conducted well, can be recognized by others – and can hopefully also be reproduced by others. It is recommended that papers that are submitted for publication in this Journal report the use of CRMs under a section entitled 'Quality Control' if standard methods have been used or under a section entitled 'Method Validation' for new or adapted methods. It is obvious, that data of reliable analytical quality are needed for evaluating the nutritional value of foods, for improving crops and for better livestock management, for natural resource management, for evaluation of soil and water quality, and for monitoring environmental contamination and land remediation.

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Appendix: Useful Websites

<http://www.aoac.org/techprog/Intro98.htm>;
<http://www.european-accreditation.org>;
<http://ptg.csl.gov.uk/schemes.cfm>;
http://www.dfrc.wisc.edu/foragetesting_nfta.html;
<http://www.lgc.co.uk/pts.asp>;
<http://www.lgc.co.uk/ref.asp>;
<http://www.vam.org.uk>;
<http://www.ukas.com>;
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