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manual of food quality control

14. quality assurance in the food control chemical laboratory

FOOD AND AGRICULTURE ORGANIZATION
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FOREWORD

National authorities have the responsibility to protect the health and welfare of their citizens. Carrying out this responsibility requires the development and implementation of a national programme which, ensures the safety and quality of food products, promotes the development of trade in food products, and protects the interest of the fair and honest food producer, processor and marketer against dishonest and unfair competition.

This manual is a practical Handbook on the establishment of a Quality Assurance (QA) Programme for a Food Control Laboratory. Its ultimate aim is to assist laboratory management in establishing an effective operating system that will ensure that the chemical laboratory produces accurate and reliable analytical results so necessary to support regulatory actions. A similar QA manual for the microbiology laboratory has been published in 1991 as FAO Food and Nutrition Paper 14/12.

Although, this manual is designed primarily for chemistry laboratory management and analytical staff, regulatory authorities, or any other concerned persons can gain useful information and insights into problems involved in establishing and operating a quality assurance programme in a Food Control Chemistry Laboratory.

FAO wishes to acknowledge the efforts of Dr. David McWeeny, FAO Consultant, who prepared the text for this manual. The contribution of Mr. John A. Day, Head of Environmental Services Division, Laboratory of the Government Chemist, UK is also gratefully acknowledged.

This publication is available to persons and organizations. Comments and suggestions for possible future editions of this publication should be sent to:

The Chief
Food Quality and Standards Service
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SPECIAL NOTE

The Laboratory procedures described in this Manual are designed to be carried out by properly trained personnel in a suitably equipped laboratory. In common with many such procedures, they may involve hazardous materials.

For the correct and safe execution of these procedures, it is essential that laboratory personnel follow standard safety precautions.

While the greatest care has been exercised in the preparation of this information, FAO expressly disclaims any liability to users of these procedures for consequential damages of any kind arising out of or connected with their use.

The quality assurance procedures are also not to be regarded as official because of their inclusion in this Manual.

QUALITY ASSURANCE IN THE FOOD CONTROL CHEMICAL LABORATORY

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GLOSSARY OF TERMS*

Accuracy (see also "Trueness" and "True Value").

The closeness of agreement between a test result and the accepted reference value.

NOTE - The term accuracy, when applied to a set of test results, describes a combination of random components and a common systematic error or bias component.

Assigned Value

The value to be used as the true value by the proficiency test coordinator in the statistical treatment of results. It is the best available estimate of the true value of the analyte in the matrix.

Bias

The difference between the expectation of the test results and an accepted reference value.

NOTE - Bias is a systematic error as contrasted to random error. There may be one or more systematic error components contributing to the bias. A larger systematic difference from the accepted reference value is reflected by a larger bias value.

Bias (Laboratory bias)

The difference between the expectation of the test results from a particular laboratory and an accepted reference value.

Bias (Method bias)

The difference between the expectation of test results obtained from all laboratories using that method and an accepted reference value.

NOTE - One example of this in operation would be where a method purporting to measure the sulphur content of a compound consistently fails to extract all the sulphur, thus giving a negative bias to the measurement method. The bias of the measurement method is measured by the displacement of the average of results from a large number of different laboratories all using the same method. The bias of a measurement method may be different at different analyte concentrations.

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* based on current ISO definitions

Certified Reference Material (CRM)

A reference material, accompanied by a certificate, one or more of whose property values are certified by a procedure which establishes traceability to an accurate realisation of the unit in which the property values are expressed, and for which each certified value is accompanied by an uncertainty at a stated level of confidence.

Limit of Detection

The smallest concentration of analyte which a given analytical method can detect with a specified degree of certainty.

Limit of Determination

The smallest concentration of analyte which a given analytical method can quantify with a specified degree of certainty.

Precision

The closeness of agreement between independent test results obtained under prescribed conditions.

NOTES

1. Precision depends only on the distribution of random errors and does not relate to the accepted reference value.

2. The measure of precision is usually expressed in terms of imprecision and computed as a standard deviation of the test results. Higher imprecision is reflected by a larger standard deviation.

3. "Independent test results" means results obtained in a manner not influenced by any previous result on the same or similar material.

Proficiency Testing Scheme

Methods of checking laboratory testing performance by means of interlaboratory tests. This includes comparison of a laboratory's results at intervals with those of other laboratories, with the main object being the establishment of trueness.

Quality Assurance Programme/Quality System

The sum total of a laboratory's activities aimed at achieving the required standard of analysis. While Quality Control and proficiency testing are very important components of quality assurance, it must also include staff training, administrative procedures, management structure, auditing, etc.

Quality Control

The set of procedures undertaken by the laboratory for continuous monitoring of operations and results in order to decide whether the results are sufficiently accurate and precise to be released; QC primarily monitors the batchwise accuracy of results on quality control materials, and precision on independent replicate analysis of test materials.

Reference Material (see also CRM)

A material or substance one or more of whose properties values are sufficiently homogeneous and well established to be used or the calibration of an apparatus, the assessment of a measurement method, or for assigning values to test material.

Target Value for Standard Deviation

A numerical value for the standard deviation of a measurement result, which has been designated as a goal for measurement quality.

Testing Laboratory

A laboratory that measures, examines, tests, calibrates or otherwise determines the characteristics or performance of materials or products.

True Value

The actual concentration of the analyte in the matrix.

Trueness (see also "Accuracy" and "True Value")

The closeness of agreement between the average value obtained from a large series of test results and an accepted reference value

NOTE - The measure of trueness is usually expressed in terms of bias.

QUALITY ASSURANCE IN THE FOOD CONTROL CHEMICAL LABORATORY

1. OBJECTIVES, CONCEPTS AND RESPONSIBILITIES

1.1 General Principles

The food control laboratory exists to provide information about the composition of food.

The quality of that information is judged by whether it is of appropriate standard, is available on time and is produced at an acceptable cost.

The criterion for "an appropriate standard" is whether or not the data is fit to be used for the intended purpose.

Quality Assurance is the system which provides confidence that:

- *the standard is adequate*
- *failures to meet that standard will be detected*
- *the causes of the failure can be identified and corrected.*

1.2 Laboratory Objectives

The objectives of the laboratory should be clearly defined and expressed as simply as possible. Their clear definition is of fundamental importance because it is on these that all laboratory activities are based. The Laboratory Director should define the objectives after taking such advice as he sees fit and such instructions as he may have received from those directing his activities. The statement should include reference to the quality of results, their timeliness and their cost-effectiveness. The objectives may be divided into a number of statements. All aspects crucial to the laboratory operation should be included but detail should be avoided.

The principal objective of a laboratory is to produce reliable results, which is therefore the activity to which the fullest attention must be paid. Indeed, a laboratory producing unreliable results to a significant extent is unlikely to be acceptable as part of any government machinery. The assurance of the quality of those results is not an extra burden or an extra activity that can be taken or left. It is one of the fundamental management tools for the director and his staff, to ensure that they achieve whatever they set out to do. The general objective of the laboratory might be defined as producing analytical data of adequate accuracy and reliability within an acceptable time and at an acceptable cost.

The objectives in relation to quality must be as realistic as any other objective. The quality objective might be considered as being as sure as possible that

approximately the right answer had been obtained. This statement requires examination. What is "as sure as possible"? It is being so sure that if subsequently shown to be erroneous, the reasons would not reflect on the integrity, probity or technical competence of laboratory staff.

And what is meant by "approximately"? It means obtaining a result which is good enough for the purpose for which it will be used. If a sample is seriously deficient in a particular analyte, the precise amount of the deficiency probably will not be of great practical importance in, for example, court proceedings or refusal of a consignment. As the amount of analyte approaches the legal limit, the accuracy of the analysis becomes more crucial until a point is reached when the result is closer to the limit than the precision of the method. Thus both accuracy and precision (see Glossary of Terms) have to be greater for marginal samples than those for which results are far from any standard or limit. With non-permitted substances, the quality requirement is somewhat different - the limit of detection and the degree of confidence attaching to presence/absence at that level will need to be considered.

In order to produce quality information it is important that the laboratory knows, at least in general terms, why the information is needed. Equally the user of the information may need to consider carefully before using analytical data outside its original context. The need for a common understanding of "quality" and its implications - and for a dialogue on the subject between the laboratory and its "customers" will emerge repeatedly throughout this Manual. For convenience, the user of the laboratory data will be referred to in this Manual as the "customer" - it being understood that although this normally implies someone outside the laboratory organisation, occasionally it may be someone within the laboratory - or even the analysts themselves.

1.3 Quality and Quality Assurance - Some Concepts

Analytical "quality" and "quality assurance" seem simple and self-explanatory concepts, but in fact the terms are frequently misunderstood. Quality assurance is often confused with another term, "quality control" and used as though the two are interchangeable when in fact - although related, they are quite distinct. A widely used definition of **Quality Assurance** is:

- all those planned and systematic actions necessary to provide adequate confidence that a product will satisfy given requirements for quality (ref 1.1). **Quality Control** is just one of the organised procedures within this systematic approach; one definition is "the operational techniques and activities that are used to fulfill the requirements for quality". Quality assurance aims to ensure that the purpose of quality control is achieved. For example, using an analytical balance, accurate to $\pm$ 0.1 mg to weigh out a sample for assay is a matter of "quality control", while the routine calibration of that same analytical balance with standard weights on a predetermined schedule is "quality assurance".

This in turn raises questions about the meaning of the term "quality". Sometimes it is mistaken as meaning "scientific excellence", when in fact its use in the analytical context is much simpler. When asking whether data quality is satisfactory the underlying question is whether or not the quality of the data is suitable for the purpose to which it is to be used. Excellence as such is irrelevant; it is a waste of effort and, most likely, an irresponsible use of resources to attempt to provide something better than the "customer" requires. The important concept is that someone has to specify the quality requirement; the concept of "fitness for purpose" (ref 1.2) is the basis on which the word "quality" is used in this Manual.

The **quality assurance programme** (sometimes described as the **quality system**) is the sum total of all the things which contribute to ensuring the capability of the organisation to deliver information which is fit to be used for the purpose for which it is intended. This hierarchy of components of the quality assurance programme - with quality assurance providing the evidence for confidence that quality control is working successfully - will be apparent throughout this Manual. With its emphasis on quality assurance, the Manual concentrates on those features which demonstrate that all the things which should have been done, were actually done and were done properly.

Inevitably this assurance that quality control actions were properly carried out, relies heavily upon their documentation - without it there is little opportunity to verify anything. The Manual will also consider quality control insofar as this is necessary to identify and illustrate the detailed requirements of quality assurance, the documentation on which it relies, and the way in which this is specified in the laboratory's Quality Manual. The latter is the key documentary component of Quality Assurance in a laboratory; its content is considered in Chapter 2, (Section 2.4).

1.4 Quality Assurance - the Need

If the laboratory is equipped for its job, if the staff are qualified and well motivated, if they are using well-documented methods and are familiar with the instrumentation needed, presumably the results should be correct. If there is no reason to think they are wrong, why go to the trouble and expense of drawing up, implementing and monitoring a quality assurance scheme? Unfortunately there are two compelling reasons why that approach cannot be justified. One is a matter of scientific/ technical fact, the other relates to enforcement/trade/consumer protection.

Firstly, over the last decade it has become increasingly apparent that the reliability of food analyses is not what it was thought to be. Evidence to that effect comes from several sources, e.g. the various Quality Assurance studies carried out by WHO in relation to laboratories producing data for the Global Environmental Monitoring Scheme for Food (GEMS/Food). The most extensive body of data probably resides in the findings of the Food Analysis Performance Assessment Scheme in UK (ref 1.3). Over 200 laboratories participate in this Scheme, which assesses the results from laboratories conducting one or more of a representative range of eight areas of food analysis. In 1992 approximately 20% of the results submitted were outside the ranges which could reasonably be expected. Many of the suspect results may have

come from smaller, less experienced laboratories with relatively low throughput of analyses and, overall, the proportion of incorrect results may be well below 20%, but the finding is certainly sufficient to challenge the assumption that in the absence of any unusual factors, results can be assumed to be correct.

Secondly, demonstrable confidence in analytical data is demanded by the rapid increase in national legislation on food control and consumer protection and by international trading agreements which are based on mutual recognition of laboratory results. That mutual recognition has to be based on some tangible evidence.

A properly constituted and properly operated Quality Assurance (QA) programme is capable of providing detailed tangible evidence about the confidence to be placed in data of a particular type from a particular laboratory - it can even provide the basis for verifying individual analytical results. An enforcement laboratory needs a QA programme because it may be faced with a challenge about the validity of its data by another laboratory with its own QA programme! Increasingly, too, major food companies will deal only with suppliers who use laboratories with quality assurance schemes which allow an external assessment of the confidence which can be placed in its results.

An effective QA programme that is working properly has several operational advantages. First, it provides a tracking record to ensure sample integrity, with documentation to verify that laboratory instruments are functioning properly and that laboratory data were generated according to approved written protocols. Such documentation is especially important in regulatory laboratories where analytical findings must withstand the scrutiny of legal proceedings.

A second advantage is a saving of analytical time and costs. Although the QA programme may initially seem to reduce a laboratory's productivity, it may actually save analytical time and costs over a long period, since analyses will tend to be done correctly the first time.

A third advantage of a QA programme is its role in identifying training needs of analysts. These needs are not restricted to new employees; they apply also to existing employees whose performance may be deficient or needs updating.

A fourth advantage is an increase in analyst confidence derived from knowing that results are reliable. This increased confidence, in turn, can lead to improved morale and performance.

Other advantages of a QA programme include:

> Ensuring errors are identified and minimised or eliminated. It is impossible to eliminate all errors but it is possible to ensure that very, very few serious errors are made without discovery before the results are transmitted outside the laboratory.

- Ensuring forensic credibility. There is usually a strong legal tradition about the test applied to evidence in court. The criteria for the development of scientifically valid evidence are just as rigid but this does not necessarily mean that the evidence will comply with court rules or be understandable to a court. For example, if the legal test is "beyond reasonable doubt", the court may have difficulty in equating this to statistical information about probability.

- Ensuring, in the event of enquiry or dispute, that management has confidence in the results produced. This confidence derives from the body of evidence that gradually accumulates about the performance of the laboratory in the range of analyses it carries out.

- Ensuring, in the event of enquiry, dispute or error that records are available to resolve the issue. Records should be kept for a considerable length of time. Five or six years is often chosen.

- Providing a review of deficiencies, errors and complaints so that remedial action can be systematic and lead to intrinsic improvement.

- Ensuring resource utilisation is optimal. This is often a slow process, but as more information accumulates about analytical performance within the laboratory, it becomes easier to evaluate the effectiveness with which the resources of the laboratory are being used. For example, it is easier to ensure that reagents and standard solutions are available and still within a "use-by" date.

- Providing results of sufficient certainty for use in databases for the purposes of food control, public health, nutrition and other food-related local, national or international policies. These databases form an extremely valuable resource for monitoring food products over a period of time. This leads to identification of changes in products over time and the ability to compare analytical results very easily. If the information in databases is not reliable, false conclusions may very easily be drawn.

1.5 Major Features of a Quality Assurance Programme

The aim of a QA programme is to provide confidence in the Laboratory's output. The most important feature of it will be the visibility and realism of management's commitment to it, and demonstrated by staff at every level. Ultimately much of this will be expressed in two ways. Firstly it will result in documentation of policies and procedures which specify what is to be done, how and by whom, and include mechanisms which ensure that any omissions in these respects are rapidly apparent. Much of this documentation will appear in the Laboratory's Quality Manual (which is discussed in Chapter 2, Section 2.4) and its satellite documents. Secondly it will necessitate appointment of a Quality Manager and the formation of a Quality

Assurance Unit which will co-ordinate the development, implementation and maintenance of the Quality Manual and will be responsible for conducting reviews and audits which monitor its effectiveness; its terms of reference must include clear statements about its responsibilities vis-a-vis those of analysts and their line management.

1.5.1 Responsibilities of Management

Initially management must decide on the level of quality which the QA programme will aim to ensure; it will do so after discussion with customers and in the light of other constraints which may exist. On an on-going basis it is part of the management function to ensure the quality of results leaving the laboratory. It is a function that should be carried out to the extent necessary, no more and no less, and should be a fully integrated part of managerial duties at all levels on a daily basis. It is important to note that QA involves not only getting the right answer but having documentary evidence to prove the right answer has been obtained. Introduction of written QA procedures into a laboratory for the first time may require considerable changes in attitudes and working practices. When introduced in the right way, QA leads to improved morale as staff gain confidence in their results and pleasure at being able to prove the veracity of those results. Quality assurance focuses attention on relevant aspects of daily activities and training needs and helps staff to develop their skills and further their careers.

It is the responsibility of management to decide upon the scope and priority of the QA programme for a particular laboratory, basing its decision on estimation of a balance of costs and benefits which takes account of parameters which are not easily quantified (e.g. customer confidence, laboratory credibility and reputation). Unfortunately, the quality assurance component of a laboratory's operations often receives inadequate attention, or may not be properly balanced. For instance, it may be so detailed that practically every analytical function is covered. Although seemingly attractive and advantageous, this type of programme can be overwhelming and frustrating for laboratory personnel. Consequently, what begins as an admirable undertaking, may end in discouragement and the absence of any meaningful quality assurance programme whatsoever. At the other extreme is a programme so abbreviated that it really has little, if any, meaning. When a particular programme has been selected, management must be clearly seen giving full support to its concept, implementation and maintenance. If management is perceived as not being supportive of the programme, the staff too can be expected to make something less than a full commitment.

Once a QA programme is included as a part of daily operations, management must commit resources to reinforce its support and establish a Quality Assurance Unit to monitor its implementation. Management must project a positive image for the QA programme. The programme should not be viewed as threatening, confrontational, or as something extra that must be done. Instead, it should be projected as a way to improve employee performance and as a mechanism to document and reward exceptional work.

Many analysts started their working lives in laboratories where quality assurance as we now know it, was not practised. There are still laboratories working in this way, even though they are diminishing in number. Thus at some point, an experienced analyst may have to change his style of working and to develop a positive attitude to quality assurance. There are many obstacles to this.

The analyst may feel all his past work is being called into question because it was not undertaken under the new system. Also quality assurance is time-consuming initially and the analyst will probably feel sure he already fills his day and that nothing he does is inessential. Thus, the new way of working may seem both unnecessary and unachievable. Management can easily compound these negative feelings by poor personnel skills, lack of commitment and understanding.

The concept of quality assurance may have to be promoted both outside and inside the laboratory. In the first place, it may have to be promoted to those with a part to play in the decisions that lead to funding for the laboratory. There can be difficulty in persuading administrators and legislators, who may have limited technical knowledge, to accept the need for what is apparently an additional overhead. It may be argued that the laboratory appeared to operate satisfactorily for a long time on the old basis and further costs cannot be justified. The changing circumstances must then be explained. Decision makers have to address changing concepts in their own use of quality assurance. In their own work increasing automation and computerisation is moving attention from the manipulative skills required for often slow and tedious methodology to the administrative and management skills required to assure the quality of large amounts of data. They may need to be shown that there is a change in the internal laboratory environment too, from ad hoc samples to sampling programmes, from single or very few samples to optimum batches of the same type of product, from analytical professionalism and (at times) virtuosity to a quality-assured management approach. In both situations, these developments bring benefits in cost-effectiveness and best use of resources, but neither can operate without a quality assurance component.

Management determines the nature and size of the Quality Assurance Unit which will be at the hub of the QA programme. In large multi-discipline laboratories the Quality Assurance Unit may consist of two or more persons whose sole responsibility is to monitor the effectiveness of the QA programme. In smaller laboratories, instead, an analyst may be assigned to spend part of his or her time ensuring laboratory-wide compliance with an approved QA programme.

The frequency of formal audits to ensure compliance with the approved QA programme is also determined by management. Some managers may elect to have quarterly, or even monthly, inspections. Others may decide to have abbreviated quarterly inspections and a comprehensive annual inspection. Regardless of the frequency of inspections, management must regularly review the findings of the Quality Assurance Unit and act on its recommendations. Management may decide to reward employees who have excelled in compliance with the QA programme. For example, the concept of "merit pay" is increasingly used to rate an employee's performance. One consideration for merit pay could be quality assurance, thus enabling management to motivate employees to excel at the approved QA programme. Where there is non-

compliance with the programme, management may need to use disciplinary action to ensure compliance.

In addition to responding to the recommendations of the Quality Assurance Unit, management should periodically review both the quality assurance policy and the programme itself. Although the policy and its provisions should be strictly enforced, there should be enough flexibility to allow for the reasonable deviations that may arise when provisions of the original QA programme are too specific or are not specific enough. If the deviations become too specific, the details of the programme itself may need to be modified to incorporate them. Management should review the quality assurance policy and programme regularly and make appropriate modifications.

In summary, Quality Assurance does not become an integral part of an organisation's activities without commitment and effort by management. There is a great deal of work to be done initially in writing a Quality Manual, generating quality control data and commissioning the quality assurance programme system. Management support at this stage is essential and can be demonstrated by encouragement, counselling and provision of adequate resources. Quality Assurance is brought into disrepute if the manager is prepared to lapse into an old way of working to achieve some short-term objective, such as reporting a sample very quickly because there is pressure to do so. That is typically the time when mistakes are made. Management can also express their commitment to the programme very effectively by insisting on adherence to the schedule of audits and reviews and then taking a keen interest in any follow up action. The Audits and the actions resulting from them provide the built-in mechanism for change in the Quality Assurance programme and thus potentially determine the on-going effectiveness of the programme.

1.5.2 Responsibilities of the Quality Assurance Unit

The first step in the formation of a Quality Assurance Unit will generally be to obtain either budgetary approval for the appointment of a Quality Assurance Officer (or "QA Manager") and any staff he may need, or agreement to divert existing personnel and other resources into this activity. Ideally the QA Manager should have formal qualifications in quality assurance but this is not often possible. More likely, an analyst will be appointed to this position and will have to self-train, attend courses and so on. This person needs to be a competent analyst of some seniority, and must command the respect of colleagues for his/her analytical skills. Above all, the QA Manager must be keen to understand the principles of QA and to apply them correctly in a way which is realistic and reflects the Laboratory's objectives. The QA programme exists to serve the Laboratory - the reverse situation must not be allowed to develop.

The QA Manager needs auditors and these will generally be appointed from among the analysts. Teams of two are sufficient in a small laboratory, auditing any section (including administration) but not their own. In a larger laboratory, the QA Manager may need his own staff but the QA Unit does not usually become large. One QA person for every ten to twenty analysts is probably adequate. The wider the range of analyses undertaken and the lower the overall level of experience, the more will quality assurance be required.

The Quality Assurance Unit is responsible for formulating the written quality assurance plan or manual (the "Quality Manual") and monitoring adherence to the programme by the laboratory staff. The Quality Assurance Unit acts as a liaison between management, who have committed resources to ensure the success of the project, and the laboratory staff, who are directly responsible for actual implementation of the programme. The Quality Assurance Unit is dependant upon the staff, specifically the laboratory bench scientist and the team leader or section head, to provide technical expertise in formulating the written quality assurance plan.

In addition to scheduling and conducting reviews, quality assurance personnel should make recommendations to management regarding the results of those reviews; they should also recommend quality assurance policy to management and assist in its formulation, identify staff training needs and provide guidelines for adhering to all aspects of the QA programme.

Keen attention to reviews and prompt follow-up action is one way in which management provides support to the quality assurance function. To allow this, the Quality Assurance Manager and, where appropriate, members of his unit must have direct access to the Laboratory Director or a delegated senior manager.

There should be review meetings once or twice a year. At this time, in consultation with the QA Manager and senior analysts, management may decide on any changes in policy and programme. Need for change will become apparent as a result of quality audits.

1.5.3 Responsibilities of the Analyst

The Analyst plays an essential role in the operation of the QA programme. The trained bench scientist has front-line responsibility for the quality of the data and related laboratory activities, and is the first with an opportunity to detect malfunctions and abnormal fluctuations of the analytical system.

Both management and the Quality Assurance Unit expect the analytical staff to provide technical input and advice in the formulation of the QA programme. Selected analysts may be asked to write a portion of the quality assurance plan, subject to review and approval by the Quality Assurance Unit and management. Being included in formulating the quality assurance plan can be a motivating factor, giving personnel a feeling of having a creative influence on the QA programme.

The laboratory staff are responsible for adherence to the provisions of the approved plan. The success or failure of the plan ultimately depends on the performance of the analyst. The bench scientist, who actually constitutes the first level of "management" of any QA programme, is responsible for doing the work properly, documenting it, and having the work reviewed to ensure that it meets acceptable standards. In specialised areas of work it may be appropriate to give the section head specific responsibilities in relation to the development and operation of the QA programme. This helps to ensure that the QA "culture" is reinforced at a critical point in the management chain.

Each of the three groups (analytical staff, Quality Assurance Unit, and management) must provide the appropriate input to ensure the success of a QA programme. The analytical staff provides the technical expertise needed to prepare the quality assurance plan and is responsible for the daily adherence to it. The Quality Assurance Unit monitors the staff's adherence to the plan and, based on its reviews, makes recommendations to management. Management, in turn, reviews the reports of the quality assurance unit and takes decisions on its recommendations.

1.6 Documentation: QA Policy and the QA Programme

The QA programme and its documentation will reflect the "quality" policy for the laboratory. Management must take a view on what represents the appropriate cost: benefit situation for their particular laboratory and develop a QA programme which matches that view. The QA programme must serve the laboratory's objectives, not dominate them. Accordingly, the policy on documentation of procedures must be that there have to be clear instructions about when action is to be taken and who must take it but the amount of detail about precisely what is to be done, how it is to be done must be carefully judged; it must provide the agreed degree of confidence in the quality of the results, without demanding a counter-productive amount of paperwork. The procedural "loops" which demonstrate that appropriate QA steps have been taken, should be designed so that they call for action automatically when - but only when - some specific intervention is needed. This helps to ensure that section leaders and managers become accustomed to knowing that QA documents come to them only for some specific action - not simply for them to "note".

Each chapter of this Manual concludes with some suggestions on topics which should be considered for incorporation in the QA programme documentation; some examples of relevant documentation are given in the corresponding Appendices. This Chapter has identified the need for documentation giving:-

Statement of Laboratory Objectives (see Appendix 1.1)

Quality Assurance Manager, Terms of Reference

1.7 References

1.1 International Standards Organisation, Standards 9000 to 9004.

1.2 Juran, J., Planning for Quality, 1988. Collier Macmillan Ltd., Purnell Distribution Centre, Bristol, U.K. ISBN 0029166810.

1.3 FAPAS Secretariat, MAFF Food Science Laboratory, Norwich Research Park, Colney, NORWICH NR4 7UQ, England, UK.

1.8 Recommended Reading

1. Garfield, F.M., 1991. Quality Assurance Principles for Analytical Laboratories: AOAC International, Arlington, Virginia 22209, USA. ISBN 0-935584-46-3.

2. FAO Food and Nutrition Paper 14/12: Manual of Food Quality Control 12. Quality Assurance in the Food Control Microbiological Laboratory (1991), Food and Agriculture Organisation of the United Nations, Rome, Italy. ISBN 92-5-103053-7.

3. ISO Guide 25 (1990). General Requirements for the Competence of Calibration and Testing Laboratories.

4. Weatherwax, J., and Martin, P.G., 1986. Manuals of Food Quality Control, 1. The Food Control Laboratory. Food and Agriculture Organisation of the United Nations, Rome, Italy.

5. Quality Assurance Principles for Chemical Food Laboratories (1990). Nordic Committee on Food Analysis c/o Technical Research Centre of Finland, Food Research Laboratory PB 203, SF-02151 ESP00, Finland.

6. NAMAS Accreditation Standard: General Criteria of Competence for Calibration and Testing Laboratories M 10: 1989, NAMAS Executive, National Physical Laboratory, Teddington, Middlesex TW11 0LW, England, U.K.

2. THE QUALITY ASSURANCE PROGRAMME

2.1 General Principles

The QA programme will cover all aspects of the laboratory's work which can affect the quality of its output.

There is no "universal" QA programme, suitable for all food control laboratories. The emphasis given to each aspect of QA will reflect the laboratory's work and objectives.

If staff are to be expected to implement a QA programme, they should be able to expect to be involved in devising it.

2.2 Definition and Scope

A Quality Assurance programme - sometimes referred to as "a quality system" - may be defined as a mechanism used to ensure that data generated by a laboratory is of the highest quality. As discussed earlier, "highest quality" does not necessarily have anything to do with analytical excellence, sophistication of technique, state-of-the-art equipment or superb accuracy, precision and detection limits; it means that the data are fully reliable, suitable for the intended purpose, presented on time and at an acceptable cost. The assurance that this quality has been achieved rests on confidence that all laboratory functions are being performed as intended - and that if needed, the documentation needed for reassessment is available.

The scope of the quality assurance system has to be developed in such a way that there is confidence that whenever data is reported

- the sample identity and integrity have not been compromised
- the analysis has been conducted by a member of staff who is competent for the task
- the equipment and methods used are appropriate and are working properly
- the laboratory can demonstrate its current capability to produce valid data of that type.

The format adopted in meeting these underlying requirements can - and should - vary from laboratory to laboratory. Each laboratory (or group of laboratories) meets different requirements, operates within a different organisational environment, experiences different constraints and should have a QA programme which takes account of these factors. There is no universally suitable QA programme, but in practice many of them have certain central factors in common, e.g.,

- the use of validated methods

- the use of written standard operating procedures (SOP's) in the laboratory.

- calibration and traceability of measurement (including use of certified reference materials where available)

- external assessment of performance

Where facilities exist for accreditation of laboratories for particular types of work it is usual to find requirements for these three features within the scope of the accreditation process along with the requirements for topics which may include organisation and management, laboratory accommodation and environment, equipment maintenance, handling of test materials, test methods and quality control procedures (and method performance characteristics), staff training and performance, security, records and reports, sub-contracting of work, outside support services, handling of complaints, quality audit and system review. These topics are discussed later in this Manual and should be considered for inclusion in the QA Manual (Section 2.4) even if accreditation is not part of the immediate plan.

2.3 Preparation

The QA programme is therefore concerned with much more than the analysis process - central though the importance of this may be. It is concerned with everything that goes on in the laboratory which may affect the "quality" of the data as judged by the criteria identified in Chapter 1. The corollary of this is that quality assurance involves everyone working at the laboratory; if they have a part to play in the function of the laboratory, then the quality assurance programme is concerned with what they do - and whether they do it properly. Each member of staff must

- be clear about what they are expected to do,
- know how to do it, and
- be able to show that they have done it properly.

Documentation, therefore, is one of the major features of a QA programme - each member of staff must be able to show that when doing a particular job, they had been given whatever training was needed, the equipment and materials used were in good condition, that the method used was appropriate to the purpose and was producing reliable results and that standard operating procedures were followed. The initial production of the documentation for all this can be demanding - particularly on skilled resources - but it is important to involve each member of staff in some way in the preparation of the documentation affecting their own job. It is essential to any QA programme that its documentation is used fully and properly; involving each member of staff in its preparation and listening to what they say about how to set it out so as to minimise the additional work for them is an important ingredient in ensuring that it is introduced smoothly and that afterwards it is used convincingly and effectively.

These considerations apply in at least general terms whether one is considering the analysis of samples, the purchasing of supplies or maintaining the laboratory facilities. The QA programme affects all the staff - and therefore if it is to be a success, each member of staff has

to feel involved in devising the programme, has to understand why it has been introduced and has to be committed to its success.

An effective QA programme will be one that is expressed in a straightforward way. The programme should be clear, concise and uncomplicated, not tedious or unduly long and cluttered with unnecessary details or trivia. A complex programme is likely to generate analyst resentment, which may lead to less than a full commitment.

An effective QA programme must be practical from the standpoint of the analytical time and costs involved. If a disproportionate amount of the analyst's time is needed to maintain it, the QA programme is not properly balanced. An effective programme should result in a long-term saving of analyst time and costs because the need for repeat analyses will be reduced.

Garfield (ref 2.1) proposes that a somewhat simpler formulation of a QA programme should contain three essential components:

a. Prevention, which requires an orderly programme of planning and positive actions before or during analyses to ensure that all analytical systems are performing appropriately (e.g. calibration and maintenance of instruments, use of reference materials and training).

b. Assessment, a form of control that includes periodic checks on analyst performance (e.g. analysis of check samples and validation of methodology).

c. Correction, an action taken to determine cause(s) of quality defects and to re-establish proper functioning of analytical operations (e.g. trouble-shooting to correct malfunctioning equipment, re-evaluation of methodology and retraining).

The final form of the QA programme should be both a scientific and a management decision. The day-to-day analytical operations of the food control laboratory should determine which features are needed in the programme. Management must then prioritise these features and determine the extent to which resources will be allocated to the programme.

2.4 The Quality Assurance Manual

Each laboratory with a QA programme should have a manual that documents the operations of the laboratory. The U.S. Environmental Protection Agency (ref 2.2) defines the QA manual as *a written document that describes the policies, organization, objectives, functional activities, and specific quality assurance activities designed to achieve the objectives of quality desired by the laboratory.* A typical Manual might consist of the following:

a. Title page with signatures of all approving officials and date of issue (Appendix 2.2)

b. Table of contents

c. Organizational structure and exactly where the laboratory fits into this structure

d. Objectives of the quality assurance programme

e. Essential elements of the quality assurance programme (see Section 2.7 and Appendix 2.1).

f. Documentation forms

g. Performance and frequency of audit

h. Corrective and follow-up action

A statement of quality assurance policy, both general and specific, is needed in the QA manual. For example, the U.S. Food and Drug Administration's Bureau of Foods Quality Assurance Manual (ref 2.3) includes a general policy statement: *"The Bureau of Foods Quality Assurance Programme is intended to serve in maintaining the highest level of quality and integrity of the Bureau data. The policies, procedures and instructions contained in this manual establish a quality assurance program uniformly applicable to all Bureau laboratory facilities. This manual covers non-clinical laboratory studies, other laboratory studies and regulatory samples. All laboratory personnel who are associated with the supervision or conduct of laboratory work are responsible for following the instructions in this manual".*

In addition to this general policy statement, specific policy statements may appear throughout the manual, for example, a definition of the responsibilities of various organizational levels in implementing the programme, a list of laboratories (regardless of location) to which the QA programme applies, reference(s) to detailed organisational procedures and laboratory methodology (which may be incorporated in "satellite" manuals, each covering the activities which are specific to a particular section within the laboratory), ownership of or property rights to laboratory data, and any exceptions to policy statements.

An example of some of the elements which could be included in a QA manual are given at Appendix 2.1.

2.5 Implementation

Actual implementation of the QA programme is a co-operative effort involving management, members of the QA Unit, section leaders and analysts. Management decides the amount of resources to be allocated to the QA programme. This decision determines the nature and size of the QA Unit. In formulating the QA programme, this Unit receives technical input from the analysts. Once formulated by the QA Unit and approved by management, the QA programme is ready for introduction. This may be done on a simultaneous laboratory-wide basis or in a step-by-step fashion, perhaps beginning with certain key functions (e.g. sample receipt) and proceeding on a Section by Section basis. An advantage of the latter is that the staff involved can have pre-introduction training which is conducted in relatively small groups and is specific to their work; it also ensures that resources in the QA Unit are available promptly for participation in resolving any operational problems which emerge. Both these considerations may be important in securing and retaining staff commitment to the programme. Equally, in

planning stepwise introduction of the programme, care must be taken to avoid situations where procedures introduced in the early stages have to be changed when later stages are implemented.

Thereafter, analysts are responsible for day-to-day maintenance of the programme. The QA Unit periodically monitors this adherence and makes its report and recommendations to management, which then decides on the action to be taken so as to achieve compliance with the programme.

2.6 Revision of the QA Manual

The QA Manual must be designed so that change is easily accommodated. It is essential that as organisational patterns evolve, work-loads shift and methodology develops, the QA Manual can react rapidly to these changes in the work of the Laboratory. The Manual must therefore incorporate procedures for its own prompt, orderly and effective revision. Normally the QA Manager is responsible for issuing revisions agreed by the Head of Laboratory to holders of the QA Manual. In order to ensure the QA Manuals are kept up-to-date, individually numbered copies of the Manual can be issued initially to each authorised holder and all revisions will be issued in the same way. (Appendix 2.3) To provide simple verification of amendments, the revisions can be identified sequentially and show the date of issue; the holder of the Manual can check by the number that he has not missed any revisions, insert the new pages, remove the old ones and return these to the QA Manager who can then check that each holder has implemented the revision.

2.7 Documentation Required for the QA Programme

Laboratory elements to be considered for inclusion in the QA Manual	Appendix 2.1
QA Manual cover and contents pages	Appendix 2.2
A revised page from QA Manual	Appendix 2.3
Statement of Quality Assurance policy	Section 2.4

2.8 References

2.1 Garfield, F.M., (1991), Quality Assurance Principles for Analytical Laboratories; AOAC International, Arlington, Virginia 22209, USA.

2.2 Guidelines and Specifications for Preparing Quality Assurance Project Plans (1980), US Environmental Protection Agency, Cincinnati, Ohio, USA.

2.3 Bureau of Foods Laboratory Quality Assurance Manual (1982): US Food and Drug Administration, Washington DC.

2.9 General Reading

1. The Quality Manual: Guidance for Preparation, M16 (1989). NAMAS Executive, National Physical Laboratory, Teddington, Middlesex, TW11 0LW, England, UK.

3. LABORATORY FACILITIES

3.1 General Principles

Facilities must allow the laboratory's work to proceed both effectively and safely.

Laboratory design should reflect the general features of the work programme anticipated in the long-term (10-20 years) rather than the specific pattern of current work.

3.2 Design of the Laboratory

Even though the final design of the laboratory is made by architects and engineers, the analytical staff should be involved in some of the decisions that will ultimately affect their working environment and conditions. This Chapter, then, presents several points for analysts to consider if, and when, they are asked to provide input into the design of their laboratory.

Weatherwax and Martin (ref 3.1) have provided a comprehensive discussion of the details involved in establishing the food control laboratory. The food control laboratory may have several functions: chemical analysis of foods for proximates, trace metals, additives, nutrients and toxicants and for some basic food microbiology as well. The discussion presented here will cover those details of concern in food chemical laboratories, insofar as they have a bearing on the quality assurance programme.

3.2.1 General Considerations

Laboratory layout should be devised with efficiency in mind. For example, the distances staff have to walk for the different steps of the analytical processes they undertake should be as short as possible, though bearing in mind that some procedures may have to be segregated from others for analytical and/or safety reasons.

There is often a 5 year period from the decision in principle to build a new laboratory to when it is accepted and operational. Also there will usually be an expectation that it will not require major alteration for a further 10 years. Given that the work load may change in this time span there are real disadvantages in designing a laboratory just to reflect the detail of the currently anticipated work load. Even within a given work volume, events may demand that the relative emphasis given to the different types of analyses may change. Additionally, advances in instrumentation and analytical methodology may alter the space and environmental requirements for a particular analysis. There is an argument for designing a laboratory in terms of "generic" activities and "specialised" activities.

Generic activities can be categorized as "wet chemistry" which will require extensive provision of fixed benches with water, power, sinks, fume cupboards, reagent shelves, glassware cleaning and storage, as compared to "instrument rooms" where less extensive servicing (though with additional piped gas supplies and perhaps stabilised power supplies) and flexible arrangements of movable tables/benches may be adequate.

Specialised rooms may be required for "clean air" work (e.g. on some environmental contaminants) or for work with substances which need to be handled with especial care either for safety or for cross-contamination reasons, e.g. radioactive materials and some particularly toxic substances or for storage and dispensing of standards of pure compounds which are being analysed at trace levels elsewhere in the laboratory. A specialised room for large-scale and/or dusty sample preparation activities, e.g. grinding, blending, mixing, stirring will be invaluable, particularly if work is envisaged on heterogenous analytes (e.g. aflatoxins in nuts or figs where primary samples of 30 kg are sometimes needed).

With this approach the important design parameters are those concerned with correctly identifying the needs for specialised activities and with estimating the relative needs for the generic activities of "wet chemistry", "instrument room" and - for that matter - "food microbiology" if, as is often the case, that is to be carried out in the same premises.

Offices are needed for management and for clerical staff. There must be toilet and washing facilities for all staff. Eating, drinking, and smoking are always discouraged, and should be prohibited, in the laboratory proper; it is the responsibility of management to provide an appropriate alternative area for these activities. A separate staff room, however small, deserves consideration since it not only provides a greater degree of safety to laboratory personnel but also helps to ensure sample integrity.

To provide for a prompt exit in the event of fire or other emergency, at least two entrances/exits must be provided for each laboratory whenever possible.

3.2.2 The Chemical Laboratory

From a quality assurance standpoint, the design features which are important are those which can lead to erroneous results or to "lost" work, leading to missed deadlines and cost over-runs. Erroneous results can arise from test materials becoming contaminated (e.g. by dust) or by cross-contamination from another sample or from a standard. Whilst good working practices will usually control most situations satisfactorily, a design which provides complete segregation of trace analyses from highly concentrated formulations and from pure substances used in preparing analytical standards is virtually essential: the segregation must apply to all facilities for washing/cleaning equipment, washing and storage of glassware, use of protective clothing and even transfer of notebooks and records.

Design features which avoid dust, whether from environmental sources or from other samples are highly desirable from the quality assurance standpoint. Dust contamination of test materials is essentially sporadic and uneven; as such it is likely it will often be missed by the normal quality control checks. Design should aim for dust avoidance by using glass-fronted reagent shelves, keeping work-tops clear of unnecessary "static" items, regular cleaning of work surfaces with absorbent cloths, floor and furniture designed so that they can be cleaned with vacuum cleaners with suitable exhaust filters or absorbent mops. Designs which involve cleaning by the traditional "duster and brush" approach which simply spread contamination more widely should be avoided. Ventilation intakes and fume cupboard exhausts must be sited carefully so as to avoid re-circulation of laboratory air and the associated risk of contamination of test materials and hazard to laboratory staff.

3.3 Environmental Control

Adequate control of temperature, humidity and dust is important to staff comfort, instrumental performance and safe working (e.g. with flammable solvents). If they are to perform properly optical instruments often require stable temperature conditions. Electronic equipment may have prescribed operating ranges for environmental temperature and humidity. Computers may need to be protected from strong magnetic fields from other equipment; any staff or visitors with heart pace-makers must avoid such fields. Cooling water, either from mains supplies, or localised refrigeration may be necessary for the proper functioning of some equipment. Test materials, reagents, standards may need to be stored under controlled conditions. Some substances are affected by sunlight or fluorescent lights and must be protected from it. Delicate balances and optical instruments may need to be protected from vibration (e.g. from blenders, shakers and centrifuges) or may even need stabilised supports. All these needs have to be identified and documented so that proper procedures for monitoring them and taking necessary action can be included in the quality assurance system.

Records will be needed which show that:

- samples are received, stored, handled and analysed under environmental conditions that will not adversely affect analyses;

- temperature, humidity and light controls are adequate in sensitive areas to protect samples, extracts from them, personnel and equipment;

- the results of environmental sampling in laboratory areas are recorded; these should include records of air-flow rates across fume cupboard apertures.

3.4 Housekeeping Control

As with any other aspect of the laboratory's activities, the responsibility for housekeeping activities must be clearly defined. Cleaning staff and laboratory staff must each have clear instructions as to their respective duties in relation to:

- cleaning of floors, vertical surfaces (e.g. cupboards, walls, windows and doors), horizontal surfaces (e.g. work surfaces, shelves), equipment, interiors of refrigerators, freezers, fume cupboards, controlled environment stores.

- control of the contents of refrigerators, freezers, fume cupboards, controlled environment stores.

- checking the performance of air-conditioning and dust extraction equipment and of fume-cupboards.

- pest control.

The quality assurance programme will include work schedules, records of observations and of action required/taken covering housekeeping activities of this nature.

3.5 Documentation for QA Programme

Procedures for monitoring and recording critical environmental conditions (i.e. affecting sample stability, instrument performance, analytical validity and safety of personnel) and any action taken.

3.6 Reference

3.1 Weatherwax J. and Martin, P.G., (1986): Manuals of Food Quality Control: 1. The Food Control Laboratory, 2nd edition. Food and Agriculture Organisation of the United Nations, Rome, Italy.

4. STAFFING AND ORGANISATION

4.1 General Principles

Each task will be carried out by a member of staff who:-

- *is aware of what they are expected to do*
- *has been suitably trained*
- *is currently competent to undertake it.*

The QA programme will include documentation of this

4.2 The Management Structure

An up-to-date chart showing the organisational structure and lines of responsibility of the laboratory is an important feature of the quality assurance programme and should appear in the Quality Assurance Manual (see Appendix 4.1). When the laboratory is part of a larger organisation it may also be desirable to have a chart showing the management and operational relationships which control the input of work requested and the output of results from the laboratory, the overall remit of the laboratory and the resources available for it (see Appendix 4.2).

4.3 Job Descriptions

In a typical food control laboratory, there are two general types of technical personnel: analysts, who perform the actual analyses, and support personnel, who, with adequate training and supervision by the analysts, prepare solutions, clean glassware and instruments, and weigh test portions for analysis.

Both analyst and support personnel need to be aware of the importance of their duties and the need to report to their supervisor any situation beyond their skill, knowledge or control. The job description of the duties and responsibilities of each position in the laboratory needs to be part of the quality assurance programme. The organisational chart and the job descriptions should make clear the interactions involved in the quality assurance system - who gives instructions to do a piece of work and knows whether or not it has been completed successfully. This "feedback loop" provides confidence that if problems exist they are promptly identified and actioned and is an important ingredient of the QA programme. Particular care may be needed to ensure job descriptions cover actions outside the normal management line (e.g. on repairs to equipment, environmental problems).

The description of each position in the laboratory should contain at least three elements. First, an introductory paragraph giving a summary description of the position, indicating exactly how the position fits in the overall organizational structure. Second, detailing all the duties and responsibilities of the analyst. Third, describing the overall degree of supervision by the supervisor and the extent of the analyst's work-related independence.

In addition to appropriate education and experience, the analyst must be physically able to perform the tasks required by the job. Many laboratory positions require the ability to manipulate flasks and other glassware in a safe and expeditious manner, operate and maintain laboratory equipment and stand and/or sit for prolonged periods.

Both the personal and the professional qualities needed for a position must be considered. Some analyst positions require a team effort, and in those situations, the analyst must be able to work successfully with his peers to achieve a common goal. Other analyst positions require independent performance with little or no team work.

4.4 Staff Training

Staff training should be an on-going activity for all grades of staff and should take account of both long-term and short-term needs.

4.4.1 Responsibilities

The immediate supervisor, who is most familiar with the nature of the position and will be working with the analyst on a daily basis, should have input into the final selection of the analyst.

Selection of staff will normally concentrate on finding the most appropriate person (either a recruit or an existing member of staff) for a particular job - and then, for quality assurance purposes, demonstrating and documenting their skills. This may not involve much in the way of additional training. However, there are also wider considerations to be borne in mind. For instance there may be a promising member of staff whose career development and long-term usefulness to the laboratory will benefit from an opportunity to acquire new skills and experience. Or in order to ensure that there is a sufficient "pool" of staff experienced in a particular area it may be desirable to deliberately transfer someone without directly relevant experience into a particular position. In these cases training requirements may be more extensive.

Training should therefore be organised to achieve defined objectives that relate to the laboratory as a whole - and as such need to be the responsibility of a fairly senior member of staff. Overall training objectives might be to:

- ensure that in each of the appropriate analytical techniques, the laboratory has a sufficient pool of skilled analysts.

- ensure analysts practice investigational skills of analysis and develop interpretative skills.

- ensure each member of staff understands the purpose of their particular job.

- ensure each member of staff understands the need to implement the QA programme requirements.

- ensure analysts produce analytical data of known accuracy which are meaningful and assist in achieving the objectives of the laboratory.

4.4.2 Facilities and Procedures

There will be a need for training in specific analytical methodology, usually given in-house, as nearby organisations are unlikely to be undertaking identical work. There is also a need to ensure that analytical staff have adequate fundamental understanding of the science underlying the processes that they use. This knowledge should have been obtained during formal study but if there are gaps, these need to be identified and filled by attendance at short courses and seminars, by reading or by in-house training. The highest level of training relates to interpretation of data. This will probably be largely in-house and relatively informal, as it may be feasible to pursue only as particular examples arise.

A new employee should be given a general orientation about his or her new surroundings. The following matters should be discussed: work schedules, work loads, introduction to co-workers and management personnel, location of library, eating facilities, leave policy, pay schedule, locations of laboratories, procedures for disposing of waste or contaminated material, safety programme and appropriate laboratory attire.

After the general orientation, the new analyst is ready to begin a scientific training programme. A supervisor or senior analyst should be responsible for training the analyst. This training should be direct, i.e. "one-on-one" between trainer and analyst. The training of the analyst should be conducted in phases. During the first phase, the analyst is familiarised with all aspects of the quality assurance programme including operation and maintenance of equipment, documentation of sample accountability, preparation of analyst worksheets, etc. Depending upon the extent and/or detail of the quality assurance programme, this period of training may require 2-4 weeks.

The second phase of training could involve reviewing general analytical techniques, ensuring good analytical practice in "bench" skills, use of equipment and preparation of solutions, standards, etc. It should also include familiarisation with detailed procedures for laboratory notebooks, work sheets, records, etc. This phase of training may require up to 2 weeks.

The third phase of training is often the most extensive. It is concerned with specific analytical techniques used in the laboratory. Familiarisation with the proper use of spectrophotometry, gas chromatography, thin-layer chromatography and high performance liquid chromatography may often be an essential part of the training at this stage but the list of competencies needs to reflect the demands of the laboratory.

In teaching these analytical techniques, the trainer should do more than just demonstrate a procedure. Precautions to be taken in using a method (e.g. avoidance of emulsion formation, losses from concentrating extracts to dryness when "to small volume" is specified, the need to conduct some analyses in diffuse light) should be mentioned as well as the need to analyse certain products immediately, and the requirements for storing test materials before analysis at a particular temperature.

In addition to precautions associated with a method, the trainer must emphasise its limitations. For example, a method may not be applicable for the analysis of all types of foods; it may not be sufficiently specific, or may have a limited sensitivity.

During all phases of the training period, the trainer and the trainee should have frequent daily conversations. The training period should demonstrate both "how" and "why" for each analytical step. If the analyst does not ask questions during the training period, the trainer may have to generate discussion by questioning the analyst about his understanding of the various training segments.

The training record provides the documentary evidence that initial orientation has been conducted, that familiarisation with quality assurance principles and concepts has been provided, that competence in general laboratory skills and in basic instrumental techniques has been demonstrated. In relation to specific analytical methods it must record the date on which satisfactory performance was demonstrated (and the analyst was authorised to use that method) and the date when that performance must be reviewed; this will depend upon the complexity of the analysis and the degree of skill involved. Before a further period of authorised use is agreed the analyst will be required to show that during the period of authorisation he had used the method successfully on a specified minimum number of test materials. If, in fact, the analyst has not carried out this minimum number of analyses then the authorisation lapses until competence has again been demonstrated under supervision. If the method has been changed significantly since the initial authorisation appropriate familiarisation with this will have to be recorded. This documentation provides the assurance that analytical data is being produced only by analysts who are currently competent in the relevant analytical method.

Training should also be noted in the records of approved users for each instrument involved. This documentation, provides the assurance that analytical data is being produced only by analysts who are currently familiar with the instruments involved. A valuable operational bonus is that it also provides management with up-to-date information about who is currently competent to take on additional, unplanned work should this arise.

4.5 Staff Performance

Review of work performance, the training status and requirements for each member of staff should take place on a planned basis and procedures for this must be included in the QA Manual. Each analyst and his line manager should review the situation; the staff training officer will also need to be involved but not necessarily on the same occasion. Reviews should be held at least annually, but probably at 6 month or even 3 month intervals during the early stages of training. Care should be taken that the review is not perceived as negative or threatening by the analyst. The purpose of the review is not just individual accountability; it should provide the following: identification and action on both immediate and long-range training needs, a forum, which may otherwise not be available, for the analyst to discuss particular work-related problems, an opportunity for supervisor to recognise, commend, and document efficient analyst performance, and the opportunity for both parties to suggest improvements.

4.6 Support Personnel

Management and training of support personnel should be based on the same principles namely that they should be asked to do only those tasks for which they have received careful instruction and for which they have shown themselves currently competent. Glassware which is not properly cleaned or a balance which is not properly used can ruin an analysis just as surely as bad analytical technique - and may be more difficult to track down because of its random impact on quality. The procedures for implementing and recording the training of support staff may be less extensive than those for analysts - but they should be no less rigorous.

4.7 Documentation for QA Programme

1. *Organisational chart showing management structure of the Laboratory* — *Appendix 4.1*

2. *Organisational chart showing relationships to parent organisation and customers and others* — *Appendix 4.2*

3. Job descriptions for each post.

4. Training record for each member of staff showing

   - initial orientation
   - external courses attended (and any outcome)
   - general laboratory techniques, record keeping, principles of QA
   - instrumental competence, include re-validation dates (see also Chapter 6, Section 6.3)
   - analytical method competence, including re-validation dates (see also Chapter 7, Section 7.5.3)

5. SAMPLES AND TEST MATERIALS

5.1 General Principles

The identity and integrity of materials being handled by the Laboratory must be ensured throughout the time when they are under the control of the Laboratory, e.g. from sample receipt to data report and authorised disposal of surplus material.

The analytical data reported must reflect the composition of the received sample as a whole.

The QA programme will include appropriate procedures and documentation.

5.2 General Considerations

The term "sample" is used traditionally to describe a small part of a larger bulk of material which is taken for some purpose. In food control work it has been used to refer to material taken from a "lot" (e.g. a consignment, container, batch, load); in the laboratory it has been used to describe a portion taken from a larger amount (usually after homogenisation) and this may or may not be the same as the portion actually used in an individual analysis.

5.2.1 Terminology

Because of the confusion arising from the several different usages, Codex Alimentarius, AOAC International and IUPAC now encourage use of the term "test material" to describe items accepted by a laboratory for analysis; if as is usually the case only a part of the test material is used for an analysis this should be described as the "test portion". In line with these recommendations, the term "sample" is used for the part of a larger "lot" (e.g. a consignment, batch, carton, container, load) which is sent to the laboratory for analysis. Usually the person taking the "sample" is not a member of laboratory staff and their actions are outside the scope of the laboratory quality assurance programme. When the "sample" is received by the Laboratory it becomes a "test material". The laboratory is responsible for its "traceability", i.e. ensuring from that point, that any individual test material (and the data relating to it) can be distinguished from all others handled by the laboratory. It also becomes responsible for its "integrity" i.e. for ensuring it does not become altered in chemical composition before the test portion is analysed. Normally the Laboratory cannot be responsible for the integrity and traceability of the sample before it arrives at the laboratory.

5.2.2 Sampling and Analysis

Since the intention in food control work is to be able to relate analytical data on a test material to the original "lot" from which the sample was taken it is important that the laboratory is involved in discussions on sampling plans and in determining minimum sample sizes. These often raise complex issues and are outside the scope of this book but they are of crucial importance when control is based upon the average content of component/ contaminant in a food

"lot". Confidence in the decision that a "lot" meets/fails to meet a specification is dependent both on:

- the effectiveness of the sampling plan in obtaining a sample which is representative of the lot and
- the quality of the analysis carried out on it.

A continuous quality assurance chain is essential to effective food control; conversely a programme which covers only part of the chain can be de-motivating to staff who see their quality assured data being used as part of a less effectively controlled overall situation.

Food materials are submitted for a variety of purposes, e.g. survey samples relating to food composition or dietary intake studies, or official samples which may be the basis for subsequent enforcement action. The quality assurance procedures are in principle the same, and aim to ensure test material traceability and integrity; however, in the case of official samples there may be additional rigour attached to the requirements for a continuous chain of documentary evidence about the persons responsible for custody of the test materials and the steps taken to preserve their integrity (including protection from malicious interference) from the time of sample arrival to time of the data report.

5.3 Sample Receipt: Test Material Identification

The receipt and identification of food samples by the laboratory is the point at which traceability of the test material starts for the laboratory QA programme. Without clear and unambiguous receipt and identification of each individual sample (including duplicates), Quality Assurance cannot be maintained. The laboratory register of test materials should be of a type where the papers are numbered and cannot be removed; loose-leaf binders should be avoided. Entries on computer-based registers must be protected against deletion and/or amendment; a "back-up" copy must be produced and stored separately from the original.

On arrival at the laboratory all samples must be promptly logged into the laboratory's test material record system. Each test material (whether a bag of cereal or a group of packaged food items) must be given an identification code - usually some form of sequential alphanumeric system is employed - which can uniquely identify that material and the records of its analysis. Optionally it may also convey coded information about the nature, origin and data of arrival of the test material. Thus, for example, the identifier 61356-901211-LK-NZ has an initial 5- digit serial number which provides primary identification; the identifier also conveys the information that this is a sample of lamb kidney from New Zealand which arrived in 1990 on December (month 12) 11. The 5-digit number must be used to identify all portions of the material being analysed or stored, all extracts during analysis and all the experimental records, chromatograms and worksheets.

The test materials register (see Appendix 5.1) will contain the following data:

Laboratory Number
Date of Receipt

Nature of Sample
Customer Name/Address*
Customer Sample Number/Identifier
Reason for submission+
Information Required
Condition of Sample as Received**
Condition of Seal (official seal/seal intact/seal damaged)
Supplier of Sample (if not same as customer)
How received (carrier)
Storage Location Code
* to whom results should be sent
+ e.g. casual, routine inspection, special survey, formal (enforcement) sample.
** indicate visual appearance, e.g. normal for the materal, mouldy, decomposing, visibly infested, can visibly "blown" or badly dented.

Identifiers provided by those supplying the sample cannot be relied upon for QA purposes. The way in which they are issued (and any mistakes, inaccuracies, ambiguities in this) is usually outside the scope of the laboratory QA programme and the laboratory must take care to limit its responsibility to those operations which it controls.

5.4 Test Materials

5.4.1 Control and Storage

In many laboratory establishments, samples are received and registered at some central point, where the identification number is issued to a test material. In other cases, samples are delivered directly to the section which is to analyse them and they are registered and identified there. In either case the registration and issue of a laboratory identification may be taken as the point at which the submitted sample is accepted by the Laboratory. From then on it accepts responsibility for the submitted sample (- which in the preferred terminology then becomes a laboratory test material), and the laboratory must record its location and the steps taken to ensure its integrity. Where appropriate, "tracking" will involve ensuring that records are kept of when, and by whom test materials are removed from - and returned to - the store (see Appendix 5.2).

The storage of test materials, is of major importance if the analytical data produced is to reflect, and be traceable, to the original sample. Deterioration of the test material invalidates any analytical results. Therefore, test materials must be stored so as to ensure their integrity, safety, legality and stability; the laboratory must guard against deterioration, contamination and loss of identity. Special care will be needed where trace analysis is involved in order to ensure that test materials and equipment are not contaminated by extraneous materials.

Foods deteriorate for many reasons. For example, meat can become infected with bacteria and it may lose water, fat, vitamins, protein, etc. if it is not frozen carefully or analysed immediately. Vegetable oils can oxidise with loss of triglyceride content with a consequential increase in free fatty acids and fruit and vegetables will decompose rapidly at room temperature due to the action of naturally occurring enzymes, and the action of natural ripening processes.

Storage of test materials should be appropriate to the food type bearing in mind the deterioration process and the length of time that, even under ideal conditions, food can be stored. There are three basic forms of storage; room temperature (dry room), refrigeration and freezing. The QA programme should specify the conditions to be used (see Appendix 5.3). There are also problems associated with the type of container in which food can be stored. Foods that contain fats and oils should not be stored in copper or metallic vessels and foods that easily dessicate, such as fruit, need to be stored in ways which avoid loss of water or "freezer burn".

Perishable, unfrozen food must be maintained between 0° and 4° C and frozen food must be kept frozen, preferably at -18° C or below. All perishable samples should be examined within 36 hours of collection. Perishable samples that cannot be examined within 36 hours after being sampled should be frozen. Canned or dry, nonperishable foods may be stored at room temperature before analysis.

Additionally, it may be acceptable to dry the test material and store the dried material pending analysis if the analyte will not be affected by drying.

Special considerations apply to test materials which are to undergo microbiological examination as well as chemical analysis (ref 5.1). If a test material is to be stored frozen and a number of separate analyses are to be performed it is preferable to sub-sample before freezing and draw on these as required.

All test materials when stored must be properly labelled so that the identification is not lost. For example it is unwise to mark samples with a pen on paper and put the label on the outside of the packet if it is to be stored in a freezer. The label will deteriorate very quickly. Likewise placing the label next to the surface of foodstuff can also ruin the label. The most effective method of labelling may be to place the label in its own plastic bag inside the test material container, but separated from the food by a suitable layer.

The sample is then stored until it can be matched with a suitable test note, containing all the above information and any other relevant information required for analysis and interpretation of results. The test note should preferably be of the type that incorporates enough space for the test results and observations. The sample and the test note should (when matched if they arrive at different times) be clearly and indelibly marked with the registration number and passed to the analyst. From this point onwards the analyst will identify everything pertaining to that sample with the laboratory number.

5.4.2 The Analytical Sample (Test Portion)

Before removing the test portion(s) for analysis, the analyst must be certain that all records are in order, integrity has been maintained, containers are intact, and seals (if any) are unbroken. Any ambiguity in the analytical requirements must be resolved; e.g. with canned sardines in oil, is the analysis to be done on the fish, the oil or the whole contents of the can.

For the analysis, the analyst first removes a test portion. If the test material comprises more than one item (fruit, vegetable, etc.) the test portion should contain material from each item - usually achieved by comminuting a number of items and removing a portion. With some

canned or packeted goods which can be assumed to have uniform composition, it may be adequate to use just one item. After the test portion has been removed the remaining test material is returned to storage.

5.4.3 Referral of Test Material

On occasions it is necessary to pass a test material to another laboratory for some specialised analysis, or because of an overload of work. Unless the other laboratory is part of the same QA programme or the two laboratories are both accredited by the same (or equivalent) schemes then this referral will mean that the test portion sent for that analysis ceases to be quality assured by the "parent" laboratory. This must be made clear in the analysis report to the customer, even though the other laboratory has been selected for its known expertise in the analyses concerned, and proper care has been taken to ensure the integrity of the test material during transfer.

5.4.4 Test Material Disposal

Quality Assurance systems go beyond ensuring that the identity and traceability of the test materials are maintained until the results are reported. Many formal Quality Assurance systems require records and sometimes samples to be kept for lengthy periods (often up to 6 years). In these cases disposal procedures will need to be documented and records of disposals maintained. The register should therefore have columns in it, for details of when, how and where the test material was disposed.

Any residual material, if valuable, such as a flavouring concentrate, hop extract or oleoresin, may be required to be returned to the originator. If so, the date of disposal and to whom, should be clearly recorded on the register and the test note and sample report; the record should be signed by the appropriate officer.

Similar considerations may apply to commodities which are taxable and/or have been illegally produced or imported.

5.5 Documentation for QA Programme

1. *Register for sample receipt: test material identification* — *Appendix 5.1*
2. *Test Material: Flow chart of sample submitted for laboratory examination* — *Appendix 5.2*
3. *Storage conditions for food test materials* — *Appendix 5.3*
4. *Instructions on marking/labelling of test materials*
5. *Format for test note*
6. *Statement to customer about QA status of samples referred to another laboratory*

7. *Statement about retention period, disposal authorisation and procedures for reserve materials*

5.6 Reference

5.1 FAO Food & Nutrition Paper 14/12: Manual of food quality control, 12. quality assurance in the food control microbiological laboratory (1991); Food and Agriculture Organisation of the United Nations, Rome, Italy. ISBN 92-5103053-7.

5.7 General Reading

"Instructions on Codex Sampling Procedures". Document CX/MAS 1 - 1987. Joint FAO/WHO Food Standards Programme: Codex Committee on Methods of Analysis & Sampling.

FAO Food & Nutrition Paper 14/9: Manual of food quality control. 9. An Introduction to Food Sampling (1988); Food and Agriculture Organisation of the United Nations, Rome, Italy. ISBN 92-5-102680-7.

6. EQUIPMENT

6.1 General Principles

Each item of equipment used in the course of work covered by a QA programme must be known to be in proper working order.

An instrument may be used only by staff with appropriate training.

For each instrument, the nature and frequency of performance checks and the person responsible for these should be specified. The outcome of the checks, any remedial action, the outcome of this, and (where appropriate) a current list of the staff trained to use it will need to be recorded.

The QA programme will include procedures for documentation of this.

6.2 Classes of Equipment

Most food laboratory equipment falls into two classes, i.e.

- processing equipment such as blenders and grinders, driers, ovens and furnaces, centrifuges, refrigerators and freezers, hot plates and water-baths, water stills and purifiers.

- Measuring equipment such as balances, pH meters, spectrophotometers (UV/visible and AA), high performance liquid chromatographs, gas chromatographs, polarimeters; although not necessarily a measuring instrument, microscopes can be included in this category.

Equipment in both categories requires some degree of maintenance and some require regular calibration checks. For each item the nature and frequency of the action to be taken and the individual responsible for ensuring it is carried out must be documented.

6.3 Restrictions on Use

Equipment that is used for quality assured analysis, should be restricted in its use. This means that the equipment should be used only by staff trained in its operation (see Chapter 4, section 4.4), and that it should be used only for recognised procedures for which it is suitable. The calibration and maintenance of such equipment should be performed only by those authorised to do so. All instrumentation and equipment used for quality assured analysis should be labelled as such and included in regular audit by the Quality Manager or his deputy.

6.4 Maintenance, Calibration and Repair

Once equipment is installed and functioning it has to be maintained and records need to be kept. How this is done will depend to some extent on the administrative system within the laboratory. In a self-contained laboratory there should be a comprehensive equipment maintenance programme. This should specify for each piece of equipment, its performance criteria; what performance checks should be made and how often; what action should be taken if an instrument fails to meet the performance criteria; what regular maintenance is required, either in-house or by an external service engineer; and a log kept of checks, malfunctions and repairs. This log may also record frequency of use, and by whom. A fairly obvious feature of such a system is that each instrument should be the responsibility of a named member of staff.

Where the instrument is capable of physical calibration, the logbook should include calibration records. This would, for example, include such things as periodic checks on wavelength calibration and absorbance calibration for spectrophotometers. With some types of spectroscopic equipment it may be desirable to carry out periodic checks on sensitivity and resolution using a standard solution of an appropriate chemical substance. For many analytical determinations a set of standards may be run every day as part of the calibration of the whole method; the results of these will provide an incidental check on the instrument.

Specific person(s) should in most cases be nominated to maintain and calibrate specific pieces of equipment and should have to sign the record book, endorsing any changes or calibrations made to the equipment. As soon as equipment has been found to need repair or to be out of calibration, it must be fully labelled so that it will not be used for analysis. The label designating the equipment unusable should only be removed after the instrument has been repaired and retested to the satisfaction of either the section leader or the Quality Manager. A record of the fault and its source, the repair and the recalibration should be logged. This maintains a basic record of the status of the equipment and the on-going record of any serious systematic faults that occur with it. The record book should also identify the location of operating instructions and technical manuals for the equipment.

The arrangements selected for maintenance, calibration and repair of equipment will depend on a variety of factors. Services may be available externally on a contract basis but may often be expensive and response times for repairs may be unacceptably long. Some expertise may exist in-house and it may be desirable to supplement this wherever new instruments are purchased; often some intensive training in maintenance and repair can be included in the purchase deal along with access to a telephone 'help-line' for technical advice on fault-finding and repairs. This will often allow a diagnosis of the problem and replacement of faulty components to be achieved without the expense and delay sometimes involved in calling in a service engineer. Purchasing decisions on instrument selection should attach due weight to cost and availability of these services in relation to the extent to which the laboratory output is dependent on speedy repairs if the instrument becomes unusable. Consideration should also be given to purchasing a carefully selected range of spares at that stage rather than seek approval and await delivery when a breakdown occurs.

6.5 Programmed Action

Some suggestions for maintenance performance checks and calibration procedures for basic laboratory equipment and glassware are given in the FAO manual on QA in food control microbiology laboratories) (ref 5.1). Appendix 6.5 gives suggestions for similar procedures for instruments and services commonly used in food control chemistry laboratories

- gas chromatographs (injectors, columns, ovens, gas supplies, detectors, data evaluation and calibration curves).
- high performance liquid chromatographs (solvents, pumps, columns, detectors)
- atomic absorption spectrophotometers (optics, burners, nebulisers)
- spectrophotometers (optics, cells, lamps)
- balances
- polarimeters

The extent and detail to which the procedures are established in the quality assurance programme will necessarily reflect the laboratory's work and objectives; in turn these should reflect conclusions reached from discussions with customers about the analytical standards their needs impose.

Equipment which is used for a wide range of work will need to be quality assured to a level which reflects the levels of performance needed in the most demanding area of work; if these are quite out of line with the major work-load it may be cost-effective to segregate the equipment used for the demanding work and use a more appropriate, modest and cost-effective level of quality assurance on the majority of the work. Accurately calibrated glassware may be appropriate to work where a $\pm 1\%$ coefficient of variation is required; it makes little sense to use it in some trace analyses where a ± 10 or 20% coefficient of variation is acceptable on the final result.

Whatever the decisions taken on this, the quality assurance documentation must make it quite clear what action is to be taken, who is to take it and how this is to be verified.

Here, as in other components of the QA programme, management must ensure that the QA programme serves the Laboratory's objectives effectively - it must not be allowed to determine what they should be.

6.6 Documentation for QA programme

1.	Lists of processing and measuring equipment used in quality assured work.	Appendix 6.1

2. Maintenance and performance checks schedule (and officer responsible) for each piece of equipment.

3. Log for each piece of equipment with

   - origin and commissioning records with current list of authorised users — Appendix 6.2

   - checks and defects record (date, symptom, action, diagnosis and remedy, date restored to use) — Appendix 6.3

4. Notice for defective equipment — Appendix 6.4

7. ANALYTICAL METHODS

7.1 General Principles

The method selected for an analysis must be appropriate to the purpose for which the results are required.

The current "in-house" performance characteristics of an analytical method must be known when it is used on test materials.

Any analytical method used on test materials must be followed in a consistent fashion and must have quality control procedures associated with it.

An analyst using a method on test materials must have demonstrated current competence with it.

There must be documentary evidence that each of these principles is being observed.

7.2 Choice of Method

Food analysis can be required for a range of purposes - for example:

1.	control of banned substances	- surveys - enforcement
2.	control of permitted substances	- surveys - enforcement
3.	compositional standards	- surveys - authenticity - enforcement
4.	food composition	- surveys
5.	dietary intake	- surveys

All analytical methods have an uncertainty attached to the results they produce and this must be taken into account when selecting the method to be used for a particular purpose. This uncertainty can have important implications where action has to be taken at a particular analyte level. There must be a clear understanding between customer and analyst about the way the data is to be used if the latter is to provide "quality" ("fit-for-purpose") results. The situation merits further consideration and some of the major factors are identified in the example considered below.

Consider a method which at an analyte level of 100 mg/kg has shown a standard deviation (SD) of ± 20 mg/kg during "in-house" use. Statistically there is 95% confidence that repeated

measurements on a test material containing 100 mg/kg will lie within 2xSD of the true value, i.e. between 60 and 140 mg/kg. So - in order to have 95% confidence of identifying all test materials containing at least 100 mg/kg, all results in the range 60-100 mg/kg must be regarded as potentially coming from material which actually had at least 100 mg/kg

- if the authenticity of a product is judged by a minimum 100 mg/kg content of a specific component then any test material giving a result in the 60-100 mg/kg range may be a "false negative" i.e. on further examination it may be shown to contain at least 100 mg/kg.

- conversely, any result in the range 100-140 mg/kg must be recognised as possibly coming from a material which actually contained less than 100 mg/kg.

- if the maximum permitted level of an additive is 100 mg/kg then any result in the range 100-140 mg/kg may be a "false positive", i.e. further examination may show it came from material which really contained less than 100 mg/kg.

- the smaller the standard deviation, the smaller the range of results which call for further analysis in order to avoid "false positives" or "false negatives", e.g. with an SD of ± 10 the 95% confidence interval is 80-120 mg/kg.

- it is important to know the current in house performance of the method: if the SD is thought to be 20 but is actually only 10 then effort is being wasted on an unnecessarily high level of re-examination of test material, but if the SD is actually 30 then some materials may be judged acceptable when in fact they are not.

- with banned substances or those with maximum permitted levels it may be necessary to consider the implications of allowing false negatives; if a single ingestion of contaminated food carries a significant risk to the consumer a 95% confidence of detecting positives may be inadequate and one may have to consider choosing a more stringent analytical parameter, but if the maximum permitted level is set, as with most additives and residues, at a concentration which can be consumed throughout a lifetime without significant risk then a pragmatic approach to setting the level of confidence in finding every "positive" may be acceptable.

- for survey purposes the requirements may be less demanding; since no action will be taken on individual results, then providing that on average the analytical method gives the true value (i.e. there is no systematic bias for the data and the numbers of false positives and false negatives are similar) then the actual value of the SD is less critically important than in enforcement work, nor is it so critically important to know its current value so closely.

7.2.1 Performance Requirement

Methods have to be chosen on the basis of their performance in relation to the agreed requirements. The most important technical ones are:

Accuracy - the lack of systematic error, how near the mean results from several determinations would be to the true value, particularly when near the concentration at which action may be taken - or, for a survey, over the range of expected concentrations.

Precision - the closeness of agreement between repeated independent determinations on the same test material particularly at concentrations near those at which action may be taken - or, for a survey, over the range of expected concentrations.

Limit of detection - specified in terms either of a given level of confidence in detecting the presence of a substance which should not be present or as a "less than" concentration which must be at least a factor of 3 below the level at which decisions may be needed.

Limit of determination - specified in terms of the lowest level at which the concentration can be quantified with a given degree of confidence.

Sensitivity - the change in response per unit change in concentration - usually specified at the concentrations around those at which action may be taken.

Specificity - the extent to which other (known) substances may give rise to an interfering signal.

Scope - the range of matrices to which the performance characteristics apply.

Practicality - usage range, relevance.

Reliability - ruggedness, relative non-dependence on operator skill.

Other characteristics which may need to be considered are simplicity, speed and cost.

7.2.2 Reference/Official Methods:

These may be specified by regulatory authorities for enforcement purposes or be stipulated by trade organisations as the basis for resolving disputes. They will have undergone extensive inter-laboratory testing and their performance characteristics will be well documented. The precision of these methods (i.e. the agreement between results obtained at different times or by different analysts or in different laboratories) is very important. On occasions these methods may not be strictly accurate, i.e. may be subject to a systematic error which leads to an incorrect result - but which affects all laboratories in the same way and does not affect valid intercomparison of results. Alternatively they may be procedures which give a method-dependent result for an uncharacterised food component, e.g. dietary fibre. Their most

important characteristic is their ability to produce comparable results in any competent laboratory.

In these cases the method must be specified in minute detail and followed with great care. Any departure from the prescribed method may introduce a systematic error which will introduce a bias to all the results from that analysis.

7.2.3 Routine/Official Methods

Again, these may be stipulated by trade organisations or by regulatory authorities and are regarded as the methods which may be used on an everyday basis for routine work. They will have been widely tested in interlaboratory comparison and although usually they do not give the same high quality of performance characteristics as the reference methods, they are quicker, more practical and less expensive methods which have adequate precision and accuracy for the majority of test materials. They must provide an acceptable level of agreement with the reference method on the "true" value of the parameter measured, particularly when this is close to an enforcement level. Where a particular result is liable to be challenged, the test material must be re-analysed using the reference method.

7.2.4 Routine/In-House Methods

These may be analytical methods which have been developed "in-house"; although some may be novel methods, more often they are based on an official method which has been simplified in some way so as to make it easier, quicker, cheaper, more convenient to use. They will not normally have been tested on an inter-laboratory basis but will have shown performance characteristics (other than inter-laboratory reproducibility) which are comparable to those of the official method. Like other routine methods they should agree well with the reference method about values close to an enforcement level and if a result is likely to be challenged, the reference method should be used in a re-analysis.

7.2.5 Screening Methods

Where it is expected that the majority of test materials will not contain the analyte at levels which will be of any further interest, a "screening" method may be used in order to select those few samples which merit further examination. Often these methods will be based on a technique quite different to that used in the reference method. A screening method will be rapid, simple, rugged and practical in the (usually) very small number of matrices in which it is to be extensively used and as the term implies it will provide a very reliable way of distinguishing between a few test materials in which the analyte concentration may approach (or exceed) an action level, and a large number which do not approach it. Quite rightly the performance characteristics by which a screening method will be judged as producing quality results will be very different to those used for a reference method. The "fitness-for-purpose" criteria are quite different - but in both cases a documented knowledge of the performance characteristics which determine their fitness for their respective purposes is equally important as far as quality assurance is concerned.

7.2.6 Standard Operating Procedures

The term "standard operating procedure" (SOP) has been used in the past mainly to describe administrative and financial procedures but it is now gaining increasing use in relation to the documentation of analytical methods for quality assurance purposes. As such it describes all the aspects of an analytical method (often using a format prescribed by ISO) which will make it possible for another analyst to repeat the analysis on some future occasion. Typically it comprises sections like Introduction, Principle, Safety, Scope, Sampling, Reagents, Standards, Apparatus, Procedure, Calculation and Quality Control.

As part of the quality assurance programme all "in-house" methods of analysis must be documented as SOPs. The Quality Manual will set out the procedure for approving a new SOP; usually this will involve review of the documentation on the procedure and its in-house validation by the Section Head and the Quality Manager. A list of SOPs currently in use and the sources of other documented methods (e.g. those of BSI, IDF, AOAC International) in use in the laboratory should be readily available; where more than one version of a published method exists, the list must make it clear which version is currently in use.

7.2.7 Authority to Vary Methods

Analysts must have readily available written copies of the methods in use and must follow the method exactly as written. Analysts must not make significant amendments to methods on their own authority; minor variations must be recorded. The decision to change to a different version of a published method will be taken by the Section Leader and the Quality Manager. They will also approve variations to in-house SOPs; this may involve a change in the procedure or its extension to include additional foods. The approval process should include a consideration of the extent to which re-validation of the method is necessary and examination of the outcome of any revalidation they call for.

7.3 Validation of Analytical Methods

7.3.1 Initial Validation

Before use in the analysis of a test material all methods must first undergo in-house validation and the quality assurance programme must specify what this is to involve. Methods developed elsewhere must be checked and shown to produce reliable results in the circumstances prevailing in the laboratory even though they have already been subjected to extensive inter-laboratory validation by expert organisations. The aim will be to verify that the performance characteristics achieved in-house are consistent with those established in the inter-laboratory validation. The Section Leader and the Quality Manager will approve the steps to be taken for in-house validation of a collaboratively tested method; these will normally be less extensive than the validation of an in-house method, and may be limited to an assessment by one analyst. They should include:

accuracy - as judged by the result obtained on a reference material and by the "recovery" obtained from a "spiked" sample

precision -	as judged from an agreed number of replicate samples and repeat analyses on different days with the same test material
specificity -	as judged by absence of interfering signals from a "field" blank of the intended matrix
limit of detection and/or determination -	as judged by either repeat determinations on a field blank or if more appropriate, a field blank spiked to a level significantly below the intended action level
sensitivity -	as judged by the ability to discriminate between closely similar concentrations in the region of the action level
scope and applicability -	if a method is to be used in a matrix different to that in which it was originally validated, its performance in the intended matrix must be checked. Methods can differ in their performance with apparently quite similar matrices, e.g. examples of cereal-to-cereal differences and species-to-species differences with meat are well documented.

Validation of a method developed in-house must additionally identify the factors which are normally revealed by an inter-laboratory trial. Thus it will include demonstration of the robustness of the method (i.e. that it works equally well in the hands of a number of analysts) and identification of any aspects of the method which are critical to its ability to produce good data, e.g. reagent quality, precautions against losses, column performance (progressive loss of resolving power, "ghost" peaks, cross-contamination).

7.3.2 On-Going Validation

Some form of on-going check that the method is continuing to work properly is necessary. The nature and frequency of these checks need to reflect the complexity of the method, the degree of skill required, its reliability "track record", the number of samples analysed, the pattern of use (e.g. on-going or sporadic) and the quality of data required. For quality assurance purposes the on-going checks on the validity of the method should be agreed between the Section Head and the Quality Manager and documented in the SOP so as to ensure that users of the method are alerted to the requirements. Usually the checks will take the form of some or all of the Quality Control procedures discussed below.

7.4 Quality Control Procedures

These will aim to provide a systematic check on the validity of every batch of results; the form this takes will reflect factors like those identified in the previous paragraph. Overall they will aim to show that the expected performance of the method is being achieved, that contamination is not a problem, recoveries of added material are at expected levels, agreement between replicates and between repeat analyses is adequate and that analytical results are in line with those expected. Some or all of the control measures outlined below will be needed and must be written into the SOP for the method.

7.4.1 Sampling Controls

In some cases it may be important to check that the test portion is representative of the material received; it will be necessary to demonstrate that the possibility of uneven distribution of the analyte in the matrix (e.g. aflatoxin in nuts and dried figs) or pesticides on some fruits and leafy vegetables) has been successfully taken into account and that results from a series of test portions taken from the same test material, agree to the extent expected from the known repeatability characteristics of the method.

7.4.2 Replicate Determinations

Where results are customarily reported on the basis of single determinations it should be the practice from time to time to include in an analytical batch replicate determinations on the same test material. Preferably the replicates should be randomised within the batch and ideally the analyst should not be aware of their presence.

7.4.3 Positive Controls

Where it is anticipated that the majority of test portions will not contain significant amounts of analyte it is important to analyse a test portion to which analyte has been added and show that the analysis produces the expected result. This approach can be used to check the effectiveness of screening tests or in presence/absence analyses or for checking the ability to correctly identify test portions containing analyte at the action level. It can be used to assess the incidence of false negatives.

7.4.4 Negative Controls

In the reverse situation, where most test portions contain significant amounts of analyte it is important to include a number of test portions which are essentially free of the analyte so as to provide confidence that laboratory contamination levels are acceptably low. This use of a "field blank" - a sample of the matrix which is essentially free of the analyte is particularly important in analysis for trace amounts of additives, residues and contaminants; it is preferred to reagent blanks - which contain no test matrix - since as well as monitoring contamination during analysis it allows an opportunity to assess the method's specificity in discriminating against interfering compounds in the matrix. Results obtained with "field blanks" also often provide the basis for determining the limit of detection and limit of determination.

The other form of negative control uses test materials known to contain analyte at levels just below the action level; results from these provide a check on the incidence of false positives.

7.4.5 In House Standards

When a large number of similar analyses are to be conducted over a period of time it is useful to check the self-consistency of the data by use of an in-house standard material. For this a relatively large amount of the matrix containing the analyte at an appropriate level, is homogenized and kept under conditions where the analyte is stable. At specified intervals a test portion from this in-house standard is included in a normal analytical batch. Over a period of time it is then possible to see whether the method is giving consistent data and to obtain an

estimate of the long-term precision being achieved. Ways of utilising the raw data for quality assurance purposes are considered in Section 7.4.7.

A large number of repeat analyses conducted on the in-house standard over a short time allow the in-house precision (repeatability) of the method to be calculated. If the method is one which has been collaboratively tested the in-house precision can then be compared with that achieved by the collaborating laboratories.

7.4.6 Certified Reference Materials

These are homogeneous test materials in which the analyte has been determined with great care, usually by a number of expert laboratories and preferably using a number of different analytical techniques. Their data is used to certify the materials in terms of its analyte concentration and the uncertainty attaching to that figure. By analysing an appropriate certified reference material (CRM) from time to time it is possible to check the accuracy of the results being obtained. The in-house standard demonstrates the precision (consistency) of the data being produced, whilst the CRM demonstrates the accuracy, i.e. how near the results are to the correct value. Although in principle a CRM could be used for both purposes it is not customary to do so, firstly because CRMs are available in only limited amounts and should be reserved for their intended purpose and secondly because it is rare to find a CRM which consists of the correct analyte at a relevant concentration in the required matrix. A CRM which does not meet these criteria is in fact a less valid check on precision than an in-house standard.

7.4.7 Control Charts

Through the use of an in-house standard, as described in Section 7.4.5 it is possible to obtain important information about the precision of a method as used within the laboratory and to identify any changes in this. By performing a large number of determinations on a standard material when the method appears to be working properly it is possible to calculate the mean and standard deviation for the analyte concentration. Results from regular re-analysis of the standard material as part of a normal analytical batch allow assessment of whether the method is under control, i.e. the standard material is giving results which are consistent with the original estimate of mean and standard deviation. The information can be displayed graphically in a number of ways, of which the control chart, described by Garfield (ref 2.1) is the simplest and most widely used. Garfield recommends that the initial control measurement comprise 2-5 determinations run numerous times over a defined period. It is suggested that 25 sets of measurements are required for the initial estimates of the mean of means and the standard deviation. The mean of means is used as the mid-point of the chart and warning limits are marked at +2 and -2 standard deviation; rejection limits are set at +3 and -3 standard deviations. Based on a normal distribution, 95.5% of means from subsequent sets of determinations should fall between +2 and -2 standard deviations and 99.7% should fall between +3 and -3 standard deviations. Indications that the analytical method is out of control are often taken as illustrated below in Table 7:1.

The procedure outlined above is based upon the means of groups of determinations. However many laboratories report results on single determinations and should use control charts based on points which represent single determinations; if so the initial calculation of the mean and standard deviation must also be based on single determinations. The absolute value of the

standard deviation will be larger but the statistical significance of the patterns in observations is not affected.

TABLE 7.1

CONTROL CHARTS: SOME INDICATIONS AND CAUSES OF A METHOD GOING OUT OF CONTROL

Observations	Possible Cause
1 or more point outside 3xSD	analyst performance, poor technique, deviation from procedure
2 consecutive points outside 2xSD	as above
4 consecutive points outside 1xSD	as above
7* consecutive points all above or all below mean	incorrect preparation of standards, incorrect preparation of reagents, incorrect instrument calibration, analyst error, contamination
7* or more consecutive points all increasing	deterioration of standard deterioration of reagents
7* or more consecutive points all decreasing	solvent evaporation from standard, deterioration of reagents

* a run of 4 or more consecutive points may trigger investigative action if the implications of a shift/drift in results is serious to the customer.

7.5 Quality Assurance Procedures

7.5.1 Analytical Method Performance

The Quality Control procedures outlined in Section 7.4.1 to 7.4.6 should be specified in the SOP for each analytical method. Typically the SOP will specify the batch size and, as appropriate, the normal frequency of:

- "reagent blank" samples - and the maximum acceptable value and standard deviation

- field blank samples - and the maximum acceptable value and standard deviation

- spiked field blank samples - with the levels of spikes and the range of acceptable recoveries at each level

- undisclosed replicates - and the maximum acceptable difference

- in-house standards - with the expected value and its standard deviation

- certified reference material - with its certified value and uncertainty

SOPs will also specify the actions to be taken when the quality control checks show the method is not meeting the standards laid down. This may involve ceasing work on test materials until satisfactory performance has been re-established (preferably on the basis of an identified corrective action); in less extreme cases an increase in the level of quality control may be specified until causes of minor deficiencies are identified and rectified. As in other aspects of quality assurance, the programme must specify the action(s) to be taken, who must do so and how this is to be verified.

7.5.2 Traceability of Measurement

In order to be confident that the reported figure is correct the analytical procedure must be based upon some "standard", to which the measured value is directly traceable. In many cases this "standard" will be a sample of the pure analyte. Traceability then relies typically upon evidence

- that the balance on which a known portion of the pure analyte was weighed was correctly calibrated and functioning properly.

- that the glassware (pipettes, volumetric flasks, etc.) in which the primary standard solution was prepared - and subsequent dilutions made - was correctly calibrated, as also was that used in dispensing aliquots for "spiked" samples etc., etc.

- that if some physical property (e.g. the extinction coefficient at a specific wavelength) is used to verify the purity of the primary standard material then both the instrumental wavelength calibration and the absorbance calibration have been adequately verified.

- that if the measurement is traceable through a comparison with the response from an internal standard or from some other substance then the relative size of the signal from the two substances must have been verified, shown to be constant over a suitable range of concentrations and all analytical measurements made within that linear range.

The accuracy (and its uncertainty) which is needed for each stage in the chain of evidence which establishes the traceability of the final measurement, should take account of the overall performance characteristics of the analytical method. Traceability criteria will be much more critical in a method with a relative standard deviation (RSD) of $\pm 0.1\%$ at the concentration

measured than it is in one with an RSD of $\pm 20\%$. Nevertheless, for quality assurance purposes the traceability of a measurement must always be verifiable from the documentary records; without documented traceability there can be no confidence in the result.

Each SOP should identify the route through which results are traceable to a standard.

7.5.3 Authorisation to Use an SOP

An analyst may not use a method on test materials until the ability to use it has been demonstrated successfully during training. As appropriate to the method he may be required to show acceptable accuracy and precision with an in-house reference material, acceptable recovery of added analyte at relevant levels, acceptably low "blanks". The SOP (or an annex) should specify the "acceptability" criteria to be adopted, how long the authorisation lasts, any minimum number of analyses to be conducted in order to retain authorisation, etc. (See Chapter 4, Section 4.4). It will also list all those analysts currently authorised to use the method and the date their authorisation has to be reviewed/renewed. This list complements the list of SOPs for which each analyst is authorised and which is on his training record.

7.6 Documentation for QA Programme

List of methods approved for use in the laboratory:

Procedure for approval of new SOP

Procedure for in-house validation of new SOPs

Procedure for in-house validation of a collaboratively tested SOP

Standard operating procedure for each approved method — *Appendix 7.1*

Procedure for variation of an SOP

Quality Control Procedures for SOPs during use (Control Chart) — *Appendix 7.2*

Authorisation to use an SOP — *- procedure* — *Appendix 7.3.1*
- record — *Appendix 7.3.2*

8. CHEMICALS, REAGENTS AND MATERIALS

8.1 General Principles

Where the purity of a substance used during an analysis is important to the quality of the result, procedures should allow for verification later that only materials of the required purity were used.

8.2 Specifications and Ordering

Use of an unnecessarily high purity material leads to excessive expense; use of an inadequately pure material may lead to unusable data. Either way, resources are wasted. Each analytical method SOP should specify the quality of each of the materials used. This can be done in general terms, e.g. "Unless otherwise stated all reagents are of at least....[GPR (General Purpose Reagent) or AR "Analytical Reagent grade". Exceptions to this general standard are then specified in the SOP text and easily recognised by the analyst.

Specifications are also needed for items like chromatography columns - where, not only the column materials, construction and physical dimensions but also the critical performance characteristics may have to be specified. This may simply be the number of theoretical plates but in some cases the ability to resolve a critical pair of compounds may be specified. Where the purity or performance specification is critically important, it is prudent to have procedures for ensuring that consignments are tested on arrival and shown to meet the necessary standards. This should allow the items to be returned to the supplier and avoid wasting resources on unsatisfactory analyses. Absorbent "clean-up" columns used during some trace analyses and the immunoaffinity columns which are increasingly used in some mycotoxin and drug residue analyses, have been known to show considerable batch-to-batch variation in user-performance (although perhaps still meeting manufacturers' specification) and procedures for checking new batches should be part of the quality assurance programme.

Recording the date when new consignments are brought into the laboratory (and preferably when use commences) may usefully be included in the procedure. This may help in identifying the source of the problem if the quality control procedures prescribed in the SOP show that analytical quality has become unsatisfactory. Even quite common materials (e.g. sodium sulphate used for drying extracts in organic solvent) can sometimes contain impurities which interfere with analyses - so a suitable record system covering a wide range of laboratory materials may be a worthwhile component of the quality assurance programme.

8.3 Shelf-life and Storage

The quality assurance programme should identify the materials and situations which may lead to changes which can affect analytical quality and should specify the procedures to be followed in minimising them.

Usually this will be accomplished by taking quite simple measures, e.g. low temperature storage, exclusion of oxygen, exclusion of moisture, prevention of evaporation, exclusion of light and limitation of storage time. The documentation will need only to describe procedures which set out the storage temperature, the type of packaging and the shelf-life. The latter is often much shorter for dilute solutions, such as working standards than for pure compounds or concentrated solutions and the procedures must recognise this distinction. Also where storage temperature is specified, there must be an on-going record of the temperature in the refrigerator/ freezer/cold room used.

The other factor which must be considered is contamination. Storage of pure compounds or concentrated solutions of analytes to be measured in test materials at trace levels must ensure their strict segregation from test materials and dilute working standard solutions. This should preclude the use of the same glassware, etc. for both standards and test material. SOPs should specify the procedures to be followed.

8.4 Hazardous Materials

Every chemical laboratory contains substances which are actually or potentially dangerous. The working practices of the laboratory must ensure that the risk to which staff (and the public at large) are exposed from this hazard is acceptably low.

This will usually be achieved through laboratory design, selection and training of staff and by introducing appropriate documented measures for controlling the hazard from fire, explosion, contact with caustic or corrosive materials, exposure to harmful vapours, etc. Insofar as the quality assurance of the analytical results is concerned, these will tend to be associated with some of the pure/concentrated standards of highly toxic/carcinogenic substances. Here the procedures necessary to avoid equipment and test materials becoming contaminated with trace amounts will often be very similar to those needed to ensure staff are suitably protected.

8.5 Documentation for QA Programme

1. *SOPs to specify minimum reagent quality, performance characteristics, etc.*
2. *Procedures for checking that consignments meet critical specifications.*
3. *Record of arrival and first use of new batches of critical materials.*
4. *SOPs to include storage conditions for critical materials.*
5. *Temperature logs for refrigerators, freezers, etc., used for storage of critical materials.*
6. *SOPs to specify precautions to prevent contamination of test materials.*
7. *SOPs to identify hazards to personnel and specify procedures to be followed.*

9. STANDARD MATERIALS

9.1 General Principles

Analytical results need to be traceable to some quantitative reference point; often this is a standard material of some sort.

The validity of the analytical results then depends upon confidence that the identity and purity of this standard has been successfully maintained.

The QA programme will include documentary evidence of this.

9.2 Certified Reference Materials

Certified Reference Materials (CRMs) are materials in which one or more properties are sufficiently well quantified for them to be used for the calibration of analytical methods. The way in which this can be done is outlined in Chapter 7 (Section 7.4). The certification process will have included an investigation of the long-term stability of the analyte concentration under prescribed storage conditions. In using the CRM as the reference point for traceability of analytical results the chain of evidence relies upon the prescribed storage conditions for the CRM being observed. The quality assurance programme will require procedures for verifying that this was the case. Usually this will involve records of the storage temperature history of the CRM and of satisfactory accuracy in the thermometer used in recording it.

Traceability also rests on the assumption that the performance characteristics of the analytical method are the same for the CRM and for the test material matrices. With the limited range of CRMs currently available only rarely is it possible to provide a matrix-matched CRM. Under those conditions all one can do is look for evidence of matrix-dependent differences in analytical performance, (e.g. interferences, recovery differences) and, failing to find any, to accept the assumption on its merits.

The range of CRMs in the food area is still fairly restricted, and although the position will improve this will take some years. The timescale reflects the care involved in demonstrating the homogeneity and stability of the material, the painstaking measurements usually in a number of laboratories and using a number of techniques and the careful review of the results in order to arrive at a certified value for the analyte concentration and the uncertainty associated with it.

Presently the largest producers of CRMs include:

- the EC Community Bureau of Reference (BCR) in Belgium,
- the National Institute of Standards and Technology (NIST) - formerly the National Bureau of Standards (NBS) in Gaithersburg, USA
- the International Atomic Energy Agency (IAEA) in Austria.

A list of suppliers and addresses is in Appendix 9.1. The Laboratory of the Government Chemist in UK operates REMAS (Reference Materials Advisory Service), a computerised database listing over 4,500 separate reference materials from a range of sources.

9.3 In-House Standards

The concept of in-house standards and their function in quality control was considered in Chapter 7 (Section 7.4). To make a valid judgement about the long-term self-consistency of the analytical results a relevant bench-mark is needed; the in-house standard material is intended to provide this. Any long-term drift in the results indicates a change in instrument performance, in the concentration of the standards used for calibrating instruments and for "spiking" experiments to determine the recovery for the method or in reagent quality.

That conclusion rests on the assumption that the in-house standard is stable. The assumption can be tested by occasionally comparing the results with those obtained from a certified reference material which has been kept under conditions known to maintain the analyte at the certified value.

9.4 Standard Substances

Most analytical methods involve the use of a material of known purity as a source of working standards for calibration of instrument response and for "spiking" experiments to determine the analytical recovery. Material is often available from commercial sources, described as >95%, >99% or >99.9%. These figures are usually produced by a single laboratory and often cannot normally be given the same level of confidence as the figures for CRMs. They can pose a particular problem in presenting analytical results which are traceable only to material of this sort. For instance if a material is stated to be >95% pure, what figure should be assumed when making up calibration standards? 95%, 100%, 97.5% or what? If the figure 95% is used and the purity is actually 99% the results will all have a systematic error of approximately +4%. A pragmatic approach would be as follows:-

If the uncertainty due to the within-laboratory reproducibility of the analytical method is significantly greater than the uncertainty about the calibration standard purity, then the latter may not be important. If the uncertainty about the purity is broadly comparable to (or greater than) the within-laboratory reproducibility then the confidence in the results is affected and when results are reported this must be made clear. If the result's "fitness for purpose" is affected, efforts must be made to obtain a standard with more precisely documented purity. Providing that the evidence is properly presented, that information may be based on in-house measurements.

9.5 Hazardous Materials - See Chapter 8 (Section 8.4).

9.6 Documentation for QA Programme

SOP to include instructions for storage of CRMs and standard substances *Appendix 7.1*

Log of storage temperatures

Thermometer calibration record

SOP to include use of in-house standards and of CRMs (where available) *Appendix 7.2*

10. EXTERNAL PROFICIENCY TESTING

10.1 General Principles

An independent external assessment of the correctness of typical results provides an impartial test of analytical quality: this can be done through an inter-laboratory proficiency testing scheme.

Procedures used when analysing test materials for such a scheme must be those normally used by the laboratory.

Proficiency tests provide only "snapshot" information; they complement but do not replace, on-going quality control and should be seen as simply one part of the total quality assurance activities.

10.2 The Role of Proficiency Testing

The quality assurance procedures described in earlier chapters do not of themselves guarantee correct results; they provide confidence that any incorrect results will be detected and will assist in identification of the source of error. Proficiency testing - sometimes referred to as "performance assessment" - is the part of the quality assurance programme which looks at the accuracy ("correctness") of the results actually being reported by the laboratory on real test materials. Usually it involves presenting the analyst with a test material and requesting specific information about its properties; in a food laboratory the analyst will be told the nature of the food item and asked to report on one or more specific compositional parameters (moisture, fat, protein, ash, fatty acid composition, lead concentration, pesticide residues or drug residues, etc.). The analyst should treat the test material like any other, it must be analysed by the normal procedures and the results reported in the usual way. Ideally the analyst will not be aware that it is a proficiency test, but often this will not be feasible.

A single bad result from random testing in this fashion will no doubt prompt action to rectify matters, but a single good result in no way guarantees that all results are of the quality achieved on the proficiency test. A series of these proficiency tests and the long-term comparison between the expected results and the laboratory's results (and any trends or any discontinuities which this reveals) may be much more significant. The proficiency test therefore needs to be conducted quite regularly and should reflect the number of test materials analysed and the quality of data required.

The importance which will be attached to the results of proficiency testing seems likely to grow as a result of regional and international developments which

- will require laboratory data to be mutually acceptable between nations for many regulatory purposes

- will place the analytical emphasis on the performance characteristics of the method used by a laboratory rather than on the mandatory use of prescribed methods.

In response to these developments a harmonised protocol for the proficiency testing of chemical analytical laboratories has been prepared by a Joint ISO/AOAC International/IUPAC Working Party (ref 10.1). This identifies and explains the major features recommended for proficiency testing schemes. The major features of the protocol are listed in Appendix 10.1; it seems likely that, in future, proficiency testing schemes used in food control chemical laboratories will increasingly conform to this protocol.

10.3 Organisational Features of a Proficiency Testing Scheme

Day-to-day running of the scheme is the responsibility of a co-ordinator, working in consultation with a small panel of practising laboratory scientists which advises on general aspects of the conduct and content of the scheme. The co-ordinator documents all the practices and procedures of the scheme in a Quality Manual. Test materials must be homogeneous and generally similar in composition and analyte concentration to the materials routinely analysed; they should be distributed regularly and at a frequency somewhere between two weeks and four months depending on needs and resources. Participants should be notified in advance about the timetable for sample distribution, analysis, the reporting of results, their interpretation and the assignment and publication of "scores" (see below). Analysts use a method of their own choice i.e. the method which the laboratory would normally choose for test materials similar to those in the scheme.

The way in which the assigned ("true", "target") value for analyte concentration is decided must be clear. Laboratories will be assessed on the basis of the difference between their result and the assigned value. The harmonised protocol provides a statistical procedure for calculating a performance score for each laboratory. The results from all laboratories should be reported to participants in, at least, histogram form. Each participant will be notified of their own score but will not be able to identify those of other participants.

For the proficiency test to be meaningful the procedures used by participating laboratories must be those normally used e.g. replicate determinations should be carried out (and means reported) only if this is the norm. It is a matter of professional integrity to avoid departures from normal practice which may be expected to improve the score and, in effect, to falsify the results of the proficiency test.

10.4 The Limitations of Proficiency Testing Schemes

A proficiency testing scheme provides an assessment on the basis of what is usually only a very small fraction of the results produced with a particular method. Statistically, the confidence that a single result is representative of the laboratory's general performance of that analysis is low. However as the series of tests continues, the features of the laboratories' performance should be easier to discern. On the other hand, any long term trend may be obscured by a "step" related to a change of analyst, instrument, standard material, environment, training. Each proficiency test provides only a "snapshot" of the situation - and a series of tests possibly provides only a rather jerky "movie" of any changes in performance. There is also the potential disadvantage that the "camera" probably views only a restricted section of the range of analyses the laboratory conducts. This restriction can be minimised by choosing test materials

which are essentially a test of proficiency in the use of a widely used technique, e.g. gas chromatography, high performance liquid chromatography, atomic absorption spectroscopy, ELISA.

Notwithstanding these limitations it is clear that proficiency testing schemes have a clear and positive contribution to make as part of a laboratory's quality assurance programme. International developments are likely to encourage their further development and use; it seems likely that they will become regarded as an essential part of quality assurance programmes.

10.5 Resource Requirements: New Schemes

A major resource demand for proficiency testing schemes in the food areas lies in producing a steady supply of test materials of known homogeneity and stability. The cost of doing this is much the same for 20 participants as for 200; except in the case of liquids (e.g. beverages and oils) the costs of producing the test materials can be significant in terms of both cash and highly skilled manpower. These considerations tend to discourage the initiation of small schemes but the availability of the ISO/ AOAC/IUPAC protocol will make it much easier to appreciate what may be involved and to arrive at a reasoned decision on the relative merits of joining an existing scheme, seeking to extend the scope of an existing one or setting up a new one.

10.6 Documentation in Support of QA Programme

Charts showing successive "scores" in proficiency testing schemes used by the laboratory.

10.7 Reference

10.1 The International Harmonised Protocol for the Proficiency Testing of (Chemical) Analytical Laboratories (November 1992, ISO/REMCO N263)

(Note: this document is to be published by AOAC Int., probably August 93.)

11. LABORATORY RECORDS AND REPORTS

11.1 General Principles

All the information that has any practical relevance to test-materials and the analyses performed on them must be documented in systematic fashion.

At any point in its passage through the laboratory records must allow a test material to be traced back to its arrival and any information that arrived with it.

Records should be such that if the need for re-analysis arose, it could be done under the same conditions and in the same way as before.

Records must be retained and protected from misuse, loss or deterioration for an agreed time.

The QA programme will include procedures for providing this documentation.

11.2 Sample Collection Records

Responsibility for sample collection often falls outside the food control laboratory and will not be covered by the laboratory quality assurance programme. However, the topic is dealt with in FAO Food and Nutrition Paper 41/12(ref 5.1) which considers quality assurance in the food control microbiological laboratory; procedures suitable for chemical laboratories are readily developed from the principles set out there.

11.3 Analyst worksheets

The analyst worksheet provides a written account of the laboratory's analytical results. Although the analyst worksheets may appear in several forms, certain requirements apply to all types of worksheets:

- All the basic information must be recorded directly on the worksheet before analysis is begun. As soon as the worksheet is obtained, it should be initialled. As many entries as possible should be filled in at the outset.

- All entries should be clearly legible and made in permanent ink.

- No entry should be erased or "overwritten". If an incorrect entry is made, the analyst should draw a line through the incorrect entry, write above it the correct figure or word, and then date and initial the corrected entry.

- Data should not be discarded without explanation. If it becomes necessary to discard data, the analyst should put a line through the entry, initial, date and explain why the data has been discarded; the original data must remain legible.

- The exact analytical method should be referenced clearly and completely. If the method has not been published or is not covered by an SOP, it should be written in full on the worksheet or as an attachment to the worksheet. Many methods do not provide details for test material preparation. If the method cited is not specific as to test material preparation, the worksheet should describe fully how the test material and test portion (analytical sample) were prepared.

- If the analysis has been made in duplicate, triplicate, etc., the result of each analysis, as well as the summary of all results, must be recorded.

- The proper number of significant figures should be used. A larger number of figures than warranted by the limitations of the method gives a false impression of the accuracy of the method.

- If more than one analyst is involved in the analysis, the worksheet must clearly indicate which analyst broke the seal and which analyst performed each segment of the analysis.

- Any continuation sheets that accompany the analyst worksheet should be numbered in a consecutive series, e.g., 1 of 8, 2 of 8,...8 of 8 pages. An example of an analyst worksheet is shown at Appendix 11.1.

- Worksheets are checked for accuracy, completeness, and compatibility with other documents by the supervisor or his designated representative. The person who checks the worksheet is the individual who signs and dates this section. If an error is found, it is brought to the attention of the analyst, who then corrects it himself.

- The date on which the worksheet was submitted to the supervisor by the analyst is indicated.

The total number of pages, the individual page numbers, and the number of attachments are indicated at the bottom of the worksheet. The back of the worksheet is used as follows:

- The sample number is indicated in the upper right hand corner before any entry is made there. This ensures the analyst is entering data on the correct worksheet (without having to refer to the front) when more than one sample at a time is being analysed.

- The exact method used is referenced. Any modifications to the referenced method are stated and the reasons for these modifications are given.

- All calculations are clearly shown, with the proper number of significant figures used.

- For all weighings, the gross, tare and net weights are given.

- An explanation is provided of how dilutions were made.

- The use of controls and their results are specified.

- Data and calculations from any attachments or continuation sheets are summarized on the back of the worksheet.

Several types of continuation sheets may be used for specific analyses, and their associated instrumental methods. If, additionallly, laboratory notebooks are used, they should be bound, hard-backed and with numbered pages. All entries should be made in ink, any mistakes being crossed out with a single line, but remain legible and be initialled. All entries will be dated and the end of each day marked by a double line with the analyst's signature and date between the lines.

11.4 Instrumental Records

Analysts rely increasingly on instruments which produce a hard-copy record of the instrumental readings. Taking chromatographic charts as an example, these must be clearly labelled (test material number, analyst, date and any other necessary identifiers, e.g. replicate injection number) and stored in a logical sequence. Annotations to the chart should show identified compounds - or the region corresponding to an analyte which was sought but not found. Chromatograms of standards, recoveries and sample extracts must be cross-referenced to each other and to the responsible analyst's laboratory note book to allow for easy checking of results. The full chromatographic conditions employed must be stated on the chromatogram or must be readily obtainable from the analyst'1s note book.

Whilst the details of procedures for the outputs from other instruments may be somewhat different, the principles will be the same; they will also apply where outputs are in the form of electronic signals stored on magnetic media (tapes, diskettes, etc.).

11.5 Laboratory Reports

The laboratory report will be a condensed version of the data appearing in worksheets and laboratory notebooks; it must contain all the information normally necessary to allow the customer to utilise the results it contains. The format may be determined by either the customer or the laboratory but typically will include:-

Name, address of laboratory

Name, address of customer

Certificate/Report number

Page Identification (page x of y)

Sample receipt details (dates, names of deliverer, receiver)

Unambiguous identification of sample/test material (description, laboratory number, etc.)

Analyses conducted, methods, procedures, any deviations from standard practices

Preparation of test material, taking of test portions

Results (specify whole product/dry matter/fat basis as appropriate; state if results have been corrected for recovery if appropriate).

Uncertainty of measurements

Comments on significance of findings (- if expected by customer)

Date of Report

Authorising signature

During preparation of the report to the customer, all results must be submitted to an appropriate level of checking. The extent to which this must be done will be determined by laboratory management, after consultation with customers where appropriate; the procedures to be followed must be documented. The principle must be that no analyst is allowed to release results on their own authority; all results are to be checked by a senior officer - often a section head, before being released in a report. This maintains uniformity, centralises authority and ensures an agreed level of checking and verification before the laboratory is committed to supporting the validity of the results.

The use of page identifiers ensures that the customer can verify the completeness of the report received; the handwritten authorising signature provides confirmation that the report is an original document.

These procedures are followed with results from, all test materials. This includes materials from proficiency testing schemes, since these are intended to assess the validity of the result as normally reported by the laboratory.

11.6 Retention of Laboratory Records

The sequence of records should form a continuity of documentation to produce a clear, accurate and indisputable history of the test material with all aspects of documentation in agreement.

All sample registers, worksheets, reports and associated documents must be retained for a period which is determined by management in consultation with customers and is documented. Typically this will be 5 years - this being thought to be the maximum period during which re-examination of the validity of any result may reasonably be requested; in some cases the period may be specified in national legislation. Special considerations may arise in relation to work with some particularly hazardous substances where any adverse effects are known to be of a very long-term nature; in these cases retention of a limited range of documents for up to 30 years may be specified.

Storage of such material should follow the normal rules of archiving in terms of indexation, traceability, security, appropriate levels of protection against fraud or tampering, and against damage from fire, flood, etc. Back up copies must be held of any records stored as electronic signals on magnetic media; these should be renewed at appropriate intervals.

Dates and signatures of individuals who withdraw and return documents in storage must be recorded.

11.7 Documentation for QA Programme

Analyst Worksheet *Appendix 11.1*
Laboratory Report
Procedures for checking of results
Procedures for authorisation of report
Period for retention of documents
Procedures for archiving and disposal of documents

12. QUALITY ASSURANCE, AUDITS AND ACCREDITATION

12.1 General Principles

The Quality Assurance Manual for a laboratory sets out the policies of the laboratory in relation to the quality of its output and describes the general procedures to the adopted in the implementation of these policies.

Quality Audits aim to provide an independent and objective appraisal of the extent to which these procedures are being used and are effective.

12.2 The Quality Assurance Manual

The Quality Assurance programme for a laboratory covers all the policies and activities which can affect the quality of its output. Earlier chapters of this book have considered the major components of this and have identified the documentation which is required by each of these components. The aim is to have adequate records of what was to be done, how and by whom it was to be done and how to be confident that it had been done. Taken together this documentation provides the ingredients for the Quality Assurance Manual. These ingredients were set out in Chapter 2 and have been considered in subsequent chapters. The detailed contents of the Quality Assurance Manual (and any documents to which it refers) reflects the nature of the work-load and the ways in which the laboratory does its work. Very probably both of these will change with time and some components of the QA Manual will need revision. So the QA Manual must include procedures for its own revision and for the documentation of this revision. Often this is achieved by having a loose leaf format, each section numbered, the pages within each section identified (page x of y) and the issue number and its issue date recorded for each page. This ensures that it is possible to check that the Manual is complete and up-to-date; this provides a means for future identification of the instructions in force at any particular date.

12.3 Audits

It is the day-to-day responsibility of each line manager to ensure that the procedures embodied in the QA Manual are implemented. For two main reasons, this needs to be verified. Firstly, it is an underlying principle of a good QA system, that it has an in-built mechanism for detecting failures to do what it prescribes. Secondly, it is almost inevitable that over a period of time, minor adjustments to working practices and management requirements will take place, and although trivial in isolation these may cumulatively lead to some QA system failure. For these reasons it is necessary to include a system for auditing the QA system and its implementation. In large laboratories this is usually conducted by a member of the QA unit. In smaller laboratories the QA manager may do the audit. Where the QA manager is also responsible for an area of laboratory work - as is frequently the case in small laboratories - it is necessary to arrange for another suitably senior member of the staff to audit the QA Manager's own area of laboratory work. His QA responsibilities will need to be audited, by someone with QA experience but preferably not by the Head of Laboratory or the senior manager to whom he is responsible for his QA work.

QA audits can be conducted in a number of ways - each valid in themselves, but none entirely adequate on their own. Often an audit will concentrate on one particular format but over a period of time the range of formats should be used. The QA Manual should specify the minimum frequency for audits - often 2-4 times a year is felt appropriate; usually these will be notified in advance but spot-checks should be permitted.

12.3.1 Current and Retrospective Audits

In a "current" audit, the auditor will identify a particular test material and ask to be allowed to track that particular item through the laboratory, from arrival to report, or through particular aspects of this. Where the analytical procedures extend over more than one day, the auditor may ask to see test materials at each of the stages of interest. The auditor will observe what is done, how it is done, compare this with what is laid down in the QA Manual (and its satellite documents), identifiying any departures in practice and any inadequacies in the procedures.

By contrast, in a "retrospective" audit, the auditor will select a particular item in the samples arrival register (or one in an outgoing laboratory report) and ask to see all the documentation relating to that test material, seeking evidence on the integrity and traceability of the test material and, checking that the reported result is the sole and correct conclusion which can be reached from the recorded details. In addition to examining experimental records this will involve checking that any equipment used in the analysis was known to be in good working order and calibrated properly at the time.

12.3.2 Vertical and Horizontal Audits

In a horizontal audit a particular aspect of the QA programme will be selected and the relevant records examined throughout the laboratory. Aspects which might be chosen are:

Training Record Sheets	Documentation of Methods
Equipment Record Sheets	Test Material Identification
Calibration Procedures	Test Reports

In a vertical audit the reserve material from one or more test materials already analysed will be called for, the identifications changed and the analyst be asked to re-analyse. If it is possible, this will be done without the analyst being aware of the audit. The auditor will compare the results with those obtained previously.

12.3.3 Audit Report and Follow Up

The report of the auditor will be factual and should avoid comment and subjective observations. It is not sufficient to say that "housekeeping was poor" - specific examples must be given. The report will go to the Section Head (and others if specified in the QA Manual) and its QA Manager. The latter will verify that corrective action has been taken within an agreed timescale. If the situation is such that confidence in the results is seriously affected, the QA Manager must promptly advise the Head of Laboratory. In extreme cases the Head of

Laboratory will stop further work in the affected area until the effectiveness of corrective action has been demonstrated - and will advise customers that results already reported may not be reliable.

12.4 Periodical Review

The QA system itself - its scope, function, format and performance - should be reviewed on a regular basis. With an established, functional system an annual review will probably suffice. The review will normally be an internal management function conducted by the QA manager along with Senior Management of the Laboratory.

12.5 Accreditation

There are a number of accreditation schemes around the world operated by government departments, professional bodies and other organisations, usually of national or international standing. These provide formal recognition that a laboratory is competent to carry out specific tests (or specific types of tests). Often a scheme, mandatory or voluntary, will operate for a particular country, region, industry or technical area. They require participant laboratories to comply with certain standards and the accrediting body will carry out assessments to ensure the standards are met. The International Standards Organisation has established in ISO Guide 25 (1) the guidelines for assessing the competence of testing laboratories and these have been developed further by other organisations, e.g. in the EN 45000 series of standards. Assessments are carried out by professional assessors, who will usually be expert in the same area of work as the laboratory, and will have undertaken a course in laboratory assessment.

There are usually three major components involved in accreditation

- preparation of a QA Manual which documents the laboratory's QA policy, system and procedures
- periodical on-site visits by assessors, and
- participation in appropriate proficiency testing programmes.

A laboratory must comply with the accrediting organisation's requirements in all three areas if it is to achieve and retain accreditation. Often the emphasis is on ensuring a good quality related management and technical infrastructure to the work, rather than on detailed monitoring of the level of scientific and technical excellence achieved. The latter is quite rightly a "fitness-for-purpose" matter for agreement between the laboratory and customer - the former is concerned with confidence in whatever data is provided.

Laboratory accreditation programmes claim several advantages:

- promotion of reliability of analytical results.

- financial savings since the results of one laboratory can be accepted by another without the added expense and time involved in retesting.

- increased credibility, recognition, and status of approved laboratories.

- feedback to standards-producing groups on the adequacy of test methods which in turn, could result in an ultimate improvement in analytical methods.

12.6 Documentation for QA Programme

Quality Assurance Manual *Appendix 2.1*

Audit procedures
- *general arrangements*
- *QA managers' work*
- *report and follow up*

Procedure for periodical review of QA programme

12.7 Reference

12.1 ISO Guide 25, "General requirements for the competence of calibration and testing laboratories (third edition 1990). International Organisation for Standardisation, Geneva, Switzerland.

LIST OF APPENDICES

Appendix 1.1

Statement of Laboratory Objectives (example)

SECTION 1

ARCHIMEDEAN LABORATORY		SHEET:	1 OF 1
QUALITY MANUAL QM1		ISSUE NO:	1
QUALITY POLICY	I	ISSUE DATE:	14 OCT 1991
		ISSUED BY:	A HOKES

1.1 QUALITY POLICY STATEMENT

1.1.1 It is the overall policy of the Archimedean Laboratory to achieve standards of analysis which meet its sponsor's requirements and then maintain that quality in every aspect of every analysis and procedure taking place or to take place in the Laboratory for the purpose of advising the King (or others from whom Crown policy is derived) on the chemical and physical analysis and microbiological examination of foodstuffs and also for the purpose of the enforcement of all relevant food legislation.

1.1.2 The Archimedean Laboratory Quality Manual QM1 prescribes the quality organisation of the Laboratory and the Head of the Laboratory is responsible for implementing the Quality Manual QM1.

1.1.3 Other Quality Manuals prescribe the quality organisation of various aspects of the Laboratory's work. These are listed below together with the persons responsible for their implementation:-

QUALITY MANUAL	REFERENCE	RESPONSIBLE FOR IMPLEMENTATION
METAL ANALYSIS	CM1	A L CHEMIST
VOLUMETRIC ANALYSIS	CM2	A BATH

1.1.4 It is the responsibility of all staff who work in or who are associated with the analytical procedures to familiarise themselves with the content of the Quality Manuals and comply with the policies and procedures laid down in each manual and its associated documentation at all times.

Appendix 2.1

Items to be considered for Inclusion in QA Programme (example)

The U.S. National Institute for Occupational Safety and Health (1) has identified more than 20 elements that potentially may be covered in a quality assurance programme:

Statement of laboratory objectives
Policy statements
Organisation
Quality planning
Standard operating procedures
Recordkeeping
Chain of custody procedures
Corrective action
Quality training
Document control
Calibration of instruments
Preventive maintenance
Reagent and reference standards
Procurement and control
Sample identification and control
Laboratory analysis and control
Interlaboratory and intralaboratory testing programmes
Handling, storage, and delivery of samples
Statistical quality control
Data validation
System audits

Ref 1: Specification for Industrial Hygiene Laboratory Quality Program Requirements, 1976, National Institute of Occupational Safety and Health, Cincinnati, Ohio, USA.

Appendix 2.2

QA Manual and Contents Pages (example)

Ministry of Agriculture, Fisheries and Food

Food Safety Directorate

Q U A L I T Y M A N U A L
(QM 1)

for the

F O O D S C I E N C E L A B O R A T O R Y

This Manual is issued under the authority of: J GILBERT
Director
Food Science Laboratory

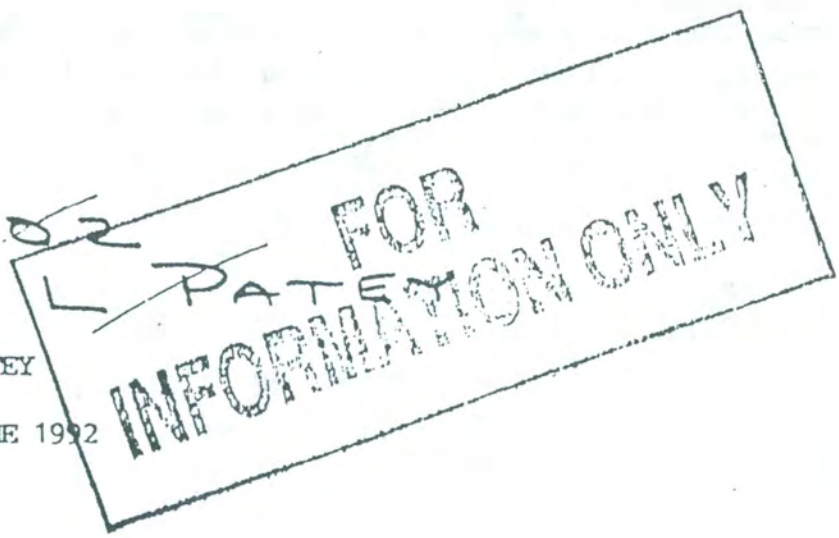
ISSUE NUMBER: 2

COPY NUMBER: 002

ISSUED TO: A L PATEY

ISSUED BY: A L PATEY

ISSUE DATE: 17 JUNE 1992



MAFF
Colney
Norwich NR4 7UQ

MAFF FOOD SCIENCE LABORATORY QUALITY MANUAL QM1	CONTENTS PAGE
	SHEET 1 OF 1
CONTENTS OF MANUAL QM1	ISSUE NO: 1
	ISSUE DATE 14 OCT 1991
	ISSUED BY: A L PATEY

SECTION

Appendix 2.3

Revised Page for QA Manual (Example)

EXAMPLE OF A QUALITY MANUAL AMENDMENT RECORD

Amendment Number: 77 Page 1 of 2

Date of Issue: 12/11/91

Laboratory Section: PESTICIDES

Manual Number: 1027

Room Number: C. 504

Signature of Quality Manager: _____

Signature Date: ___/___/___

DISCARD

SECTION	PAGE NUMBER	DATE OF LAST AMENDMENT
I	5 & 6	3 May 1991
II	27	1 November 1990

INSERT

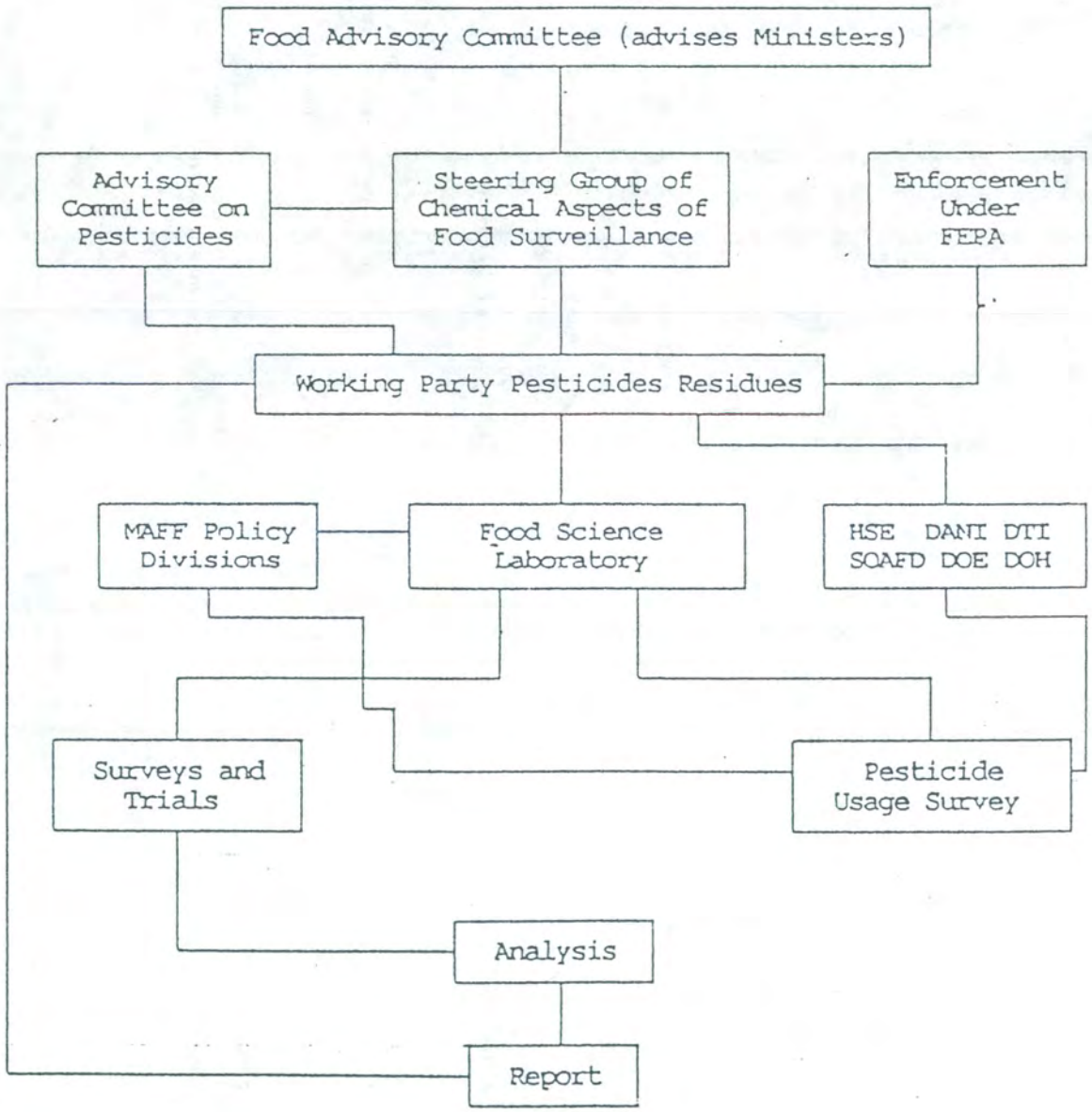
SECTION	PAGE NUMBER	DATE OF LAST AMENDMENT
I	5 & 6	13 November 1991
II	27	13 November 1991

Laboratory Management Structure (Example)

Appendix 4.2

Relationship Between Laboratory and other Organisations (Example)

Issue Date 8 Nov 1991
Issued by
Issue No 2



Example of a Sample Record

EXAMPLE OF A SAMPLE RECORD

PRODUCT:

Lab No: Product Code:

Date Received:

Storage Conditions: Storage Location:

Security Classification:

Sample Description:

Transfer Information:

Name Date out Location Analysis Date returned

References to Files, Records, Certificates of Analysis and Results

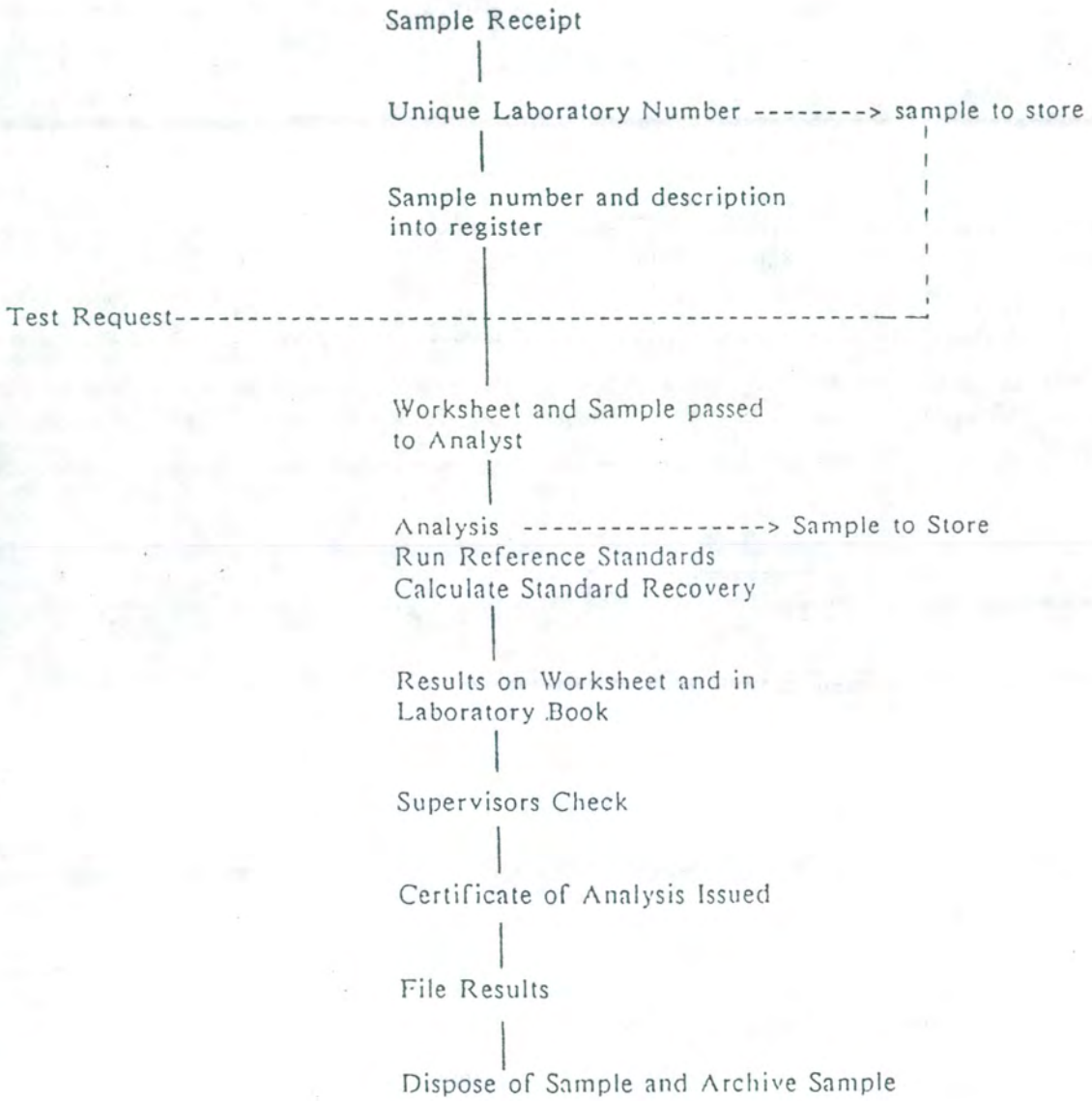
Number Location Subject Classification

Records Officers Name: Signature:

Date:

Appendix 5.2

Example of a Test Material Analysis Flowchart



Appendix 5.3

Storage of Test Materials (Food Items)*

Food	Storage
Baked Goods	
Ready-to-eat bread, rolls and buns	Freeze
Refrigerated or frozen unbaked breads, rolls, and buns	Freeze
Frozen sweet goods and cookies	Freeze
Ready-to-eat pies	Freeze
Refrigerated or frozen dough	Freeze
Crackers, biscuits	Freeze
Other bread and bread products	Freeze
Custard and cream-filled sweet goods	Freeze
Beverages and Beverage Materials	
Water	Refrigerate
Soft drinks	Refrigerate
Coffee, instant	Refrigerate
Coffee beans	Refrigerate
Tea	Refrigerate
Tea, instant	Refrigerate
Confectionery Products	
Chocolate and cocoa products	Refrigerate
Candy and confectionery products	Refrigerate
Chewing gum	Room
Syrups and molasses	Refrigerate
Honey	Refrigerate
Sugar, liquid	Freeze
Sugar, dry	Freeze
Dairy Products	
Butter	Refrigerate
Butter products (oil)	Refrigerate

derived from Ref.5.1

Food	Storage
Dairy Products	
Churning cream	Refrigerate
Cheese	Freeze
Cheese products	Freeze
Fluid whole milk	Freeze
Fluid milk products	Refrigerate
Concentrated liquid milk products	Freeze
Imitation dairy products	Freeze
Dried whole milk	Room
Nonfat dry milk	Room
Casein	Room
Ice cream	Freeze
Ice milk	Freeze
Frozen ices and sherbert	Freeze
Ice cream mix	Freeze
Ice milk mix	Freeze
Eggs and Egg Products	
Liquid and frozen eggs and egg products	Freeze
Dried egg and egg products	Room
Shell eggs	Refrigerate
Fish, Shellfish, and Seafoods	
Frozen fish	Freeze
Fresh fish	Freeze
Canned fish	Refrigerate
Dried fish	Freeze
Other fish (paste, roe)	Freeze
Frozen shellfish	Freeze
Fresh shellfish	Freeze
Canned shellfish	Refrigerate
Dried shellfish	Freeze
Seafood products (crab cakes, cocktails)	Freeze
Frog legs	Freeze
Smoked fish	Freeze
Smoked shellfish	Freeze
Smoked crustaceans	Freeze

Food	Storage
Flour and Flour Products	
Macaroni products	Room
Noodle products	Room
Pretzels, chips, and speciality foods	Room
Flour	Room
Corn meal	Room
Prepared mixes containing dried milk and dried eggs	Room
Fruits, Fruit Juices and Fruit Products	
Fresh fruits	Refrigerate
Frozen fruits	Freeze
Canned fruits	Refrigerate
Dried fruits	Refrigerate
Fruit juices	Refrigerate
Frozen fruit juices	Freeze
Jams, jellies, preserves, and butters	Refrigerate
Fig paste	Refrigerate
Olives	Refrigerate
Grain and Cereal Products	
Breakfast cereals	Room
Whole grains and beans	Refrigerate
Rice	Room
Oatmeal	Room
Infant Foods	
Infant cereals	Refrigerate
Milk bases, dehydrated infant food	Refrigerate
Milk bases, liquid	Refrigerate
Canned infant food	Refrigerate
Meats and Poultry	
Meat and meat products	Freeze
Poultry and poultry products	Freeze
Miscellaneous By-Products	
Oilseed by-products (cottonseed meal)	Refrigerate

Food	Storage
Miscellaneous By-Products	
Slaughtered animal by-products (bone meal)	Refrigerate
Fish and marine by-products (fish meal)	Refrigerate
Poultry and fowl by-products	Freeze
Fruit and vegetable by-products	Refrigerate/Freeze
Dairy by-products	Refrigerate
Cereal by-products	Refrigerate
Nuts and Nut Products	
Nuts	Refrigerate
Nut products	Refrigerate
Pet Foods and Animal Feeds	
Animal feeds, dry	Refrigerate
Animal feeds, moist	Freeze
Animal feeds, canned	Refrigerate
Pet foods, dry	Refrigerate
Pet foods, moist	Freeze
Pet foods, canned	Refrigerate
Processed and Prepared Foods	
Desert mixes, dry	Room
Pudding mixes, dry	Room
Frozen dinners	Freeze
Canned dinners	Refrigerate
Prepared salads	Freeze
Canned soups	Refrigerate
Dehydrated dinners	Refrigerate
Gelatin (dry)	Room
Yeast (dry)	Room
Spices, Flavourings and Condiments	
Whole spices	Room
Ground spices	Room
Mixed spices	Room
Extracts and flavours	Refrigerate
Essential oils	Refrigerate
Raw materials for extracts	Refrigerate

Food	Storage
Spices, Flavourings and Condiments	
Dressing for salad	Refrigerate
Dry salad dressing mix	Refrigerate
Other condiments	Refrigerate
Vegetables and Vegetable Products	
Fresh vegetables	Freeze
Frozen vegetables	Freeze
Canned vegetables	Refrigerate
Dried vegetables	Room
Pickles	Refrigerate
Vegetable oil	Refrigerate

Storage temperatures should be as follows: room temperature (20° - 23° C); refrigeration (2° -5° C); freezing (-18° to -22° C).

Appendix 6.1

Example of Equipment List for Quality Assured Analysis

SECTION 5 OF MANUAL CM2
ISSUE No 3

SHEET 1 OF 2
ISSUED BY A L PATEY

NAMAS "SIGNIFICANT" EQUIPMENT LIST
VETERINARY DRUGS DEPARTMENT
ISSUE 11.11.1992

No	Equipment	Supplier	Serial no	Nominee
51	autosampler, Gilson 231	Anachem	125571	D Tyler
13	autosampler, WISP 710B	Waters	710B-05374	D Tyler
19	autosampler, WISP 712	Waters	712-006986	D Tyler
3	autosampler, WISP 712	Waters	712-003572	D Tyler
8	autosampler, WISP 712	Waters	712-005730	D Tyler
16	autosampler, WISP 710B	Waters	710B-04021	D Tyler
39\2	balance analytical 4 place	Sartorius	37120106	K Chapman
39\1	balance universal 2 fig top	Sartorius	37030043	K Chapman
38\3	balance 2 place, P120	Metler	314225	K Chapman
38\1	balance 2 place, P1200	Metler	314601	K Chapman
38\2	balance 2 place, P1200	Metler	307111	K Chapman
56	fluorescence det, FD300	Spectrovision	89F3136	J Taylor
55	fluorescence det, HP1046	Hewlett Packard	2702G01241	J Taylor
6	fluorescence det, LS-4	Perkin Elmer	821445	J Taylor
40	fluorescence det, PU4027	Phillips	repair	J Taylor
23	gas chromatograph HRGC 5300	Carlo Erba	227293	G Stubbings
49	hplc pump, Gilson 307	Anachem	12557	J Bygrave
1	hplc pump, 2150	LKB	3676	J Bygrave
5	hplc pump, 2150	LKB	5458	J Bygrave
10	hplc pump, 2150	LKB	9001/8501/07157	J Bygrave
27	hplc pump, 2150	LKB	5045	J Bygrave
28	hplc pump, 2150	LKB	3621	J Bygrave
29	hplc pump, 2150	LKB	9001/8501/07203	J Bygrave
50	hplc pump, 2150	LKB	80/1305/45/0265	J Bygrave
17	hplc pump, 590	Waters	590-005376	J Bygrave
15	integrator Chromjet ch1	Spectra Physics	SP 030 04776	J Bygrave
9	integrator Chromjet ch2	Spectra Physics	SP 020 04514	J Bygrave
4	integrator 4290	Spectra Physics	SP 126 5455 120	J Bygrave
12	integrator 4290	Spectra Physics	SP 027 5052 120	J Bygrave
20	integrator 4290	Spectra Physics	SP 027 5812 020	J Bygrave
41	integrator 4290	Spectra Physics	SP 126 5416 020	J Bygrave
42	integrator, Chromjet ch1	Spectra Physics	SP 020 04513	J Bygrave
52	integrator, Chromjet ch1	Spectra Physics	SP 100 07309	J Bygrave
53	integrator, Chromjet ch1	Spectra Physics	SP 100 07308	J Bygrave
14	liquid chromatograph 1090	Hewlett Packard	2836G02656	J Tarbin
54	mass selective detector	Hewlett Packard	3050A01866	G Stubbings
25	pH meter AGB 2000	CSI	10692	K Chapman
26	pH meter model 292	Pye Unicam	167431	K Chapman

NAMAS "SIGNIFICANT" EQUIPMENT LIST
VETERINARY DRUGS DEPARTMENT
ISSUE 11.11.1992

No	Equipment	Supplier	Serial no	Nominee
44\5	pipetman (Gilson), F500	Anachem	J-79-10049	K Chapman
44\7	pipetman (Gilson), F500	Anachem	J-79-1000	K Chapman
44\1	pipetman (Gilson), P100	Anachem	D12550M	K Chapman
44\13	pipetman (Gilson), P100	Anachem	D12549W	K Chapman
44\3	pipetman (Gilson), P1000	Anachem	E24437L	K Chapman
44\4	pipetman (Gilson), P1000	Anachem	M-84-12157	K Chapman
44\8	pipetman (Gilson), P1000	Anachem	G-81-19963	K Chapman
44\10	pipetman (Gilson), P1000	Anachem	D12200A	K Chapman
44\14	pipetman (Gilson), P1000	Anachem	M-81-10000	K Chapman
44\20	pipetman (Gilson), P1000	Anachem	C17541B	K Chapman
44\9	pipetman (Gilson), P2OO	Anachem	C13532N	K Chapman
44\12	pipetman (Gilson), P20	Anachem	C29909L	K Chapman
44\17	pipetman (Gilson), P20	Anachem	A-80-15398	K Chapman
44\18	pipetman (Gilson), P20	Anachem	M-80-13665	K Chapman
44\2	pipetman (Gilson), P200	Anachem	C13536N	K Chapman
44\15	pipetman (Gilson), P200	Anachem	D-82-12290	K Chapman
44\16	pipetman (Gilson), P200	Anachem	C19322B	K Chapman
44\19	pipetman (Gilson), P200	Anachem	C11595N	K Chapman
44\6	pipetman (Gilson), P5000	Anachem	E-81-19101	K Chapman
44\11	pipetman (Gilson), P5000	Anachem	E18212R	K Chapman
44\21	pipetman (Gilson), P5000	Anachem	H18430J	K Chapman
46	UV absorbance det, AB 759A	Anachem (Gilson)	unavailable	J Taylor
48	UV absorbance det, LCUV	Pye Unicam	294718	J Taylor
11	UV absorbance det, SA6504	Severn Analytical	800688	J Taylor
45	UV absorbance det, SA6504	Severn Analytical	800267	J Taylor
2	UV absorbance det, SA6510	Severn Analytical	3030115	J Taylor
7	UV absorbance det, SA6510	Severn Analytical	3033	J Taylor
43	UV absorbance det, SA6510	Severn Analytical	3035	J Taylor
47	UV absorbance det, Spec' 100	Spectra Physics	02192	J Taylor
18	UV absorbance det, 484	Waters	484-PRB 611	J Taylor

Appendix 6.2

Example of Origin, Commissioning and Authorisation-to-use Record for Equipment

EQUIPMENT RECORD SHEET

SHEET 1

Reference No: (issued by Quality Manager)

Department: Veterinary Drugs

Project: _____
[BLOCK CAPITALS]

Name of Project Technical Manager: ~~Dr~~/Mr/~~Mrs/Miss/Ms~~ W.H.H FARRINGTON

Name of officer named by Technical Manager with overall responsibility for this item of equipment and for ensuring sheets I - IV describing the equipment are up to date:

Dr/Mr/Mrs/Miss/Ms _____
[BLOCK CAPITALS]

Name of item of equipment: _____
[BLOCK CAPITALS]

Name of manufacturer: _____
[BLOCK CAPITALS]

Model Number: Serial No: _____ Stock list no: _____

Date equipment received: _____19_____

Condition when received: new / used / reconditioned / on loan

Date equipment made operational: _____19_____

Current location - Lab no: _____ Bench no: _____

Location of manufacturer's operating instructions: _____
[BLOCK CAPITALS]

Names of officers authorised to use equipment:

(1) _____ (2) _____ (3) _____

(4) _____ (5) _____ (6) _____

(7) _____ (8) _____ (9) _____

[BLOCK CAPITALS]

OFFICERS AUTHORISED TO USE EQUIPMENT

File Number _____.

.....

Authorisation codes:

.....

Name of officer	Authorised by.	Date	Comments

Appendix 6.3

EQUIPMENT RECORD SHEET

SHEET II

Reference No:
(issued by
Quality Manager)

Name of item of equipment: _____
[BLOCK CAPITALS]

Stock list no: _____

Details of any checks made for compliance with relevant calibration or test standard specification:

DATE	DETAILS OF CHECK	RESULT

EQUIPMENT RECORD SHEET

SHEET III

Reference No:
(issued by
Quality Manager)

Name of item of equipment: _____
[BLOCK CAPITALS]

Stock list no: _____

Details of maintenance/repairs carried out. Note results of action taken.

DATE	DETAILS	PERFORMED BY

EQUIPMENT RECORD SHEET

SHEET IV

Reference No:
(issued by
Quality Manager)

Name of item of equipment: _____
[BLOCK CAPITALS]

Stock list no: _____

History of any lapses of validity or modifications.

DATE	DETAILS	RESULT

DO NOT USE THIS INSTRUMENT!

This instrument is not functioning properly and is not to be used for 'quality assured' work. Any enquiries to me, please

Quality Manager.....Date.....

[N.B. Removal of this notice is a disciplinary matter!]

Care of some Commonly Used Laboratory Equipment

1. Maintenance and Repair

1.1 Gas Chromatographs

As with any instrument, the reliability of a gas chromatograph requires that attention is paid to various aspects of the equipment and its mode and usage. Neglect of any individual part of the system can invalidate the results of analysis. To achieve reliable instrument performance it is essential to avoid build-up of contamination on the major components. This, in turn, is dependent on the nature of the test materials being analysed and for some applications may well be the most important factor in analytical quality. This is particularly so with trace analysis. Furthermore, there is no substitute for experience of this type of instrumentation as a total system. Continual monitoring by experienced chromatographers, particularly of the chromatograms and instrumental parameters is an important part of the checking procedure.

Maintenance, therefore, must be recognised to be a responsibility shared between the manufacturer's service staff for the overall instrumental system and the users of the components exposed to test materials, subject to deterioration in use and needing frequent attention in between service engineers' visits. The basic hardware is most efficiently maintained and repaired by trained service personnel with the requisite product information and equipment. However, it is often necessary for users to repair faults when it is impossible to obtain the services of a service engineer or when faults are recurrent and users become familiar with their rectification. Users need to develop their own maintenance schedule for the system components which come into contact with the test materials or which are subject to deterioration. Components that require regular attention include injection systems, columns, detectors, gas supplies and pneumatics.

1.1.1 Injectors

Regular replacement of septa and cleaning of internal surfaces, particularly injection liners is always necessary. On a daily basis septa should be checked for leakiness using a "SNOOP" type bubble fluid. Usually septa only last for between 25 and 30 individual injections, before needing replacement. Leaky septa should automatically be replaced with new septa that have been properly cleaned either by solvent extraction or by treatment in a vacuum oven prior to usage.

If a GC is in regular use the injection liner should be removed from the injection port (most injection liners are now glass), and either replaced with a new injection liner or the original injection liner thoroughly cleaned in a mixture of 50% nitric acid containing 7.5% potassium dichromate. The injection liner should then be thoroughly washed out with water and ethanol and replaced after drying into the injection port. The septa should automatically be replaced at this stage.

1.1.2 Columns

On a daily basis the column connections within the gas chromatograph should be leak tested using a "detergent or soap" type bubble fluid and tightened as necessary. The column should be inspected regularly by eye to make sure no gaps have appeared in the column packing or in the case of a capillary column that no breaks have occurred on the column itself. Column breaks can usually be checked by measuring the flow at the detector end of the column. Normally packed columns have a flow rate of approximately 10 to 25 ml per minute with capillary columns usually having a flow of somewhere between 1.0 to 2.5 ml.

On both a daily basis and a weekly basis the columns should be checked for resolution using a calibration standard (see Section 2.2). On a weekly basis packed columns should be tapped gently with either a pencil or a piece of wood to make sure that the packing within a packed column has not been disturbed. If resolution of peaks has been found to be impaired the column should be removed and an inspection made on the injection end of the column which may have deteriorated due to contamination from sample extracts. Solutions of appropriate standards, which need to be analysed to provide a basis for quantification, can provide vital, extra diagnostic information. A chromatogram should be examined in respect of the constancy of retention times, relative peak heights and symmetry of peak shapes, the absence of extraneous peaks and the regularity of the base line and signal to noise ratio. Unexpected changes normally indicate problems of degradation or contamination, either in the column itself or the injector or detector. Many packed and capillary columns are supplied with test mixes of components of various polarities, etc. and a corresponding test chromatogram, which can also be used to test column performance. Such a test mix can confirm if a column has deteriorated signficantly. (see Section 2.2.1 of this Appendix).

1.1.3 Ovens

Beyond checking the correct operation of the air circulating fan and venting system for cooling, the user has to rely on the temperature indication provided by the manufacturer. It is feasible to insert a calibrated thermocouple or a platinum resistance thermometer to check isothermal oven temperature control. Using this to monitor temperature programming may reveal disquieting sinusoidal variations about the ramp and overshoots of temperature when a set isothermal is reached. Precise, reproducible temperature control is essential for capillary work. However, some manufacturers are reluctant to reveal exactly what specifications and tolerances they have for programmed control. However it is the precise nature of the temperature control of the oven which is important. Providing the temperature is maintainable and constant, it does not matter if the oven is 1 or 2 degrees higher or lower than the thermal readout.

1.1.4 Gas Supplies and Pneumatics

Operating gas pressures and flow rates should be checked periodically so as to be kept in specification and appropriate for the system and application. Unexpected changes can indicate the presence of leaks which must be eliminated without delay. Fittings using rubber seals are prone to deterioration and should be replaced with all-metal unions. The purity of gases is important. Carrier gases should be purified using filter units to remove oxygen and moisture; helium will normally be used for capillary columns. Gases used in detectors should have hydrocarbon impurities removed by graphite filters. If a nitrogen carrier gas is used for

pesticide residue analysis using electron capture detectors a purity greater than 99.999% should be used wherever possible; this should still be scrubbed free of oxygen using an oxygen trap.

1.1.5 Detectors

By their nature and diversity, it is difficult to measure and monitor the sensitivity of detectors for gas chromatography, either as isolated units or independent of other gas chromatographic system components. In practice, they continue to be used until a noticeable falloff in sensitivity attributable to the detector is observed. Periodic cleaning often restores performance as does replacement of sensitive parts. Manufacturers cleaning instructions must be carefully observed.

Typically, 0.1 mg of methyl stearate should give full scale deflection of a flame ionisation detector. The sensitivity of an electron detector can easily be checked using a solution of lindane in n-hexane; typically 20-40 pg of lindane injected onto a gas chromatograph should give a full scale deflection. It should be noted here however that these are only guidelines to the sensitivities and responses of detectors will vary considerably with individual gas chromatographs and different manufacturers.

1.1.6 Data Evaluation

Peak heights or areas are usually evaluated by chromatographic data systems. These depend on the accuracy of analogue to digital conversion which may need to be confirmed over several orders of magnitude. The measurement of isolated, well resolved peaks which are situated on level baselines present little problem. However, if unresolved peaks on non-level baselines are to be quantified the operation of the integration system needs to be understood, particularly as regards its perceived detection of the baselines and dissection of overlapping peaks. Peak shape can also be an indicator of the efficiency and effectiveness of the GC column. Skewed peaks or non-Gaussian peak shapes often indicate that there is column deterioration, dirty injection systems, flow rate incompatibilities or incorrect compatibility between the analyte and the polarity of the column, or that overloading is preventing proper partitioning between gaseous and liquid stationary phases.

1.1.7 Calibration Curves

A series of standard solutions should be prepared such that the range of concentrations is wider than that of the unknown solution to be analysed. This enables the limits of linear response to the analyte of the detector to be determined.

1.2 High Performance Liquid Chromatography (HPLC)

HPLC is a technique that needs constant attention if optimum sensitivity and separation are to be maintained. Pumps can become damaged, columns can become blocked and can deteriorate, detectors can become contaminated; low flow rates, high back pressures, background absorbency and interferences can be the results. Care has to be taken during close-down procedures to ensure that any extraneous material is flushed from the system.

Components of an HPLC system that need regular attention are the pump, injection system column, detectors and solvents.

1.2.1 Pump

The HPLC pump's prime function is to deliver a steady flow of mobile phase at constant pressure to the separation column. The main working parts of the reciprocating pump are the pistons, the piston seals and check valves, the motor and the push rod cams.

The pump should be checked before each day's usage for constancy of flow. If the flow is found to be pulsing or intermittent then the system should be flushed with degassed solvent to remove entrapped air, the check valves should be thoroughly cleaned and, if fitted, the pulse damper should be checked for correct function. When seals and check valves are replaced, care is needed to avoid damage to them by over-tightening the fittings. The pumped value should be precise. However, it needs to be accurate only when the analyst is initially setting up and validating an analytical system. Once the system is validated only the precision of flow is important. At regular intervals the pumped column should be checked by setting the pump to a set volume and passing the pumped solvent into a measuring cylinder or other measuring vessel.

1.2.2 HPLC Columns

As with any chromatographic separation medium the HPLC column needs to perform with at least the efficiency necessary for the separation of analytes. Therefore the column should be regularly calibrated and the resolution monitored using standards (see section 2.3).

Columns can deteriorate for a number of reasons such as contamination, polarity change due to loss of activity, and disturbance in the geometry of the column. Contamination of the column can occur in the body of the column, through irreversible bonding of a component to the stationary phase, or more usually by retention of extraneous material on the top of the column. Columns need attention only if deterioration of resolution is analytically significant. If required the inlet end of the column can be removed, the retaining "frit" can then be removed and the column packing inspected; the removal of a few millimetres of packing and its replacement with new packing and a new "frit" may solve the problem, but this may cause other problems with some high-performance packings.

Loss of sensitivity of a column is the most serious problem and the reasons vary widely depending on the column type (Ion exchange, Silica, RP, bonded phase). Attempts to clean the column should be made by flushing the column with the usual solvent used for analysis and if that does not achieve an improvement then a different polarity solvent should be used, bearing in mind the need to choose a miscible solvent and the nature of the column packing.
Back flushing does not usually achieve results since it disturbs the packing, and causes uneven solvent flow. If the equipment and skills are available the most cost effective action may be to pack a new column.

1.2.3 HPLC Detectors

Various detectors are used in food analysis, the most common being based on the UV - visible, fluorescence, refractive index, and electrochemical properties of analytes.
The response of these detectors should be checked on a daily basis using the appropriate standard as recommended by the manufacturer, so as to detect "drift" in performance, often an early warning of impending detector failure.

Detectors, especially those that are used to measure post-column derivatised components must be thoroughly rinsed at the end of each day or on completion of an analytical run. Special care should be taken with detectors when used in conjunction with buffered amine or sulphonate modified mobile blocks. After use, the detector must be disconnected and flushed by drawing diluted nitric acid in water (1:10) through the unit. After standing for ten minutes, rinse thoroughly with water. This procedure ensures the removal of components that can produce insoluble deposits and block the instrument.

1.2.4 HPLC Mobile Phases

Ideally mobile phases should be prepared as necessary from a known stock of HPLC-grade solvents and filtered through a 0.45 micron filter to remove any undissolved matter. The mobile phase must then be degassed using helium or ultrasonic techniques and impurities assessed by flushing the HPLC system with the new solvent and measuring any background interference at the detection wavelengths. This can be a particular problem when using acrylonitrile with UV detection.

All solvents should be maintained in such a manner as to avoid contamination from airborne particles and atmospheric contamination from such pollutants as carbon dioxide, hydrogen chloride, chlorine and volatile sulphur compounds. Care must also be taken to avoid the formation of unstable reaction products (e.g. peroxides in tetrahydrofuran after removal of stabilisers during re-distillation).

1.3 Spectrophotometers

UV/Visible spectrophotometers are widely used in the food control laboratory. Their maintenance is a highly skilled job and usually requires the services of an engineer appropriately trained in the particular instrument.

The analyst should never tamper with the optical bench, mirrors, prisms, filters or photomultiplier, since in unskilled hands the slightest misalignment can make the spectrophotometer unusable.

General maintenance of spectrophotometers, including the cleaning of cuvettes and cell chamber should be performed before and after analysis. Spillages of acidic or alkaline solutions can do irreparable damage, even if left for only short periods of time.

Maintenance and changing the light source can be performed by the analyst providing that the manufacturer's manual is available and the maintenance is performed in the presence of an

experienced analyst. Immediately after such an action is taken a permanent record should be made. The instrument then should be recalibrated (see section 2.1).

Basic checks on wavelength and absorbance should be made using calibrated filters and standard solutions (see section 2.1) as recommended and supplied by manufacturers. Only rarely is it necessary to check against a primary standard.

1.4 Balances

Balances must be maintained in a thoroughly clean state, free from dust and corrosion. The balance, whether accurate to the nearest gram or milligram is sensitive to vibration, air flow, temperature fluctuation and horizontal "trueness" of the surface on which the balance rests.

All balances should be regularly checked to test that they have not been moved and that they are still on a level surface. Basic check weighings using calibrated weights should be performed daily. A permanent record should be kept of these checks, which should be cross referenced with the analyst's work book.

1.5 Polarimeters

Polarimeters are used primarily for the determination of the concentration and type of sugar solution. Two types are in common use, those which rely on the rotation of monochromatic light emitted by an incandescent sodium source and are mechanical in operation and those that are electronic in nature and use polarised polychromatic light. In both cases regular cleaning of light windows and lenses should be performed.

With modern electronic instrumentation, polarimeters have in-built systems that detect malfunctions, such as loss of power from the lamp. They also have a self calibration system. However both the mechanical type and electronic polarimeters need regular checking using standard quartz wedges, standard sucrose solutions or standard saccharimeter tube (see section 2.4).

1.6 Atomic Absorption Spectrometers (AAS)

Atomic Absorption Spectrometers (AAS) are used in the food control laboratory for the analysis of nutrient and toxic inorganic species. Because most of these species are at the trace level, it is necessary to expend considerable effort on the prevention of cross contamination, adsorption on to the surfaces of various parts of the spectrometer (such as the nebuliser, torch, sampling tube, etc.) and the alignment of the flame or furnace and the wavelength and source geometry of the light from the hollow cathode lamps.

The need for maintenance of atomic absorption spectrometers may be indicated by results being erroneously high or low, detection limits not being achieved, characteristic concentrations not being attainable, noisy data, high blanks, noisy base lines, inability to ignite the flame, irregular flame profile or that the burner flashes back. Another characteristic indicator is that the performance for flame/flameless analysis does not fall in line with manufacturer's specification.

If the the manufacturer's specification is not being met, there are various maintenance checks that can be made. However, most of them are only to be performed by a recognised manufacturer's service engineer or a highly skilled technician. Some of the simpler maintenance checks that can be made are to the optics, the burner and the nebulizer.

1.6.1 Optics

Avoid putting finger prints on the windows of the sample compartment or on the face of the source lamp, and also avoid touching the reflecting surfaces of mirrors or gratings. If dust collects on the optical surfaces, blow it off carefully with a clean and dry air bulb. Do not rub the surfaces with a cloth. Window surfaces may be cleaned with a tuft of cotton with a dilute solution of a mild liquid detergent followed by several rinsings with deionised water. If mirror surfaces become dirty (e.g. from exposure to laboratory vapours), cleaning should be done by a qualified service engineer. If a service engineer is not available, a skilled technician may clean the surfaces by careful use of clean cotton moistened with a clean solvent such as alcohol. Quick drying of the surfaces is important. Do not rub optical surfaces. Care must be taken to avoid scratches, which require resurfacing of the mirrors. Grating surfaces should never be cleaned or touched.

1.6.2 Burner

The nature of the samples aspirated determines the burner cleaning interval. In ordinary operation, the burner will need infrequent cleanings. Recondition the burner chamber after working with organic solvents or when samples with high solid contents are aspirated.

The burner should provide an even flame over the length of the burner slot. An uneven flame may indicate the slot needs cleaning. With the flame off and air on, carefully work along and through the slot using a thin metal strip. Do not nick the edges of the slot. Cleaning stubborn deposits from the burner head slot may require removing the burner head from the burner chamber. The burner head can then be soaked overnight in a detergent solution and then rinsed with deionised water and blown dry with a clean air supply.

If aqueous samples are aspirated or after aspirating organic samples such as oil or methyl isobutyl ketone extracts, the absorption signal produced can be noisy and erratic. After aspiration of organic samples, a clean organic solvent known to be miscible with the samples that have just been aspirated should be aspirated for 5 minutes. Acetone should then be aspirated for 5 minutes, and this should be followed by 5 minutes of aspiration with 1% nitric acid. If deposits have formed on the burner chamber, dissassemble and clean the burner by soaking in the detergent solution by using a bottle brush. Do not soak the mixing chamber in acid or use kitchen-type cleaners and avoid damage to the plastic lining of the mixing chamber from abrasive tools.

Flush the waste drain tube thoroughly with water. Empty the collection vessel and refill with water and dispose of hazardous or corrosive solutions properly and observe local codes concerning the effect of the waste on the environment.

Always clean the burner after aspiration of high concentrations of silver, copper or mercury samples in to an acetylene flame since unstable acetylides which are likely to explode

when permitted to dry may be formed. Thoroughly flush the burner mixing chamber and waste drain tube with water immediately after this type of analysis, and inspect the burner mixing chamber visually to be sure that all traces of residue have been removed.

1.6.3 Burner Drain System

The burner drain system must be flushed thoroughly with water to remove caustic, corrosive or organic waste materials that could otherwise damage the drain tubing and chamber. It is recommended that this procedure be performed at the end of each working day.

1.7 Processing Equipment

The maintenance of blenders, grinders and pin-mills, is normally restricted to cleaning, electrical fault diagnosis and the preventative maintenance of the degradation of seals. N.B. Only spark-proof blenders may be used with flammable solvents.

Cleaning is the most important aspect of care for this sort of equipment. Cleaning prevents cross-contamination of samples, particularly those that are fatty in nature, such as meats, confectionery and oilseeds.

Regular cleaning using water and detergent should be followed if necessary by a suitable solvent and then a detergent again. Typically, ground meat and fish should be removed from blenders by using hot water, a detergent and vigorous scrubbing. If this does not remove all evidence of fat then cleaning with a solvent, such as n-hexane may be indicated.

The maintenance of ovens, muffle furnaces, refrigerators and freezers should be limited to regular checks concerning temperature.

Although absolutely accurate temperatures for furnaces, refrigerators and freezers are usually not critical in food analysis, these pieces of equipment should perform to a minimum standard, although that standard will vary according to external temperature and manufacturer's specification. Typically, a refrigerator should attain 2° C to 5° C, a freezer -18° C to -22° C.

Refrigerators and freezers should be checked at specified intervals, typically a month, using a certified, total immersion thermometer; a continuously recording thermocouple must be used if temperature control is crucial. Furnaces should be checked using a pyrometer or a thermocouple device. If the temperatures are significantly different from those quoted in the specification for the instrument then the temperature control unit probably needs maintenance by a qualified engineer.

Drying and vacuum ovens are somewhat different, since some standard methods (AOAC, BS, etc.) specify drying to constant weight, either by a specific temperature, i.e. 100° C $\pm$ 1° C. Regular checking for temperature control on a daily basis with the aid of a certified thermometer is adviced.

The maintenance of vacuums in vacuum ovens can be checked by using a suitable calibrated manometer. The vacuum of a vacuum oven should be less than 100 mm of mercury as standard. Pumps and pipework may also need to be checked.

1.8 Glassware

Every food analysis laboratory still does some wet chemistry, and some do nothing else, so the maintenance of glassware and reagents is of great importance. It is also sound practice to segregate glassware used for trace analysis from that used for macro determinations. Established procedures for cleaning glassware (and also teflon, polythene, polypropylene, etc., if used) should be produced. It should be remembered that these depend on the calibre and training of staff that actually do the cleaning. For general use most glassware can be adequately cleaned with warm water and detergent without the need to use corrosive materials like chromic acid. However, there are some applications where glassware needs special cleaning and handling procedures. For instance, when measuring trace elements in water, for preventing adsorption or leaching from glassware, or not using glass at all. For trace analysis, in general, badly scratched or etched glassware should be avoided.

2. Instrument Performance and Calibration

Performance assessment and calibration techniques for some widely used analytical instruments are outlined below:

2.1 Ultraviolet and Visible Spectrophotometers

Spectrophotometers require calibration for both wavelength and optical density (absorption) accuracy. Wavelength accuracy can most easily be checked using either standard didymium and holmium glass filters. The accuracy of absorbance may be checked by scanning a 50 mg or 100 mg per litre solution of pure potassium dichromate in 0.005M sulphuric acid, between 200 nm and 400 nm.

The following absorbances are typical:

Wavelength (nm)	50 mg/l	100 mg/l
235	0.626 ± 0.009	1.251 ± 0.019
257	0.727 ± 0.007	1.454 ± 0.015
313	0.244 ± 0.004	0.488 ± 0.007
350	0.536 ± 0.005	1.071 ± 0.011

2.2 Gas Chromatographs

The most important aspects of the performance of gas chromatographs are in the areas of sensitivity and separation. Sensitivity is a function of the several factors including the correct functioning of the detector, proportion of active sites on the stationary phase of the column, the gas tightness of connections and analyte decomposition effects due to any local hot spots and metallic surfaces.

2.2.1 Gas Chromatographic Columns

Gas chromatographic columns are best calibrated with standard solutions of the analyte to be analysed, but information about the condition of a column can be obtained with the aid of standard test mixtures. Some examples are given below:

Test Mix 1 - For systems with a flame ionisation detector a useful mixture contains - 2-octanone, 1-octanol, 2,6-dimethylaniline, 2,6-dimethylphenol, decane, undecane, dodecane and tridecane in dichloromethane. This formulation can confirm column efficiency, the presence of leaks, the dead volume in the column, metallic absorptive sites, acid/base characteristics, hydrogen bonding or silanol groups and Lewis acids. This mix is used in a sensitive, isothermal or temperature programmed analysis to test a column's affinity for many compounds (Grob, et al., J. Chromatography, 156, 1, 1978).

Test Mix 2 - Also using a flame ionisation detector, a simple check on column efficiency for GC analysis of fatty acid methyl esters can be conducted by dissolving coconut (or rapeseed) oil in iso-octane, shaking with 10% methanolic potassium hydroxide for 2 minutes and, after allowing the phases to separate, injecting a suitably small volume of the iso-octane, upper layer directly onto the GC. Resolution of the methyl stearate and methyl oleate peaks is a good indicator of satisfactory column performance.

Test Mix 3 - For systems with an electron - capture detector a useful mixture for testing and evaluating gas chromatography columns for pesticide analysis can be purchased from companies such as Supelco in the USA. Alternatively an iso-octane solution containing 0.1 μg/ml of α-HCH, β-HCH, γ-HCH (lindane), p,p' - DDT, o,p' - DDT, p,p' - TDE, p,p' - DDE, aldrin, dieldrin, endrin, heptachlor and heptachlorepoxide. Some users also incorporate o,p' - TDE and o,p' - DDE but these occur only as degradation products from o,p' - DDT (itself normally only an impurity in p,p' - DDT) and are rarely encountered as residues in food. Careful use of text mixes (including "spiking" into extracts from test materials) and intelligent interpretation of the results is necessary if the all too common mis-identification of peaks is to be avoided. N.B. the MRL for total DDT = Sum of p,p' - DDT, o,p' - DDT, p,p' - TDE and p,p' - DDE.

Test Mix 4 - Interferences in pesticide analysis often occur through contamination with polychlorinated biphenyls (PCB). A useful test mix for calibrating and evaluating a GC column for PCB interference contains 10 mg per ml of either Aroclor 1242, or Aroclor 1254 in dichloromethane.

2.3 High Performance Liquid Chromatographs

The most important aspects to be considered when checking performance and calibrating a High Performance Liquid Chromatograph are the pumping/delivery system, the columns (efficiency and peak symmetry) and the detector.

2.3.1 Delivery System

The delivery system is normally a reciprocating pump or a single off-set dual piston pump. Whichever the system used, the correct calibration of flow delivery is of paramount importance,

since as with gas chromatography, the flow rate determines the speed of elution. The speed of elution governs the ability of the separation column to achieve equilibrium between the mobile and stationary phases and thus the efficiency of the whole system is affected. Without precise calibration of the flow rate, repeatability and reproducibility cannot be achieved. Calibration of the flow delivery system is a simple matter and is accomplished by allowing the eluent of the whole system to pass into a grade A measuring cylinder for an accurate time. Care should be taken to register a large enough volume for an accurate measurement to be made (i.e. 25 ml at 1 ml per minute into a 25 ml measuring cylinder). Greater accuracy can be achieved by weighing. It is always preferable to measure the flow rate of the whole system, since the back pressure from the column and its associated detector can vary, thus varying the pressure drop across the system and hence the flow rate.

2.3.2 Columns

Performance and calibration of columns varies according to the components being separated and measured. However, as a general rule, calibration can be performed using pure compounds and measuring various parameters, such as, elution time, resolution skewness and theoretical plate number.

Elution time is calculated as the time between the mid point of the eluted peak and the solvent front. If no solvent front is observed then the elution time can be calculated as the time between the mid-point of the eluted peak and the point of injection. Columns must be kept at constant temperature if variation of elution times is to be avoided.

Skewness of the eluted peak gives information as to the condition of the column and the state of the "frit" at column head. A skewed peak can be due to excessive active sites, deactivation of the column, tunnelling within the column, too fast a flow rate or the incorrect solvent. The skewness of the eluted peak is calculated by measuring the distances between the midpoint of the peak and the position of the trace on each side of the midpoint at half the peak height. The distances should be identical for a Gaussian peak shape.

The theoretical plate number of a column is a measure of the efficiency of the column. The higher the theoretical plate number the more efficient a column is and the greater the separation between two compounds will be. The theoretical plate number (N) is calculated by dividing the length of the column by the "height equivalent to a theoretical plate" (HETP):

$$N = \frac{\text{length of column}}{\text{HETP}}$$

The HETP is calculated by:

$$\text{HETP} = 16 \times \frac{(D)}{(W1/2)}$$

where "D" is the distance of the midpoint of the eluted peak from the point of injection, and "W1/2" is the width of the peak at half height.

2.3.3. HPLC Detectors

On UV/visible detectors the wavelengths are usually preset by the manufacturer at 254nm and 360nm but the absorption at these wavelengths can be used as a check on UV lamp deterioration. With variable wavelength detectors the smoothness of the changeover from deuterium to tungsten lamps (usually at around 320nm) should be checked.

Refractive index detectors can be checked with a solution of a suitable substance, e.g. 0.1% glucose for sugar analysis.

2.4 Saccharimeters and polarimeters

The most common methods for the calibration of instrumentation used for the determination of sugar content (by EWERS method, etc.) is by the use of a standard sugar solution. However, as a general rule, varying climates, temperatures and lengths of time for the solution to obtain equilibrium mean that results vary from laboratory to laboratory. A robust method is to use an optical activity tube that has been calibrated by an accredited competent authority and designated to have a specific rotation of x° sugar using a particular wavelength of light which is usually sodium light (4900A). If sucrose is the only sugar present , then a 26% solution in a 200 mm tube of a saccharimeter at 20° C will read 100%.

Polarimeters can be checked using the following concentrations of sugars in water:

Sugar	10%	20%
Sucrose	+66.5	+66.5
Glucose	+52.7	+53.1
Fructose	-90.7	-93.3
Lactose	+52.5	+52.5
Maltose	+138.3	+138.1

The above specific angular rotations are observed at 20° C using a 1 decimetre thickness and sodium "D" line light. All solutions should be made up using a few drops of 0.880 ammonia, to remove the effects of mutarotation and equilibrate the optical activity .

2.5 Atomic Absorption Spectrometers

The performance of a spectrometer using flame atomic absorption may be checked by the following procedures.

Set up the spectrometer for analysis of aqueous copper solution using the conditions of:

Wave length	324.8 nm
Slit	0.7 nm
Lamp current	15 mA (see manufacturer's specification)
Flame	air/acetylene oxidising (lean blue)
Burner configuration	flow spoiler installed

Using these conditions optimise the lamp position, the burner position and nebulizer. Then aspirate distilled water and zero the instrument. Aspirate an aqueous solution of copper having a concentration of 4 mg/l. Adjust the burner position and flame conditions for maximum absorbance. With most instruments the absorbance obtained should be approximately 0.2 absorbance units or greater.

The calibration of Atomic Absorption Spectrometers is best performed using standard hollow cathode lamps and standard solutions. The table below gives recognised validated absorbance wavelengths for a number of elements.

Element	Wavelength(nm)	Gases	Sensitivity[1]	Notes
Al	309.3	N-Ac	50.0	2
As	193.7	A-Ac	45.0	3
Ba	553.6	N-Ac	20.0	2
Bi	223.1	A-Ac	20.0	3
Ca	422.7	A-Ac	4.0	
Cd	228.8	A-Ac	1.5	3
Co	240.7	A-Ac	7.0	3
Cr	357.9	A-Ac	4.0	3
Fe	248.3	A-Ac	5.0	3
Ge	265.1	N-Ac	100.0	
Hg	253.7	A-Ac	200.0	3
K	766.5	A-Ac	2.0	2,3
Li	670.8	A-Ac	2.0	3
Mg	285.2	A-Ac	0.3	3
Mo	313.3	N-Ac	30.0	
Na	589.0	A-Ac	0.5	2,3
Ni	232.0	A-Ac	7.0	3
Pb	283.3	A-Ac	20.0	3
Sb	217.6	A-Ac	25.0	3
Se	196.0	A-Ac	30.0	3
Sn	286.3	N-Ac	150.0	
Sr	460.7	N-Ac	5.0	2
U	351.5	N-Ac	5500.0	2
V	318.4	N-Ac	90.0	2
W	255.1	N-Ac	450.0	
Zn	213.9	A-Ac	1.0	3

Notes:

1. Element concentration (mg/l) in aqueous solution giving approximately 0.2 absorbance units

2. Addition of an alkali salt (K, La or Cs as chloride) recommended to control ionisation

3. Use of the impact bead will improve sensitivity by about 2 x

For most purposes the use of "spectrosol" solutions supplied by major chemical manufacturers provides a relatively inexpensive route to calibration which is sufficiently accurate for most food analysis purposes; the standard solution should be accompanied by a manufacturers certificate of analysis; a batch number and an expiry date. A useful guide to calibration and concepts of Atomic Absorption Spectrometers are given by Barnett (1,2).

3. References

1. Barnett, W, (1984), Spectrochimica Acta 39B(6), 829-836.

2. Barnett, W, (1985), Spectrochimica Acta 40B(10-12), 1689- 1703.

Appendix 7.1.1

Standard Operating Procedure (SOP) for a Chemical Analysis (Example)

SOP 1(10)	1 of 9	TETRACYCLINE
Page Revision 1		

Method: 1
Issue: 10
Revision: 30 March 1993

DETERMINATION OF TETRACYCLINES AT RESIDUE LEVELS IN ANIMAL TISSUES AND MILK

Issued by _____
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1. SAFETY

This method involves the use of several hazardous chemicals and procedures likely to produce an increased risk to the operator.If in doubt always consult the appropriate Safety Advisor.

1.1. General precautions

1.1.1. Protective clothing including laboratory coat (buttoned), safety spectacles and gloves (latex or similar) should be worn at all times.

1.1.2. Tissue samples should be regarded as a biological hazard. Direct contact with skin is best avoided and proper attention to hygiene must be maintained. The use of scalpels or other sharp knives must always be carried out with care. Cuts must be reported immediately and attended to by a trained First Aider.

1.1.3. Operators should familiarise themselves with any risks attributable to chemicals and reagents.

UNDER THE CONTROL OF SUBSTANCES HAZARDOUS TO HEALTH (COSHH) REGULATIONS THIS METHOD MUST BE ASSESSED FOR ANY RISK TO HEALTH TO OPERATORS THROUGH ITS USE. A FULL ASSESSMENT REQUIRES THE COMPLETION OF COSHH1 AND COSHH2 DOCUMENTS BOTH KEPT ON FILE AND ACCESSIBLE TO STAFF. BEFORE STARTING THIS METHOD ANYONE USING THIS PROCEDURE MUST HAVE A COPY OF THE COSHH2 DOCUMENT. THE CODE OF PRACTICE RELATING TO VETERINARY DRUGS SHOULD BE CONSULTED BEFORE STARTING THE METHOD.

Particular attention is drawn to some organic solvents and chemicals which may be toxic and/or flammable. Contact by inhalation, skin absorption or ingestion must be avoided, work in a fume cupboard if necessary.

Methanol and acetonitrile are toxic.
Tetracycline antibiotics are listed as toxic.
b-Mercaptopropionic acid is corrosive and toxic.

There is no implied safety for any other compounds excluded from this list.

1.2. First Aid

Any injury must be reported, in the first instance, to a qualified First Aider and recorded in the accident book kept with all first aid kits. The First Aider will decide on further action. All accidents, incidents and near misses should be reported on form DHSU 1.

2. INTRODUCTION

Tetracyclines comprise a group of antibiotic agents that are used therapeutically and prophylactically in animal husbandry. The most widely used include tetracycline (TC), chlortetracycline (CTC) and oxytetracycline (OTC).

Concern about the possible occurrence of residues of these drugs in human food has produced a need for analytical methods to monitor for these agents at trace residue levels.

This standard operating procedure (SOP) can be used to monitor for residues of the common tetracyclines (TC, CTC, OTC) in animal tissue, namely kidney and muscle from several species including pig, cattle, sheep, fish (muscle), poultry (muscle), prawns, corned beef pate and honey. Bovine milk may also be analysed by this method. This method is intended for use only as a quantitative surveillance method capable of screening samples for the possible presence of tetracyclines.

3. PRINCIPLE

Sample is extracted by buffer and the extract applied to a chelating Sepharose column previously loaded with copper ions. Tetracyclines are eluted with ethylene diamine tetra-acetate buffer (EDTA). Residual copper and other organic contaminants are then removed by passing the extract through an XAD-2 resin column from which the tetracyclines are eluted with methanol. Determination is made by HPLC with UV detection.

4. SAMPLING

Procedures to be followed for the identification, logging and storage of all samples are given in the Food Science Quality Manual CM 2 Sections 9 and 10.

For analysis sub-samples are taken from thin slices through the frozen sample to gain as representative a sample as possible. Homogenisation of whole sample is best avoided as loss of analyte through increased enzymic activity may result.

5. APPARATUS

5.1. Glassware

5.1.1. Centrifuge tubes 250 mL and 50 mL capacity.
5.1.2. Glass columns 200 mm x 20 mm i.d. fitted with sintered glass frit and stopcock.
5.1.3. Glass filter funnel 250 mL
5.1.4. Conical flasks 250 mL.
5.1.5. Pear shaped flasks 50 mL.
5.1.6. Round bottomed flasks 250 mL.
5.1.7. Bulb pipettes 5 and 10 mL
5.1.8. Vials, low volume for autosampler.
5.1.9. All glass filter holder, 47mm — Millipore
5.1.10 10 ml tapered test - tubes

5.2. Equipment

5.2.1.	Homogeniser	Ultra Turrax or equivalent.
5.2.2.	Vortex mixer	Whirlimixer (Fisons) or equivalent.
5.2.3.	Centrifuge	MSE high speed 18 or equivalent.
5.2.4.	Ultrasonic bath	L&R 140S or equivalent.
5.2.5.	Rotary evaporator plus water bath	Buchi or equivalent.
5.2.6.	Filter paper, 15cm, type 541	Whatman.
5.2.7.	Safety pipettes 0.2 mL and 1.0 mL	Gilson Pipetman.
5.2.8.	Centrifuge bottles 250 mL.	
5.2.9.	Membrane filter 0.45 um "Durapore", 47mm	Millipore U.K. Ltd.
5.2.10	Thermostated dri-block and sample concentrator.	Techne

5.3. High Performance Liquid Chromatography

5.3.1.	Pump	LKB 2150 pump or equivalent.
5.3.2.	Column	20 cm x 3 mm i.d.Chromsep (Chrompack) cartridge column assembly packed with Chromspher C8 with integral (10 mm x 2.1 mm i.d.) guard column packed with pellicular (30-40 μm) reverse phase. Alternative reverse phase columns may be acceptable. Mobile phase pumped at 0.4 mL/min (6.2.4).
5.3.3.	Detector	UV detection at 350 nm (Severn Analytical,SA6510 or equivalent).
5.3.4.	Data/handling	Computing integrator (Perkin Elmer/Nelson Turbochrom software).
5.3.5.	Injection	Autosampler (Waters WISP 712 or equivalent capable of 10 μL injections).

6. REAGENTS

Chemicals and solvents are analytical grade reagents except where stated. Deionised double distilled (or equivalent) water is used throughout.

6.1. Chemicals

6.1.1.	Tetracycline free base	Sigma Ltd
6.1.2.	Chlortetracycline HCl	Sigma Ltd
6.1.3.	Oxytetracycline HCl	Sigma Ltd
6.1.4.	Succinic acid	BDH
6.1.5.	1.0 M sodium hydroxide solution	BDH
6.1.6.	Ethylene diamine tetra acetic acid, disodium salt (EDTA)	BDH
6.1.7.	Methanol	BDH
6.1.8.	Ethanol	Burroughs Ltd
6.1.9.	Acetonitrile	Romil
6.1.10.	Oxalic acid	BDH
6.1.11.	Chelating Sepharose Fast Flow in 20% ethanol	Pharmacia
6.1.12.	Amberlite XAD-2	BDH
6.1.13.	Copper sulphate	BDH
6.1.14.	b-Mercaptopropionic acid (Thiolactic acid)	Aldrich
6.1.15.	Acetone	Rathburn

6.2. Solutions

6.2.1. Succinate Stock Solution:
24 g succinic acid (6.1.4.) dissolved in 1 litre. as a stock solution. water adjusted to pH 4.0 with 1.0M sodium hydroxide (6.1.5) and 1 made up to 1 litre with water.

6.2.1.1 Succinate Buffer pH 4.0:
Take 250 ml succinate stock solution (6.2.1.) dilute to 800 ml with distilled water adjust to pH 4.0 with 1.0M sodium hydroxide (6.1.5) and make up to 1 litre with distilled water.

6.2.2. EDTA - Succinate Buffer:
As above but with 37.2 g EDTA (6.1.6) disodium salt added before adjusting pH and making to 1 litre with water.

6.2.3. 0.01M Oxalic Acid:
1.26 g oxalic acid (6.1.10) dissolved in and made up to 1 litre water.

6.2.4. HPLC Mobile Phase:
To 500 mL 0.01M oxalic acid (6.2.3) add 500 mL acetonitrile (6.1.9). Mix and pass through a 0.45 μM filter (5.2.9), assisted by vacuum (5.1.9). Further de-gas using ultrasonication (5.2.4) in conjunction with reduced pressure.

6.2.5. Copper Sulphate Solution:
Dissolve 5.0 g copper sulphate (6.1.13) in 1 litre distilled water.

6.2.6. b-mercaptopropionic Acid Solution:
Dissolve 0.5 g b-mercaptopropionic acid (6.1.14) in 10 mL methanol. (6.1.7)

6.3. Column Preparations

6.3.1. Chelating Sepharose

Thoroughly mix the suspension of chelating sepharose (6.1.11). Take an approximately 5 mL aliquot and place in a 200 mm x 20 mm i.d. glass column (5.1.2), allow to settle (bed height 1.5 cm) and remove excess liquid. Wash column with a further 15 ml water. Pour 2 x 10 mL of copper sulphate solution (6.2.5) through the column, followed by 15 mL of succinate buffer (6.2.1.1.).

NB Vortex mix column after first 10 mL copper sulphate solution to ensure even coating. After use columns are rinsed with 15-20 mL water. They may be stored in 20% aqueous ethanol at ambient temperature overnight or refrigerated for longer periods. Before use the aqueous ethanol is drained from the column and the column reloaded with copper sulphate solution and the cycle continued as before. If channels form in the sepharose bed the column should be vortex mixed to ensure even dispersion before loading samples.

6.3.2. Amberlite XAD-2

Take an aliquot of an aqueous slurry of resin (6.1.12) and place in glass columns 200 mm x 20 mm (i.d.) (5.1.2), pack to a bed height of 10 cm. Resin is prepared by washing in sequence with 100mL acetone (6.1.15), 100 mL methanol (6.1.7) and 200 mL water. Columns should be stored in acetone prior to use.. Re-disperse the column packing by inverting the column several times to ensure even distribution. Remove excess liquid before use.
(After use, columns can be regenerated by the same procedure).

7. STANDARDS

7.1. Tetracycline Standards:

Solutions of tetracyclines (6.1.1/2/3) should be stored in refrigerators. Stock solutions should be made fresh weekly and working standards on day of use. The procedures to be used when handling and preparing standards are given in the Food Science Laboratory Quality Manual CM 2 section 10.

7.2. Stock Standard (1 μg/μL):
Add 20 mg tetracycline standard to 20 mL methanol (6.1.7).

7.3. Intermediate Standard (10 ng/μL):
Take 1 mL of stock (7.2), dilute to 100 mL with water.

7.4. Working Standard 1 ng/μL):
Take 1 mL of 10 ng/μL (7.3), dilute to 10 mL.

7.4.1. Spiking solutions should be prepared in water.

7.4.2. Hplc standard (1ng/μL) prepared in hplc mobile phase.
Take 5 mL of intermediate standard (7.3), dilute to 50 ml.

8. PROCEDURE (*Revisions for fatty samples below)

8.1. Extractions should be performed on batches of up to 8 samples per day.

8.2.* Weigh 10 g finely sliced tissue or take 10 mL milk and add 100 mL succinate buffer (6.2.1.1.) into a centrifuge bottle (5.2.8). Place in an ultrasonic bath (5.2.4) (approx 3 mins). Homogenise (5.2.1) (approx 2 min) and centrifuge (5.2.3) (approx 5 min) at 14,00 r.c.f.

8.3.* Filter supernatant through Whatman 541 filter paper (5.2.6) and load on to a prepared chelating sepharose column(6.3.1).

8.4. Allow sufficient time for the filtrates to absorb into the sepharose gel then wash the column in sequence with 10 mL water, 30 mL methanol(6.1.7) and 2 x 10 mL water (Flow rate not to exceed 2mL/min.)

8.5. Pass 40 mL EDTA-succinate buffer (6.2.2) through the column followed by a further 10 mL EDTA-succinate buffer. Wash column with a further 2 ml water than collectt and combine both fractions.

8.6. Load the EDTA-succinate fractions directly onto a prepared XAD resin column (6.3.2). Allow the extract to absorb onto the resin.

8.7. Pass 2 x 100 mL water through the column and discard eluate. Pass 100 mL methanol (6.1.7) (4 mL/min) through the column. Discard the first 10 mL of eluant, collect remainder.

8.8.* Reduce methanol to small volume by rotary evaporation and quantitatively transfer extract to a pear shaped flask (5.1.5)with 3 x 2 mL methanol (6.1.7).

8.9.* Add approx 50 μL 5% ß-mercaptopropionic acid solution (6.2.6) to residue and remove methanol by rotary evaporation (5.2.5) (temperature 40 ± 5°C). The addition of acetonitrile at this stage will assist evaporation.

8.10.* Redissolve residue in 0.5 mL HPLC mobile phase (6.2.4). Care should be taken to ensure complete recovery of sample residue. Both vortex mixing (5.2.2) and ultra sonication (5.2.4) should be used to ensure residue is dissolved. Transfer extract to autosampler vial (5.1.8).

8.11*. HPLC is performed on 10 μL of extract.

Revised Procedure for fatty samples (corned beef)

8.2.1 Weigh 2 g finely sliced tissue and add 100 mL succinate buffer (6.2.1.1.) than as 8.2.

8.3.1 Filter 50 ml of the supernatant through Whatman 541 filter paper (5.2.6) and load on to a prepared chelating sepharose column(6.3.1).

8.8.1 Reduce methanol to small volume by rotary evaporation than add acetonitrile (5 - 10 ml) to aid evaporation to dryness. Quantitatively transfer extract to 10 ml tapered test tube with 4 x 2 mL (5.1.10) (acetonitrile (6.1.9).

8.9.1 Place test tubes in an aluminium dri-block (5.2.10) (temperature 40 ± 5°C) and evaporate acetonitrile under a stream of nitrogen.

8.10.1 Redissolve residue in 0.2 mL HPLC mobile phase (6.2.4) than as 8.10.

8.11.1 HPLC is performed on 20 μL of extract.

9. INTERPRETATION OF CHROMATOGRAPHIC DATA

9.1. Identification of analyte.

Identification of tetracyclines is by comparison of the retention times of the standard compound, either as reference standards or in spiked samples, with those of any similar peaks in the sample. Identification is accepted if peak retention time and shape are similar to the standard or spiked sample.

If in doubt suspect positive samples may be overspiked with one of the standard tetracyclines to measure degree of co-chromatography.

Demeclocycline (or other tetracycline) can be used as an internal standard to establish relative retention times and as a measure of recovery.

9.2. Calculation of results.

9.2.1 Screening.

A minimum of 3 injections (10 μL) of standard tetracycline (1ng/1μL) are carried out to determine the retention time and average peak height (10 ng injection = 50 ng/g ppb) tissue concentration). Concentration of sample (ng/g or ppb) is given by -

$$\frac{\text{Peak height sample}}{\text{Peak height standard}} \times 50 \text{ (conc'n of standard as ng/g tissue standard conc'n)}$$

9.2.2. Positives.

All suspect positives must be repeated in duplicate. A full standard curve (5 standards injected in triplicate) over the estimated concentration range of the suspect samples should be constructed. The lower standard should be 0 and the upper 10% greater than the highest concentration estimated from the screening result. A correction for recovery must be made (see 9.2.2.1).

9.2.2.1. Correction for recovery.

Recovery of analyte is determined during re-analysis by including with each batch blank samples (minimum duplicate) spiked with tetracyclines at a similar level to the sample estimated from screened result. The concentration in the sample should be adjusted by multiplying by 100 and dividing by X where X is the % mean recovery figure for that batch obtained from the spiked samples.

9.3. Reporting of data.

All qualitative and quantitative data must be recorded (see Food Science Laboratory Quality Manual CM 2 section 10). For final reports the ACTION LEVEL (concentration of analyte found in an individual sample) at which re-analysis of a sample is required will vary according to programme. In general surveillance programmes will have limits based on the maximum residue level (MRL) however all reporting levels must be confirmed with the Project and Technical Manager prior to commencement of analysis. Unless otherwise requested all results must be corrected for recovery. Reports must contain a clear statement as to whether results are corrected or uncorrected.

10. QUALITY ASSURANCE PROCEDURES

10.1. Validation of method prior to use.

When this method is to be used by an operator for the first time it must be fully validated for in house repeatability and reproducibility. The full procedure should be performed on one batch of samples on each of 3 separate days. Each batch will consist a known blank tissue divided into of a minimum of 5 blank samples. 4 samples are spiked at an appropriate level (set by the Project Manager) but will normally correspond to the 0.05 mg/kg level (500 μL of 1ng/1μL working standard 7.4). The remaining sample will be used as a reference blank.

A full validation must be carried out for any sample types, other than those specified in the introduction, and for which there is no previous validation records.

When returning to the method after a break the method must be validated on at least one single batch before proceeding. Recovery of analyte should fall within the range 50-110%. CV values should be less than 15% for inter and intra batch precision.

10.2. Quality Control of method for screening.

As a quality control reference each batch should always contain either :-

10.2.1. One sample spiked with tetracycline, chlortetracycline and oxytetracycline at the 0.05 mg/kg level. If recovery of analyte from the spiked sample falls outside the acceptable ranges (defined above 10.1.) then that batch is rejected.

Or

10.2.2. Demeclocycline (or other tetracycline) may be used as an internal standard in all samples as a measure of recovery during extraction. Recovery of demeclocycline from spiked samples should be no less than 40% and no greater than 110%. When recovery is found to be outside these ranges the batch should be rejected. Where a suspect positive is found there is a possibility that breakdown products (particularly from chlortetracycline) can interfere with the demeclocycline peak. Should this occur than a repeat analysis should be carried out using an alternative tetracycline (usually oxytetracycline) as the internal standard.

10.3 Quality Control of method for repeat analysis.

Two blank samples spiked with tetracycline, chlortetracycline and oxytetracycline at an appropriate level (see9.2.2.1). If recovery of analyte from the spiked sample falls outside the acceptable ranges (defined above 10.1.) then that batch is rejected.

10.4. Limit of Determination.

The quantitative limit of determination is taken to be the lowest level at which the procedure was validated with spiked samples. This may vary between sample types but in both offal and muscle a level of 0.010 mg/kg has been demonstrated for tetracycline, chlortetracycline and oxytetracycline.

Cover and Contents Page for an SOP (Example)

Method Number:-
Issue Number:- 5
Issue Date:- 19-03-92
Last Issue Date:- 20-11-91

ANALYSIS OF PESTICIDE RESIDUES IN PRODUCTS OF ANIMAL ORIGIN

CONTENTS

Hypothetical Control Chart showing features calling for QA action on the basis of weekly analyses of "in-house" reference material

a: 4 consecutive points all decreasing (weeks 2-5)
b: 1 point outside 3 x SD from Mean (week 6)
c: 4 consecutive points all outside 1 x SD from Mean (weeks 7-10)
d: 5 consecutive points all below Mean (weeks 8-12)
e: 4 consecutive points all increasing (weeks 10-13)
f: 2 consecutive points outside 2 x SD from Mean (weeks 19-20)
g: possible tendency to negative bias (weeks 1-26)

Appendix 7.3.1

Authorisation to use an SOP (Example)

AUTHORISATION/ACCEPTANCE RECORD SHEET

Project: VETERINARY DRUG RESIDUE - ANALYSIS

Method: _____
(BLOCK CAPITALS)

Name of Officer: _____
(BLOCK CAPITALS)

I, the Technical Manager, state that the officer mentioned above has had the appropriate training to perform the above method, has been tested to my satisfaction on this method and, in my view, is fully conversant with and able to perform the above method to the standards laid down in the Laboratory Quality Manuals.

Name of Project Technical Manager: W.H. FARRINGTON

Signature: _____ Date: _____

Review Date: _____

I state that I have been trained in the method described above and consider myself at this time to be fully conversant with and able to perform the said method to the standards to which I have been trained, laid down in the Laboratory Quality Manuals to which I have access at all times whilst in the Food Science Laboratory. If, in the future, for whatever reason, I consider that I am not at that time fully conversant with the method or am not able to perform it to the standards to which I have been trained and/or those standards pertaining to the method in the Laboratory Quality Manuals or do not have access to the said Manuals, I will inform the Project Technical Manager as soon as possible.

Name of Officer: _____
(BLOCK CAPITALS)

Signature: _____ Date: _____

This sheet is to be kept with the appropriate Training Record, the Officer retaining a copy for his/her own records. The Quality Manager is also to be provided with a copy.

AUTHORISATION TO PERFORM ANALYSES.

APPENDIX 7.3.2.

ISSUE DATE
ISSUE NO

ISSUED BY

LISTING BY NAME

ANALYST	METHOD	SOP	REVIEW DATE
A	IVERMECTIN	2	5.6.93
A	TETRACYCLINES	1	10.12.93
B	BENZIMIDAZOLES	3	2.4.94
B	CHLORAMPHENICOL	7	10.2.94
B	IVERMECTIN	2	10.11.93
B	SULPHAQUINOXALINE	4	12.12.94
B	SULPHONAMIDES	8	1.2.95
C	BENZAMIDAZOLES	3	1.10.94
C	CHLORAMPHENICOL	7	3.12.93
C	SULPHONAMIDES	8	2.1.95
D	IVERMECTIN	2	5.6.93
D	TETRACYCLINES	1	10.12.93
E	BENZIMIDAZOLES	3	10.4.94
E	CHLORAMPHENICOL	7	6.11.93
F	IMIDOCARB	6	6.5.95
F	LASALOCID	5	10.4.94
F	SULPHAQUINOXALINE	4	3.5.94
G	TETRACYCLINES	1	10.12.93
H	BENZIMIDAZOLES	3	7.10.94
H	IMIDOCARB	6	8.5.95
H	LASALOCID	5	6.4.94

LISTING BY METHOD

METHOD	ANALYST	SOP	REVIEW DATE
BENZIMIDAZOLES	B	3	2.4.94
BENZIMIDAZOLES	C	3	1.10.94
BENZIMIDAZOLES	E	3	10.4.94
BENZIMIDAZOLES	H	3	7.10.94
CHLORAMPHENICOL	B	7	10.2.94
CHLORAMPHENICOL	C	7	3.12.93
CHLORAMPHENICOL	E	7	6.11.93
IMIDOCARB	F	6	6.5.95
IMIDOCARB	H	6	8.5.95
IVERMECTIN	A	2	5.6.93
IVERMECTIN	B	2	10.11.93
IVERMECTIN	D	2	5.6.93
LASALOCID	F	5	10.4.94
LASALOCID	H	5	6.4.94
SULPHAQUINOXALINE	B	4	12.12.94
SULPHAQUINOXALINE	F	4	3.5.94
SULPHONAMIDES	B	8	1.2.95
SULPHONAMIDES	C	8	2.1.95
TETRACYCLINES	A	1	10.12.93
TETRACYCLINES	D	1	10.12.93
TETRACYCLINES	G	1	10.12.93

Appendix 9.1

Some Suppliers of Certified Reference Materials and Standard Compounds

1. Certified Reference Materials

Community Bureau of Reference (BCR)
Commission of the European Communities
Rue de la Loi, 200
B - 1049 BRUSSELS — Tel: 2-295-3115
BELGIUM — Fax: 2-295-8072

International Atomic Energy Agency
Wagrainerstrasse 5
PO Box 100 — Tel: 431 2360
A-1400 VIENNA — Fax: 431 234564
AUSTRIA — Telex: 1-12645

National Institute for Standards and Technology
Standard Reference Materials Programme
Room 2014, Building 202
GAITHERSBURG, Maryland 20899 — Tel: 301-975-6776
USA. — Fax: 301-948-3730

Reference Materials Advisory Service
Laboratory of the Government Chemist
Queens Road
TEDDINGTON, Middlesex TW11 0lY — Tel: 81-943-7565
ENGLAND, UK. — Fax: 81-942-2767

National Research Council of Canada
Marine Analytical Chemistry Standards Programme
Division of Chemistry
Montreal Road
OTTAWA — Tel: 613-993-2359
CANADA K1A 0R6 — Fax: 613-993-2451

National Institute of Environmental Studies
Japan Environmental Agency
PO Yatabe
Tsukuba
IBARAKI, -305
JAPAN — Tel: 298-51-6111

2. Specialist Suppliers of Standard Materials

Prochem Ltd (Pesticide Residue Analysis)
PO Box 255
ST. ALBANS, Herts AL1 4LN Tel: 727-833847
ENGLAND, UK Fax: 727-43395

Prochem GmbH (Macro- Micro- and Trace- Element Analysis)
Postf 1246
D - 4230 WESEL
GERMANY Tel: 281-530081

British Greyhound Chromatography and Allied Chemicals
Grange Road West (Pesticides, PCB's, environmental conaminants)
BIRKENHEAD,
Merseyside L43 4XF Tel: 51-653-3232
ENGLAND, UK Fax: 51-638-1846

Supelchem Ltd (Environmental, pesticide, PCB's, mycotoxins,
Supelco Chromatography Supplies food related standards)
Shire Hill
SAFFRON WALDEN,
Essex CB11 3AZ Tel: 799 513288
ENGLAND, UK. Fax: 799 513283

Supelco
Supelco Park
BELAFONTE, Pa, 16823-0048 Tel: 800-2476628
USA Fax: 814-3593044

Appendix 10.1

Proficiency Testing Schemes: Contents of the Joint ISO/AOAC International/IUPAC Harmonised Protocol

Appendix 11.1

Example of an Analytical Worksheet

PRODUCT:

Lab No: Manufacturers Code: Date:

Nett WT (grms):

Description:

Analyses to be Performed:

Determinand	Control	Result (1)	Result (2)	Average
Starch (%)				
Fat (%)				
Moisture (%)				
Ash (%)				

Inferences

Name of Analyst: Signature: Date:

Name of Supervisor: Signature: Date:

FAO TECHNICAL PAPERS

FAO FOOD AND NUTRITION PAPERS

1/1 Review of food consumption surveys 1977 – Vol. 1. Europe, North America, Oceania, 1977 (E)
1/2 Review of food consumption surveys 1977 – Vol. 2. Africa, Latin America, Near East, Far East, 1979 (E)
2 Report of the joint FAO/WHO/UNEP conference on mycotoxins, 1977 (E F S)
3 Report of a joint FAO/WHO expert consultation on dietary fats and oils in human nutrition, 1977 (E F S)
4 JECFA specifications for identity and purity of thickening agents, anticaking agents, antimicrobials, antioxidants and emulsifiers, 1978 (E)
5 JECFA – guide to specifications, 1978 (E F)
5 Rev. 1. JECFA – guide to specifications, 1983 (E F)
5 Rev. 2. JECFA – guide to specifications, 1991 (E)
6 The feeding of workers in developing countries, 1976 (E S)
7 JECFA specifications for identity and purity of food colours, enzyme preparations and other food additives, 1978 (E F)
8 Women in food production, food handling and nutrition, 1979 (E F S)
9 Arsenic and tin in foods: reviews of commonly used methods of analysis, 1979 (E)
10 Prevention of mycotoxins, 1979 (E F S)
11 The economic value of breast-feeding, 1979 (E F)
12 JECFA specifications for identity and purity of food colours, flavouring agents and other food additives, 1979 (E F)
13 Perspective on mycotoxins, 1979 (E F S)
Manuals of food quality control:
14/1 Food control laboratory, 1979 (Ar E)
14/1 Rev. 1. The food control laboratory, 1986 (E)
14/2 Additives, contaminants, techniques, 1980 (E)
14/3 Commodities, 1979 (E)
14/4 Microbiological analysis, 1979 (E F S)
14/5 Food inspection, 1981 (Ar E) (Rev. 1984, E S)
14/6 Food for export, 1979 (E S)
14/6 Rev. 1. Food for export, 1990 (E S)
14/7 Food analysis: general techniques, additives, contaminants and composition, 1986 (C E)
14/8 Food analysis: quality, adulteration and tests of identity, 1986 (E)
14/9 Introduction to food sampling, 1988 (Ar C E F S)
14/10 Training in mycotoxins analysis, 1990 (E S)
14/11 Management of food control programmes, 1991 (E)
14/12 Quality assurance in the food control microbiological laboratory, 1991 (E)
14/13 Pesticide residue analysis in the food control laboratory, 1993 (E)
14/14 Quality assurance in the food control chemical laboratory, 1993 (E)
15 Carbohydrates in human nutrition, 1980 (E F S)
16 Analysis of food consumption survey data for developing countries, 1980 (E F S)
17 JECFA specifications for identity and purity of sweetening agents, emulsifying agents, flavouring agents and other food additives, 1980 (E F)
18 Bibliography of food consumption surveys, 1981 (E)
18 Rev. 1. Bibliography of food consumption surveys, 1984 (E)
18 Rev. 2. Bibliography of food consumption surveys, 1987 (E)
18 Rev. 3. Bibliography of food consumption surveys, 1990 (E)
19 JECFA specifications for identity and purity of carrier solvents, emulsifiers and stabilizers, enzyme preparations, flavouring agents, food colours, sweetening agents and other food additives, 1981 (E F)
20 Legumes in human nutrition, 1982 (E F S)
21 Mycotoxin surveillance – a guideline, 1982 (E)
22 Guidelines for agricultural training curricula in Africa, 1982 (E F)
23 Management of group feeding programmes, 1982 (E F P S)
23 Rev. 1. Food and nutrition in the management of group feeding programmes, 1993 (E)
24 Evaluation of nutrition interventions, 1982 (E)
25 JECFA specifications for identity and purity of buffering agents, salts; emulsifiers, thickening agents, stabilizers; flavouring agents, food colours, sweetening agents and miscellaneous food additives, 1982 (E F)
26 Food composition tables for the Near East, 1983 (E)
27 Review of food consumption surveys 1981, 1983 (E)
28 JECFA specifications for identity and purity of buffering agents, salts, emulsifiers, stabilizers, thickening agents, extraction solvents, flavouring agents, sweetening agents and miscellaneous food additives, 1983 (E F)
29 Post-harvest losses in quality of food grains, 1983 (E F)
30 FAO/WHO food additives data system, 1984 (E)
30 Rev. 1. FAO/WHO food additives data system, 1985 (E)
31/1 JECFA specifications for identity and purity of food colours, 1984 (E F)
31/2 JECFA specifications for identity and purity of food additives, 1984 (E F)
32 Residues of veterinary drugs in foods, 1985 (E/F/S)
33 Nutritional implications of food aid: an annotated bibliography, 1985 (E)
34 JECFA specifications for identity and purity of certain food additives, 1986 (E F**)
35 Review of food consumption surveys 1985, 1986 (E)
36 Guidelines for can manufacturers and food canners, 1986 (E)
37 JECFA specifications for identity and purity of certain food additives, 1986 (E F)
38 JECFA specifications for identity and purity of certain food additives, 1988 (E)
39 Quality control in fruit and vegetable processing, 1988 (E F S)
40 Directory of food and nutrition institutions in the Near East, 1987 (E)
41 Residues of some veterinary drugs in animals and foods, 1988 (E)
41/2 Residues of some veterinary drugs in animals and foods. Thirty-fourth meeting of the joint FAO/WHO Expert Committee on Food Additives, 1990 (E)
41/3 Residues of some veterinary drugs in animals and foods. Thirty-sixth meeting of the joint FAO/WHO Expert Committee on Food Additives, 1991 (E)
41/4 Residues of some veterinary drugs in animals and foods. Thirty-eighth meeting of the joint FAO/WHO Expert Committee on Food Additives, 1991 (E)
41/5 Residues of some veterinary drugs in animals and foods, 1993 (E)
42 Traditional food plants, 1988 (E)
42/1 Edible plants of Uganda. The value of wild and cultivated plants as food, 1989 (E)
43 Guidelines for agricultural training curricula in Arab countries, 1988 (Ar)

44	Review of food consumption surveys 1988, 1988 (E)
45	Exposure of infants and children to lead, 1989 (E)
46	Street foods, 1990 (E/F/S)
47/1	Utilization of tropical foods: cereals, 1989 (E F S)
47/2	Utilization of tropical foods: roots and tubers, 1989 (E F S)
47/3	Utilization of tropical foods: trees, 1989 (E F S)
47/4	Utilization of tropical foods: tropical beans, 1989 (E F S)
47/5	Utilization of tropical foods: tropical oil seeds, 1989 (E F S)
47/6	Utilization of tropical foods: sugars, spices and stimulants, 1989 (E F S)
47/7	Utilization of tropical foods: fruits and leaves, 1990 (E F S)
47/8	Utilization of tropical foods: animal products, 1990 (E F S)
48	Residues of some veterinary drugs in animals and foods, 1990 (E)
49	JECFA specifications for identity and purity of certain food additives, 1990 (E)
50	Traditional foods in the Near East, 1991 (E)
51	Protein quality evaluation. Report of the Joint FAO/WHO Expert Consultation, 1991 (E F)
52/1	Compendium of food additive specifications – Vol. 1, 1993 (E)
52/2	Compendium of food additive specifications – Vol. 2, 1993 (E)
52 Add. 1	Compendium of food additive specifications – Addendum 1, 1992 (E)
52 Add. 2	Compendium of food additive specifications – Addendum 2, 1993 (E)
53	Meat and meat products in human nutrition in developing countries, 1992 (E)
54	*Ex situ* storage of seeds, pollen and *in vitro* cultures of perennial woody plant species, 1993 (E)
55	Sampling plans for aflatoxin analysis in peanuts and corn, 1993 (E)

Availability: October 1993

Ar	–	Arabic	Multil	– Multilingual
C	–	Chinese	*	Out of print
E	–	English	**	In preparation
F	–	French		
P	–	Portuguese		
S	–	Spanish		

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This manual is a practical handbook on the establishment of a quality assurance (QA) programme for a food control chemical laboratory. Its ultimate aim is to ensure that a chemical laboratory produces reliable high-quality analytical results with a continuity of documentation providing a clear, accurate and indisputable history of the analysis, with all segments of the documentation in agreement. A similar QA manual for the microbiology laboratory was published as FAO Food and Nutrition Paper 14/12 in 1991.

The manual is designed for chemistry laboratory management and analytical staff, but regulatory authorities or any other concerned persons can gain useful information and insights into the problems involved in establishing and operating a quality assurance programme in a food control chemistry laboratory.

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