



SECOND EDITION

THE ICC HANDBOOK OF
**Cereals, Flour, Dough
& Product Testing**
Methods and Applications

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DEStech Publications, Inc.

The ICC Handbook of Cereals, Flour, Dough & Product Testing, Second Edition

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Preface

The International Association for Cereal Science and Technology (ICC)

FIRST called the International Association for Cereal Chemistry (ICC), ICC was founded in 1955 on the occasion of the Third International Bread Congress in Hamburg with its main objectives being to develop and standardize investigation methods for cereal grains and cereal-based products (ICC, 2015). Today these objectives remain at the centre of the ICC's functions to which has been added the development of international cooperation and the dissemination of information related to cereal science. A non-political, non-religious and non-profit-making organisation the ICC offices are based in Vienna, Austria, where the Secretariat is run by Ms Michaela Pichler.

ICC's strength lies with its excellent international network and contacts with cereal scientists in the many fields of study related to cereal grains. Since its inauguration the ICC has been at the forefront of method standardisation and new method development and now has over 200 standard methods, recommended and rapid methods. Through its working groups and international networks the ICC continues to examine and introduce new standard methods for application to cereal grains. In that task it works closely with other standardisation bodies, such as AACC (American Association of Cereal Chemists) International and other international bodies to work towards the harmonisation of existing and new methods.

A key role for the ICC has been and continues to be to provide a forum for the exchange of information and cooperation in the cereal science field. It does this through sponsored scientific meetings such as the International Cereals and Bread Congress (held every four years in

different parts of the world), through its involvement and promotion of many international conferences and workshops, and through its coordination and project-based activities. Important examples of the continued relevance of ICC are its significant involvement with the MoniQA Network of Excellence, previously coordinated by ICC and funded by the European Union with the aim of the international harmonization of analytical methods for food quality and safety assessment, the ICC-led Task Force on Mycotoxins and Sampling and the Healthgrain Forum.

For more information on the ICC, its activities, standard methods and publications visit: www.icc.or.at.

As part of its continuing involvement improving food quality and safety, the ICC has an agreement with Wageningen Academic Publishers for the regular publication of *Quality Assurance and Safety of Crops and Foods*. QAS, as the journal has become known, is available 6 times per year, and publishes research and review papers associated with the quality and safety of crops. It targets plant-based primary raw materials, their harvesting, storage and conversion to human foods.

QAS has a strong focus on the development and application of new analytical tools and their potential for quality assessment, assurance, control and safety. The scope also includes issues of risk assessment, traceability, authenticity, food security and socio-economic factors.

For more information and to subscribe to QAS go to: www.wageningenacademic.com/journals/qas.

Reference

ICC (2015) *A short history of the ICC*, compiled by S. Cauvain, R. Cracknell and R. Poma, ICC, Vienna, Austria

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Introduction to Testing Methods and Their Applications

STANLEY P. CAUVAIN

THE cereal taxonomic family comprises wheat, triticale, rye, barley, oats, rice, maize, sorghum and millets. Within each member of the cereal family there are many species and varieties which have found use as both human and animal food. New varieties are being bred and added to the list on a regular (annual) basis. Many of the new varieties are designed to deliver improved and very specific characteristics for agricultural purposes. Such characters as disease resistance, stress resistance (e.g. drought resistance) and yield are high on the list of desirable properties for the cereal breeder. Equally important are the end-use performance characteristics of the cereal grain but they have probably received less attention from breeders and growers than the agricultural traits until relatively recently in the development of new cereal varieties. In part this is because much of the historical emphasis on developing new cereal varieties has been driven by the need to increase the basic food supply and this still remains a key requirement in many parts of the world. However, in areas where the supply of cereals exceeds the demands of basic nutrition there is inevitably more opportunity to place greater emphasis on delivery of those quality traits which are more closely linked with the quality of the end product.

As cereals are traded on a global scale the needs for appropriate and standardised quality testing methods are significant. Historically the evaluation of grain was carried out in a largely subjective manner. Obvious problems of grain dampness and spoilage would have been relatively easy to see and agree (hopefully amicably) between supplier and user. There is no doubt that the experienced miller and baker could make some sort of prediction about milling and baking quality with a form of crushing test—probably achieved by chewing a small sample of the grain or a small test milling. In this scenario agreement between supplier and user as to the ultimate performance of cereal grain and the flour obtained from it would certainly have been harder to achieve.

Even today in some parts of the world farmer, miller and baker or brewer can have diverging opinions on the quality attributes of grains grown in a particular location because, despite the advances of cereal science, it remains hard to predict the ultimate performance of the basic raw material. In part this is because of the wide variety of uses to which cereal grains are put by humankind. Not only do they provide food for animals but also food and drink for humankind (more recently cereal grains are being viewed as a source of fuel energy though such uses are outside the scope of this book). With such a diversity of end-uses and potential products it is inevitable that different testing methods have evolved to assess the quality of the raw material and it is with this complexity in mind that the concept of this book developed.

The objective of the contributions in this book is look closely at the applications of cereal testing methods from the differing points of view in the 'grain chain', starting with the cereal breeder and ending in the bakery, brewery and animal feed compounder. Each contribution considers a range of available testing methods for their applicability in a given set of circumstances. It is not the intention of the work to identify the 'best' or the 'right' way to test cereal grains in a particular environment rather, to consider what is available and how it might be applied. The same cereal test may well be referred to in several places throughout this book and this reflects the way in which the determination of a particular cereal grain characteristic may be used in different ways in different parts of the grain chain. It is also not the intention of contributors to be unduly critical of existing methods though it is inevitable that there will be recognition that nature of the results from some existing testing methods may still require a high degree of expert interpretation to make them genuinely 'useful'.

It is in the nature of cereal science that research and technology change the type of information that we require and so it is also inevitable that new methods will evolve for the testing of cereals. The challenges facing the cereal scientist when evolving new testing methods are significant. For example, consider the manufacture of bread in which the special properties of the gluten developed during dough mixing are readily appreciated when the dough is mixed and processed by hand. The baker can feel the changes which occur as first the proteins hydrate and the gluten network gradually begins to develop and change. Dough consistency and properties of elasticity and extensibility could be readily evaluated using fingers and with experience the rheological properties of the dough could be linked with the final food product. So ingrained is this need to evaluate gluten quality in dough that even today experienced bakers working in large automated plant bakeries will take the opportunity to take a small piece of dough to feel and stretch. They can always tell you whether the dough feels 'right' or 'wrong' but when pressed can seldom tell you what it is they are assessing or looking for.

It was inevitable that the application of science to the natural world during

the 19th century would lead to studies on the processing and use of grains and these examinations form the basis of the cereal testing methods that we know today. In developing objective methods for the evaluation of grains and the flours produced there were a number of clear, mainly chemical, tests which could establish quality characteristics. Less clear would be how to define those characteristics that related mainly to the performance of the flour in the ultimate manufacturing environment, e.g. the bakery or brewery. In this case a series of mainly physical tests evolved many of which were related in some way to more abstract sensory properties, for example gluten quality.

Historically the standardisation and publication of testing methods suitable for application to grains has been carried out by organisations like the International Association for Cereal Science & Technology (ICC), the American Association of Cereal Chemists (now AACC International), the American Oil Chemists Society (AOCS) the International Organisation for Standards (ISO) and national standards bodies. However, while the publication of such standardised methods has ensured their dissemination around the world there is no collective work which considers the applications of cereals testing methods to the various stages of the transition from wheat grain to final product. It is important to recognise that many of the international methods used today started out as a 'locally' preferred means of evaluating grain and products quality before attaining international acceptance. Often there is no perfect predictive test for cereals at any stage of processing and it is inevitable that new ideas will be introduced in an effort to improve our ability to manufacture foods from grains. By including new ideas in this book we hope that we will continue to stimulate the development of new testing methods, some of which may eventually make the transition to that of approved 'standard method'.

The Reliability of Measurements

In order for analytical data to have value users must be confident that the values that they are given have both relevance and meaning for their particular needs. Confidence in analytical data comes from using accredited service providers with methods developed and approved by appropriate national and international bodies. As discussed above the ICC has been pre-eminent in the development and publication of standard methods relevant to the use of cereal grains and many of the standard methods will be referred to in subsequent chapters.

Confidence in measured values comes with an understanding of what is actually being measured and the reliability of the data. Many analytical techniques used in cereals-related testing do not measure fundamental properties of materials, or indeed use fundamental units. In the cereals industry the mea-

surement of protein is one such example in that analytical techniques measure nitrogen and interpret the value in terms of protein with two different factors, one for human and one for animal feed. It is also very important to recognise that no analytical measurement is without error and there are a number of potential sources of errors which need to be considered when making comparison between the materials being tested and the methods being used.

In order to help the reader in the discussions which follow in subsequent chapters some relevant definitions are given which attempt to place measured values which may be quoted into their appropriate analytical and applications contexts.

Expected Standard Uncertainty

This term is associated with analytical validation and the performance of a particular method. Rose et al (2011) considered that “The standard uncertainty associated with a method is a single parameter that gives an estimate of the combined effect of the individual factors that describe the method on how far we can expect a measurement result to lie from a true concentration”.

Accuracy

This term refers to the precision of data or closeness of estimates to the exact or true values of the property that was measured and as such it reflects the degree to which the information provided correctly describes the phenomena that is was designed to measure. It includes providing the results of the assessment of source data for sampling and non-sampling errors and may be decomposed into bias (systematic error) and variance (random error).

Measurement—Sources of Error

There are a number of potential sources of error in analysis. These include those associated with sampling, sample preparation and analytical error. Obtaining ‘relevant’ samples for testing is one of the major challenges facing the grains industry.

Reliability

In statistical terms reliability is viewed as the closeness of the initial estimated value(s) to the subsequent estimated values.

Repeatability/Reproducibility

These two terms are sometimes viewed as having the same meaning in statistical terms; being defined as ‘The concept that survey procedures should be repeatable from survey to survey and location to location; the same data

processed twice should yield the same results'. In cereal testing the two terms have tended to be given slightly different, though clearly related meanings.

Repeatability

Repeatability is a measure of the ability of single instrument to give the same reading on the same sample at different times of testing.

Reproducibility

Reproducibility measures the degree of agreement in readings between different laboratories using the same type of instrument on the same samples.

Standard Error

Defined as 'The positive square root of the variance of the sampling distribution of a statistic' it includes the precision with which the statistics estimate the relevant parameter as contrasted with the standard deviation that describes the variability of primary observations.

Ring and Proficiency Testing

In the establishment of an accepted standard method it is important that the method is robust, reliable and able to deliver meaningful results. A key part in the establishment of a standard method is the conducting of a 'ring test'. This requires collaboration between a number of laboratories, each of which is equipped to carry out the analysis concerned. The participating laboratories have access to appropriate samples from an agreed central source. After the various analyses have been completed the individual results will be collated and analysed to provide relevant precision data which usually become part of the standard method. If there are problems in agreeing the precision of the data it may be necessary to carry out further ring tests with fresh samples.

The principles and processes associated with the statistical analysis of results from collaborative studies in ring tests are discussed in detail in ICC Recommendation No. 203.

The establishment of a standard method is not the end of the analytical process. Laboratory expertise may vary with time through changes in laboratory personnel and equipment performance. In order to ensure that data from a given laboratory remain accurate and relevant it is common practice to engage in some form of proficiency testing using centrally supplied materials (e.g. AACC International Check Sample Series).

Sampling

The evaluation of a batch of any raw material, processed material or product relies on the evaluation of small numbers of samples being taken from the batch and the assumption that the sample will be representative of the batch.

Thus, sampling is one of the most, if not the most critical step associated with testing. The application of sampling practices is discussed in more detail in subsequent chapters, here it is just necessary to introduce the concept and comment in general and some of the key issues and practices.

Laboratory Data

The largest part of grain evaluation and testing data will be obtained through measurements made off-line in the laboratory where trained staff and carefully controlled environments can be found. These conditions ensure a high degree of precision and reliability associated with the measurements. These are ideal circumstances in which to carry out testing for quality control and research purposes though it should always be borne in mind that such evaluations are not always being made under the conditions in which the material will be used. This is especially true for flours which are utilised in baking processes.

The value of testing data for quality control purposes is to ensure that the raw material has been manufactured to an agreed specification. This should not be confused with the prediction of the ultimate performance in a given manufacturing process. The specification should have been previously agreed on the basis of understanding that the raw material will deliver the required final product quality and what variations to a given parameter value can be tolerated. To achieve this situation a common understanding between supplier and user is required.

In the research environment laboratory data are commonly used to investigate relationships between raw material properties and processing requirements and final product quality. Laboratory-based research studies offer opportunities for insights which can improve process efficiencies, optimise final product qualities and lead to new developments. Once again it is not common to carry out such studies under typical processing conditions and care must be taken over the interpretation of data with respect to practical conditions.

At-line and On-line Measurements

In view of some reservations expressed above with respect to laboratory-based data there appears to be a powerful case for measurements to be made at the production line, ideally on-line. These are not easy tasks for a number of reasons.

Firstly, many of the measurements require highly specialised equipment not best suited to the environments which are typical of many processing sites. Often the tests involve the passage of some period of time before the results are available and so do not lend themselves to providing data which might be used in 'real time' to adjust process recipes or operating conditions; there are a few exceptions which are further discussed below.

Perhaps the major drawback for at-line or on-line testing remains the appli-

capability of the testing method. As noted above (and will be discussed in detail in later chapters) many cereal analytical methods are ideally suited to quality control purposes. Historically cereals testing methods have (with a few exceptions) been based on the analysis of some core chemical properties or a form of ‘quality’ evaluation using a mixture of flour and water. The latter includes possibly the easiest way to assess flour protein content and quality but few wheat flour-based products comprise a simple mixture of flour and water.

Thus far the most effective on-line measurements in the grains industry has been the application of near infra-red (NIR) measurements to wheat and the flour produced from it. Indeed many wheat flour ‘certificates of analysis’ are based purely on NIR data. However, a cautionary note is that many flour properties included in the output from NIR measurements have been calibrated against some form of standard reference which in themselves are not robust fundamental measurements and therefore the data can be prone to misinterpretation. A note to ‘Use with care’ should be included in the assessment of the data, at least in the users mind if not on the C of A.

The development of increasingly sophisticated image analysis techniques does in the case of final products provide opportunities for true on-line measurement and data collection. Bread height, hamburger bun shape and crust colour are now relatively easy to achieve on-line, at least in larger industrial bakeries. The challenge (further discussed below) is to how to link process data with raw material analysis.

Test Baking and Similar Small-scale Quality Assessments

The link between raw material qualities and final products has been traditionally established through test baking or by mimicking key processes on a small scale. Many different test baking methods exist which for example reflect the variety of breadmaking methods employed around the world (Cauvain, 2015a). Test baking plays a significant role as a potential quality control function and a research tool but given the wide variety of baking methods employed cannot be seen as an absolute predictor of bread making performance. For example, evaluating the breadmaking potential of wheat flour using a pan bread test does not necessarily reflect its potential for baguette production. Major challenges of using small-scale assessments such as test baking are associated with the problems of scale-up and batch processing times. Never the less small-scale quality assessments often come closest to predicting performance in ultimate processing. They are of course, time-consuming and should be carried out in a controlled environment such as a laboratory.

Using Test Data

As noted above, common uses of test data include the facilitation of grain trading and quality control functions. In many cases the use of test data as a

means of predicting ultimate performance relies on the ‘expert’ interpretation of the data. This is a perfectly reasonable approach as human beings are good at ‘pattern matching’. Essentially an expert will view the pattern of information in the analytical data of a raw material, or groups of raw materials, and match this to performance patterns based on their experience. Commonly experts develop a set of heuristic rules (sometimes referred to as rules of thumb) which enable them to predict the direction of change without necessarily being able to quantify the magnitude of change. A simple example, could be the increase in the protein content of a wheat flour which would be expected to lead to an increase in bread volume or height while recognising how great the increase will be depends on the dough recipe and processing conditions, such as mixing.

The adjustment of recipe and processing conditions to compensate for variations in raw material qualities has traditionally been the province of the expert or less reliably, has been determined by ‘trial and error’ during processing. With the increasing industrialisation of processes such as breadmaking tolerances for variations for product quality become smaller and the need for stronger and more reliable processing models linking raw materials and product qualities increases. Inevitably this leads to the consideration and application of mathematical models.

A specific problem for the development of mathematical models for grain processing technologies is the large number of interactions that occur in the transition from grain to final product. This is particularly true of breadmaking where final product quality depends on a myriad of ingredient-recipe-process interactions. Given the complexity of grain processing to final product, it is not surprising that mathematical modelling has tended to concentrate on narrow sections of the transition; that way they are easier to model but the challenge remains that such narrow models still have to be integrated into a more holistic model which could describe the whole process.

Gathering and Using Data in a Commercial Context

The significant advances in computing power has made the capture of data in commercial environments an increasingly feasible option. Today many pieces of equipment used in commercial operation have the capacity to continuously record and the potential to download data which are important in commercial operations. One such example is the recording of individual dough piece weights in commercial bread bakeries, in some cases to ensure that final products will meet any point-of-sale legislation but in all cases to optimise dough yields from a given batch and process efficiencies in general.

While process data may be gathered on a regular basis it is not necessarily true that it is stored and used effectively in commercial contexts. Part of the challenge of gathering data in a commercial context is the sheer quantity that

becomes available and the need for large data storage capacities; until recently it has not been common practice to even consider the provision of data storage, let alone detailed and integrated analysis.

If the data storage capacity is available the next issue to consider is what data to collect. Many cereal processing systems are batch fed even if the subsequent processing is continuous (e.g. wheat bins to the break rolls or dough batch to the dough divider). Also a significant period of time elapses from the moment the raw material first enters the process to the moment that the final product leaves. This requires that data will need to be related to a fixed time line in order to facilitate effective analysis. For example, the rheological properties of a batch of dough may be assessed some 2-4h before the subsequent bread qualities can be measured.

The analysis of very large data batches very quickly has now become possible as a result of the increases in computing power. Specialist analysis programs do exist in non-food industries which allow the visualisation of complex industrial processes on a continuous time related basis though there are few instances of their application to cereals grain processing. With increased automation of industrial scale processing their application will increase.

Future Challenges for Testing Methods and Their Application

The challenges facing the development of new testing methods, improving existing methods and their application are many.

They include the following:

- The continued development of rapid testing methods, especially in the area of food safety. Examples of possible applications are discussed in subsequent chapters.
- Improvement to the limits of detection and the reliability of measurements. Again, many of these will be related to food safety but in some cases such improvements can be a 'double edge sword' so that as the limits of detection are pushed ever lower, questions over what are 'safe limits' for contaminants become increasingly important.
- Increasing development and application of at-line and on-line testing methods designed to be used in the field prior to or at harvesting, as well as subsequent grain storage and processing to final products.
- Opportunities for on-line analysis which can be linked with improved process control to improve process efficiency, reduce energy requirements and minimise waste.
- The development of material testing methods with greater predictive ability, commercial relevance and practical applications. In the laboratory this may, for example, be associated with a move towards using more commercially relevant recipes while in the production environment

this may be the development of easy-to-use methods for plant operatives. A number of tests already exist which can be used to assess final product quality in a plant environment but their application is limited and because they are carried out at the end of the manufacturing process they do not provide information for real-time control of manufacturing operations.

- Improvements to predictive models which link raw materials qualities with recipe and processing information. For grain-based processed foods this remains a significant challenge because of the large number of complex interactions involved but equally the potential rewards are significant.

Food Safety and Quality Systems

Cereals food quality may be encompassed under two general headings; technical quality which is aimed at delivering a consistent product that consumers will enjoy and products that are safe to eat and will not cause consumers any harm. When references are made to quality systems the most common expectation is that of ensuring food safety though the principles by which manufacturers deliver safe food may be equally be applied to the delivery of products of consistent quality and as such may be addressed using commonly applied systems such as Hazard Analysis Critical Control Points (HACCP) and Good Manufacturing Practice (GMP).

HACCP (Wallace and Mortimer, 2000) is extremely effective as a tool in delivering safe food products and is based on identifying and assessing potential hazards and then establishing control systems that implement preventative measures. This focus on preventative measures is designed to stop hazards entering the food chain rather than relying on time consuming and expensive end-product testing. The implementation of HACCP which looks at individual stages of the food processing chain still requires that food safety checks by way of raw material and sample analysis be carried out. A key practical benefit of implementing HACCP can be a significant reduction in raw material and product testing but it does not eliminate risk. Regular, on-going reviews of HACCP should be undertaken, not least in response to potential new hazards.

GMP (Manning, 2013) is commonly used alongside HACCP with perhaps a greater emphasis hygienic practices in relation to the handling of raw materials and their processing into food. Eliminating the potential for food spoilage microorganisms is of course, as much about delivering safe food as the elimination of foreign bodies and chemical contaminants. Where perhaps GMP and HACCP both differ and overlap is the nature of the hazard which is being assessed and the measures which need to be put into place to limit or eliminate

that hazard. In addition to GMP one sees Good Agricultural Practices (GAP) and Good Hygienic Practices (GHP).

HACCP, GMP and the like are increasingly being used in international trade and the movement of raw materials, intermediates and final products. Such procedures are essential in the global nature of food market places today and this may mean that standards applied in one part of the world are demanded in another where the interpretations of 'acceptable' practices differs from that of the final market place. This is particularly true for the many major food retailers that directly provide food to consumers. In some cases the standards which the retailer imposes on their supplier may even exceed those which apply locally or in a specific region. In some ways one may argue that major retailers have become guardians of public health, not least because in these days of instant information exchange food retailers cannot afford the negative publicity which food safety scares quickly attract.

There are other techniques which can be applied to delivering efficient process control and which will make a significant contribution to consistent production. In this short introduction it is only the intention to provide brief comments on the overall concepts involved food quality systems. An all-embracing term which might be applied to processes involved in the provision of consistent quality and safe food might be Total Quality Management (TQM). Many of the tests, techniques and procedures which will be discussed in detail in the following chapters form the basis of the tools which are essential to the operation of TQM but to be effective they need to be used in a structured environment. While their application to raw materials and finished products are commonly seen as the responsibility of the Quality Control or Technical Departments, their effective implementation is the responsibility of all those involved in food production. It is now common to see statements which describe Quality Systems in terms of "farm to fork" and "plough to plate". Such descriptions are intended to show the need for integration of quality systems from the growing of raw materials right to the arrival of the final product with the consumer. However, a critical examination of many such systems shows (rightly) a strong emphasis on food safety, though perhaps without the same rigorous consideration of technical manufacturing constraints and final product. Ultimately you can have the safest food in the world but if it does not also have the consistent qualities sought and purchased by consumers it has no value to anyone involved in the food production chain.

In much the same way that HACCP seeks to eliminate or minimise the risks of food safety TQM seeks to eliminate or reduce risks associated with product quality defects; i.e. loss of quality attributes which would mean that the product does not reach consumer expectations. In modern manufacturing environments high productivity at minimum costs and minimal (zero) defects are the ideal. However, meeting those manufacturing expectations are not so

easy when dealing with ‘natural’ raw materials. No matter how well controlled agricultural practices are, the impact of geography and the environment will play a major role in determining the suitability of raw materials for processing into human food. In this context the cultivation of wheat is a classic example of the challenges facing growers and users of the raw material. Each harvest delivers a raw material which will be subtly different from the previous harvest and so significant attention has to be paid to the assessment of raw material in the sense of its suitability for end use.

To achieve this quality testing methods have to be put into context with the recognition that wheat will be used to make a wide range of products each with its specific quality characteristics and employing a wide range of processing methods. Even after many years of study an inescapable conclusion is that there is no single quality testing method which can predict sufficiently well the performance of a given variety or parcel of wheat in the food factory. Most of the quality data which is gathered during wheat and flour testing requires further interpretation by human experts to ensure that concepts like TQM are met. It has been argued that wheat and flour testing data are at best indicative of end performance rather than predictive (Cauvain, 2015b). Some of the potential challenges for cereals testing methods and their application have been noted above.

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Further Information on Testing and Standards

There are a number of internationally recognised bodies engaged in the development of cereal testing methods, they include:

The American Association of Cereal Chemists—AACC International—www.aaccnet.org

The American Oil Chemists Society—AOCS—www.aocs.org

The International Association for Cereal Science and Technology—ICC—www.icc.or.at

The International Standards Organisation—ISO—www.iso.org

Secretariat of the Codex Alimentarius Commission, Joint FAO/WHO Food Standards Programme—www.codexalimentarius.net

European Food Safety Authority—www.efsa.europa.eu

Moni QA—www.moniqua.org

There are also national bodies who are also engaged in such activities (e.g. BSI in the UK, CEN in France) and they will be linked to an appropriate international body, commonly ISO.

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