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Review

# Accreditation in the UK for pesticide residue analysis in foods

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**Abstract**

A brief background is provided of the European national bodies which accredit laboratories to the EN 45001 standard and their mutual recognition through the European Cooperation for Accreditation of Laboratories. Some of the requirements for accreditation of a laboratory, for example, the need to demonstrate traceability of measurements, are selectively illustrated with particular reference to the determination of pesticide residues in foods. The costs and benefits of accreditation are discussed.

*Keywords:* Reviews; Food analysis; Environmental analysis; Pesticides

## Contents



## 1. Introduction

An awareness of the need to ensure the quality of analytical data, especially in trace chemical analysis, has been one of the most significant and far-reaching developments over the last 10–15 years. There has been an increasing recognition that, in order to produce consistently reliable data, a laboratory must introduce, and subsequently maintain, a programme of quality assurance procedures. Accreditation by an appropriate national body is recognition both internally for the laboratory and externally to its clients that these standards have been achieved.

It must be stated that the views expressed in this paper are the personal opinions of the

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authors, based on their experience as scientists working in a United Kingdom Accreditation Service (UKAS) accredited laboratory for the determination of pesticide residues in foods. They should not be taken to represent the official policy of UKAS or any other accreditation body.

## 2. National and international accreditation bodies

UKAS is the national accreditation body in the UK. It was formed in August 1995 through an amalgamation of the work of the National Measurement Accreditation Service (NAMAS) and the National Accreditation Council for Certification Bodies (NACCB).

UKAS accredits testing and calibration laboratories to its own M10 and M11 standards, which are equivalent to the European standard EN 45001 and ISO Guide 25. UKAS is a member of the European Cooperation for Accreditation of Laboratories (EAL), which was formed in June 1994 through the amalgamation of the Western European Calibration Cooperation (WECC) and the Western European Laboratory Accreditation Cooperation (WELAC). The GATT treaty and the single European market require cross-frontier acceptance of test results. One of the objectives of EAL is the acceptance of accredited test reports throughout Europe, thus eliminating the need for multiple testing of samples. The members of EAL are listed in Table 1. Each of the member bodies accredits laboratories to EN 45001 and itself operates in accordance with the EN 45002 and EN 45003 standards. Mutual recognition agreements currently exist between most of the member bodies of EAL.

Table 1
EAL member bodies

| | | | |
|---|---|---|---|
| Austria | BMwA | Netherlands | NKO |
| Belgium | BELTEST | | /STERIN |
| Denmark | DANAK | | /STERLAB |
| Finland | FINAS | Norway | NA |
| France | COFRAC | Portugal | IPQ |
| Germany | DAR | Spain | RELE |
| Greece | ELOT | Sweden | SWEDAC |
| Iceland | ISAC | Switzerland | SAS |
| Ireland | ICLAB | UK | UKAS |
| Italy | SINAL | | |

Note: these are for testing laboratories only. Belgium, Germany, Greece and Italy have separate bodies for the accreditation of calibration laboratories.

The International Laboratory Accreditation Conference (ILAC) has similar aims to EAL on a world-wide basis. Bilateral agreements exist between EAL and several other national accreditation bodies, including those of Hong Kong, Australia, New Zealand and South Africa.

## 3. Requirements of an accredited laboratory

Some of the requirements which an accredited laboratory must fulfil are listed below:

(1) Technically competent and well trained staff
(2) Use of validated methods and on-going analytical quality control procedures
(3) Use of suitable calibrated equipment
(4) Measurements traceable (as far as possible) to national standards
(5) Clear procedures must be in place for reporting results and, where relevant, the uncertainty of measurements
(6) Clear organizational structure
(7) Suitable laboratory facilities and working environment
(8) Impartiality, independence and integrity
(9) Effective quality system, fully documented in a quality manual and related procedures

It is clear that a significant volume of paperwork, in terms of records and procedures, must be produced in order to meet these requirements. However, all laboratories must already have some form of quality system in operation, even if it is loose and possibly not fully documented. Therefore, in many cases, it is only necessary to formalize the commonly used procedures of the laboratory, which need not be too daunting a task.

The accreditation process and requirements have been very fully covered elsewhere [1,2]. It is not the intention to discuss the more general issues in this paper, but instead selectively to consider a few of those aspects which are of the

most interest and are perhaps the most difficult to achieve in practice when applied to pesticide residue analysis.

## 4. Analytical methods

Unlike the Good Laboratory Practice (GLP) scheme, individual analytical methods are accredited, not the work of the whole laboratory. There is no requirement to use nationally or internationally collaboratively tested methods, such as those published by AOAC International, although these are preferred if available and suitable. For the multi-residue determination of pesticides in foods, because of the wide variety of matrices and the rapid pace of analytical developments, it is likely that most of the methods in regular use in laboratories will have been developed internally. This is no barrier to accreditation, provided that adequate validation data are generated and quality control procedures are in place.

## 5. Analytical quality control procedures

In the UK, the analytical quality control (AQC) procedures of laboratories which submit data to the MAFF Working Party on Pesticide Residues (WPPR) have been fully documented in a recent annual report [3]. These procedures have proved acceptable, in practice, to UKAS.

For quality control, on a daily basis, every batch of samples (even if consisting of only one sample) must be accompanied by a "blank" (analyte-free) sample to guard against false positives and the same blank sample spiked with known concentrations of pesticides to measure recoveries. With certain food commodities, e.g. eels, it may be very difficult or even impossible to find pesticide-free samples. The recovery spike should then be added to a previously analysed sample at a level at least twice that of the sample. In these cases, it is essential to include also a reagent blank with each batch of samples. In certain circumstances, a simulated blank matrix may be used for spiking.

In all cases, the recovery of each pesticide being sought must be determined with every batch of samples. Thus, with multi-residue analyses based on GC or HPLC, where many pesticides are sought in the same extract, this requirement can create severe practical difficulties with regard to co-eluting analyte peaks. If it is not possible to resolve all the peaks of interest in the spike chromatogram using conventional detectors, then GC–MS or LC–MS may need to be employed. However, in situations where this fails to solve the problem, owing, for example, to similarity of the ions formed, then the only alternative is to include more than one spike with a batch of samples, each spike containing one member of a pair of unresolvable compounds.

## 6. Traceability of measurements

In contrast to physical measurements such as length, mass, time or temperature, the traceability of pesticide analysis to national and international standards is difficult to achieve. Fortunately, this is accepted by accreditation bodies whose assessors are often themselves experienced analysts in the field, and are thus aware of the problems and likely to take a pragmatic view. A laboratory must, however, demonstrate that it is doing everything that is reasonably practical to achieve traceability. This can include purchasing standards of certified purity, analysing Certified Reference Materials (CRMs) and participating in proficiency testing schemes. However, there are problems associated with all these aspects, as considered below.

### *6.1. Standards of "pure" pesticides*

Wherever possible, laboratories should only purchase pesticides of certified purity from reputable suppliers. However, these are rarely, if ever, truly traceable to international standards. In most cases, these compounds can be relied upon. However, there have been two cases in the authors' laboratory where a certified standard proved to be not just of incorrect purity but was, in fact, a different compound to that stated. If there is any doubt surrounding the authenticity or purity of a purchased standard, it must be

checked by appropriate means, e.g. GC, MS, IR, NMR.

It is good practice occasionally to exchange working standard solutions of a mixture of pesticides with another accredited laboratory. This will provide checks both on the purity of the individual compounds and also on the procedures in place in each laboratory for the preparation and storage of pesticide standard mixtures.

### *6.2. Certified Reference Materials and internal laboratory reference materials*

Only a few CRMs, with certified values, are available for the determination of pesticide residues in foods. These are exclusively for organochlorine (OC) compounds. There is an obvious requirement for CRMs containing organophosphorus (OP) and other pesticides to be produced and made widely available to laboratories for incorporation into their quality procedures. However, owing to the relative long-term instability of OPs compared with OCs, there are many production and storage problems which remain to be addressed. It may be several more years before CRMs, other than for OCs, become commercially available.

A laboratory which determines OCs should include the analysis of an appropriate CRM within its quality system. However, CRMs are expensive and it is rarely practical to use them for quality control on a batch-by-batch basis. Each laboratory must assess the frequency with which it needs to analyse CRMs. The decision will depend on a number of factors, including the total number of samples analysed in the laboratory and the significance of any particular set of samples. Monthly analysis of CRMs is appropriate for most laboratories and possibly when a new batch of internal laboratory reference material is introduced into the system.

Internal laboratory reference materials (such as previously analysed samples of known residue content) have some advantages over spiked recoveries as, like CRMs, they additionally provide a check on the extraction efficiency of the method for the determination of incurred residues. Although these are not traceable to international standards, they do demonstrate an ongoing commitment to quality control within the laboratory. These reference materials should be analysed more frequently than CRMs. If the laboratory has plentiful supplies of material, then they can be incorporated into every batch of samples analysed. However, as the material is unlikely to contain all the compounds being sought, this will not negate the need to also employ spiked recoveries in the AQC programme.

### *6.3. Proficiency testing schemes*

In proficiency testing schemes, participating laboratories receive samples containing one or more compounds of unknown concentration at regular intervals throughout the year. The laboratory analyses the samples and returns its results to the organizing body, which calculates the "true" results (usually based on the mean results of all participating laboratories or the mean of several selected "expert" laboratories). The participant is then awarded a score according to its performance. To achieve a good score in a proficiency testing round can be considered one of the best tests of an analytical laboratory and is good evidence that the quality procedures which have been put into place are actually working in practice. However, proficiency testing should not be regarded as a substitute for ongoing batch-by-batch AQC. Accreditation bodies are increasingly making participation in appropriate proficiency testing schemes a requirement for accreditation.

To the authors' knowledge, there are three such proficiency testing schemes available for pesticide residues in foods:

(1) Food Analysis Performance Assessment Scheme (FAPAS), which is organized by the CSL Food Science Laboratory, Norwich, UK [4].
(2) World Health Organization GEMS/FOOD EURO, which is also organized by the FAPAS Secretariat.
(3) CHEK, which is organized by the In-

spectorate for Health Protection, Groningen, Netherlands.

## 7. Confirmation of pesticide residues

Residues should not be reported on the basis of a single GC or HPLC trace, unless MS detection was employed or the sample was of known treatment history. *Minimum* confirmation would be a second chromatographic run on a column of different polarity and/or using a detector based on a completely different mode of action. However, MS confirmation is desirable in all cases and is essential where "unusual" residues appear to be present or action limits, such as maximum residue levels (MRLs) may possibly have been exceeded.

MS confirmation must not be based solely on the judgement of the operator, however experienced and expert the individual. Professional opinion cannot be accredited. Therefore, it is essential that a set of MS criteria are designed which must be met for a residue to be confirmed. Such criteria have been published by the WPPR [3].

## 8. Costs/benefits of accreditation

There is no doubt that it can take a long time to develop a comprehensive quality system which will be acceptable to the accreditation body. Once accredited, there is an on-going time commitment to maintain records and the cost of additional AQC samples which may not otherwise have been analysed. However, these items should not constitute an unacceptable burden of work or cost.

The benefits of accreditation are many and varied. By far the most important advantage is the credibility and confidence which can be attached to the results generated by the accredited laboratory. This is of great importance both to the staff of the laboratory, who gain a sense of pride and achievement in their work, and also to clients of the laboratory, who are able to have confidence in the results provided and take decisions accordingly. Many customers, both commercial companies and also government departments, are beginning to demand that a laboratory is accredited before placing work with them. There may also be efficiency savings in an accredited laboratory as there are likely to be fewer occasions on which work has to be repeated.

In summary, in the authors' opinion, the benefits of accreditation far outweigh the costs.

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