

## ANALYTICAL QUALITY ASSURANCE AND METROLOGICAL TRACEABILITY OF MEASUREMENT DATA IN FOOD CONTROL ASSESSMENT: ADVANCES IN ANALYTICAL SYSTEMS

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**Abstract** – Innovative analytical approaches are being devised in response to emerging food safety and quality issues, as well as for food authenticity and traceability testing.

Traceability fosters quality assurance as a drive for analytical reliability: to this aim, validation of analytical processes and metrological comparability of measurements are required. Accreditation practices and implementation of quality assurance programs improve the quality of analytical results in food control.

**Keywords:** food safety, food authenticity, analytical testing, metrological comparability of analytical results, quality assurance.

agencies and consumers. Food processing industries in developing countries have to face problems with timely delivery of raw material, as well as variations in overall quality due to a lack of infrastructure in the producing areas. This calls for greater vigilance by the food processing units and for food control activity to be implemented at all stages along the food supply chain. In addition, with the emergence of food safety challenges and new health threats posed by contaminants and residues in foods, food industry groups are establishing programs to reduce risk factors and innovative cutting-edge analytical methods that are capable of detecting harmful substances are increasingly becoming a centerpiece of food safety programs.

### 1. INTRODUCTION

Effective food control systems are essential to protect the health of consumers and are also vital in enabling countries to assure safety and quality of food products for international trade. As for the legal framework for food safety in the European Union Member States, in 2000 the EU launched the *White Paper on Food Safety* to create a transparent set of rules for safe food and animal feed production and food control [1]. To assure that the control systems at national level are effective, the Health and Food Audits and Analysis Directorate of the Directorate-General for Health and Food Safety (DG SANTE) of the European Commission carries out inspections in EU Member States and in non-EU countries exporting to the EU to evaluate compliance with EU standards. To coordinate food standards activities undertaken by international organizations and to ensure fair practices in the food trade, the Codex Alimentarius Commission develops standards, guidelines and recommendations that serve as a reference for international food trade to reduce food safety risks.

Food safety across the supply chain is also a high priority for industry stakeholders, regulatory

### 2. ANALYTICAL SYSTEMS FOR FOOD CONTROL ASSESSMENT

Analytical testing of food products can be done to detect or confirm microbial contamination, chemical contamination, allergens, and food authenticity.

#### 2.1. Food safety and quality testing

As for the intentional or accidental contamination of food products by undesirable or undeclared ingredients, the development and application of new analytical tools represents a great potential for quality assessment, assurance, control and safety of foods.

Techniques such as liquid chromatography-tandem mass spectrometry (LC-MS/MS) and gas chromatography-MS/MS have the capacity to detect chemical contaminants at very low concentrations, improving confidence in the food supply [2]. To improve the quality of surveillance, industry has developed a number of multiresidue methods in order to analyze multiple substances simultaneously. However, there are drawbacks in that some residues and metabolites may not be included in the screens. Furthermore, due to

different extraction protocols, the same method cannot always be used for all matrices. On the other hand, the ability to more completely monitor the food supply for known contaminants have to improve in order to meet the constantly advances in the control of foodborne hazards.

The availability of more sensitive, accurate and fast analysis methods is enhancing testing efficiencies, improving food quality programs and helping create a safer food supply: sensitive rapid test methods are required so that products, particularly those that are highly perishable, can be assessed and moved into the food supply if deemed safe. In this context, recent advances in online and high throughput screening strategies based on array-based detectors and imaging technologies using optical biosensors and hyperspectral imaging in food safety assessment as well as electrochemical and mass-based transducers have provided techniques that can detect multiple contaminants at one time [3]. These biosensor technologies are revolutionary in terms of food safety assessment and have been recently applied in various food products, including meat and poultry, seafood, fruits and vegetables. However, in general it is expected that the use of benchtop instrumentation, integrating sample treatment and analytical measurement, will favor the use of MS techniques for routine analysis in food safety control [4]: implementation of these technologies presents challenges in terms of effective identification of unknown compounds, food profiling, or candidate biomarker discovery.

Other food safety challenges relate to cross-cutting issues such as new food chain technologies: in this context, the use of nanotechnology is increasing, but concerns remain about their safety in the food chain so that the specific properties and characteristics of nanomaterials need to be considered for any potential health risks [5]. Recently, the Commission has proposed new regulation on novel foods aimed at simplifying the authorisation process and at clarifying the definition of novel food, including new technologies like nanotechnology which have an impact on food [6].

## 2.2. Food authenticity testing

As for the presence of unwanted or undeclared ingredients, food authenticity testing is important component in many quality and safety programs: if

targeted analytical approaches are useful when the chemical compound is known and specifically screened for, improvements in analytical instrumentation and data analysis software are allowing the development of nontargeted testing methods to create a “fingerprint” of an ingredient or food product for assessment of possible contamination [7]. Mass spectrometry using multiple reaction monitoring (MRM) acquisition mode has been recently proposed to devise a multiplex targeted method for the rapid detection and relative quantitation of the adulteration of meat with that of an undeclared species, the approach being based on the use of corresponding proteins from the different species under investigation and selected peptide markers for species detection [8]. Various technologies are being explored for nontargeted screening. Besides to LC-MS/MS coupled with multivariate analysis, e.g. principal component analysis, to recognize adulterated and unadulterated foods, other technologies have also been shown to identify potentially adulterated materials. Near-infrared analysis with chemometric data analysis is being used in the food industry as a quality assurance tool, and this technology has been expanded to monitor for food adulteration. As attested by these examples, chemometrics is of utmost importance to solve real-world food-related issues. Besides to food safety, authenticity and traceability testing, another field where chemometrics is effective tool is in investigating relationships between consumer preferences, sensory descriptors and molecular profiling of food [9].

Among other techniques, isotope ratio mass spectrometry (IR-MS) has proved to have great potential for assessing the authenticity of food of animal origin, being capable of determining geographical origin, animal diet and the technological process [10]. The main drawback of the application of this technique is the need of databases for each food commodity, which are expensive to create and to maintain in terms of both time and financial costs.

Other analytical methods used for species identification and authenticity of foods rely on protein and DNA analysis [11]. Among DNA-based procedures, methods based on the highly specific amplification of one or more DNA fragments by means of polymerase chain reaction (PCR) have been proposed as useful means for identifying species of origin in foods, as well as undeclared

allergenic ingredients and genetically modified organisms (GMO). PCR-based methods present a high potential due to their high specificity and sensitivity, as well as rapid processing time and low cost. These methods may be also used for the identification of meat or fish species and the recognition of genetically altered foods [12]. Molecular traceability based on DNA molecular markers proved also effective to protect premium products with European quality labels as well as to check the food label declaration [13].

### **2.3. Analytical quality assurance and metrological traceability of data**

In addition to developments in food science and technology, with the liberalization of food trade and improvements in transport, new legal requirements in mainly developed countries relating to traceability have recently been implemented [14]. Traceability throughout food chain combined with traditional food safety programmes requires the development of reliable analytical methods capable of detecting harmful residues and contaminants, thus diminishing risk and protecting the consumer. To this aim, analytical methods used by enforcement laboratories for the implementation of legislation must be subject to validation procedures. The prerequisite for reliability of measurement results is also comparability on the basis of known uncertainties which in turn are based on traceability to recognised references [15]. Estimating the measurement uncertainty needs a thorough knowledge about the measurement process and its sources of variation, the accuracy and precision of the measurements performed, and skill of the operators involved in the measurements and calculations. To ensure international metrological comparability, mutual recognition of analytical measurement results is required. The CIPM Mutual Recognition Arrangement (CIPM MRA) is the framework through which National Metrology Institutes demonstrate the international equivalence of their measurement standards leading to the internationally recognized Calibration and Measurement Capabilities (CMCs) of the participating institutes. Approved CMCs and supporting technical data are publicly available from the CIPM MRA database (<http://www.bipm.org/en/cipm-mra/>), which has become an essential reference for the accredited

laboratory community amongst others.

The ILAC Mutual Recognition Arrangement (ILAC MRA) delivers confidence in the acceptance of results providing significant technical support to the calibration, testing, and inspection results of the accredited conformity assessment bodies that are signatories to the ILAC MRA. In addition, the ILAC MRA enhances the acceptance of products across national borders and supports the provision of local or national services, such as providing safe food. By removing the need for additional calibration and testing, technical barriers to trade are reduced. In this way the ILAC MRA promotes international trade and the free-trade goal of “accredited once, accepted everywhere” can be realised. Under ILAC MRA the Member States shall ensure the quality of the results of the analysis of samples taken pursuant to Directive 96/23/EC, by monitoring tests and/or calibration results according to the International Standard ISO/IEC 17025:2005, which is designed for the accreditation of Testing and Calibration Laboratories. As for food control, Authorities may only designate laboratories that operate and are accredited in accordance with the European standard EN ISO/IEC 17025.

In the context of laboratory accreditation, the importance of validated methods as well as of internal and external quality control is well recognized. Great effort has been also made to set up harmonization documents for method validation and quality criteria for the identification and confirmation of residues and contaminants by MS-based methods particularly for food safety purposes [16]. To obtain reliable analytical data an effective measurement assurance requires also that the equipment must be used properly and personnel should be both qualified and competent to undertake the task.

EU Member States are responsible for enforcement of the EU food chain legislation including EU legislation on official controls (Regulation No. 882/2004). Competent authorities organize official control systems on their territory to check compliance of goods placed on the EU market with relevant standards and requirements. All business operators have to ensure compliance with EU food chain requirements at all stages of production, processing and distribution. The official control should be performed using appropriate techniques including routine surveillance checks and more intensive controls such as inspections, audits, sampling and sample testing. Sampling and

analysis methods used in official controls shall comply with relevant Community rules or with internationally recognised protocols in the absence of such rules or with other methods fit for the intended purpose in the absence of internationally recognized protocols. In this context, laboratories shall comply with the provisions of Article 12 of Regulation (EC) No 882/2004 and shall be able to demonstrate that they use internal quality control procedures. Quality control procedures should also include participation in external Proficiency Testing Schemes which comply with the 'International Harmonised Protocol for the Proficiency Testing (PT) of (Chemical) Analytical Laboratories developed under the auspices of IUPAC/ISO/AOAC. The role of the EU is to assure that the control systems at national level are effective. This is the task of DG SANTE. PT schemes ensure that technical competence is also maintained in the accredited laboratory.

Quality control and quality assurance systems are established to guarantee the robustness of analytical processes. Such systems make use of criteria related to: e.g. minimum sampling variability, representative sampling, recovery rates, minimum number of 'suspected false negatives and positives', linearity of the calibration curve, within- and between-laboratory repeatability and reproducibility of results, etc. Representative sampling is of primary importance. If samples are improperly collected and mishandled or are not representative of the sampled lot, the laboratory results will be meaningless. The criteria above mentioned are either specified in or related to regulations, by international bodies (e.g. Codex Alimentarius), in norms/written standards, in industrial product requirements, and/or consumer health needs. To ensure that the final laboratory results are correct, the quality assurance program incorporates those planned and systematic laboratory activities that guarantee the accuracy and defensibility of testing results. Besides to the development of harmonized systems of measurement, the high degree of consistency in experimental data required for the implementation of European Directive on food control can be achieved by the appropriate use of reference materials (RMs) and certified reference materials (CRMs). A CRM is the most important tool for achieving traceability of the measurement results and for obtaining and maintaining robust analytical processes. Certified Reference Materials contribute

to the validation of analytical procedures and sampling plans. CRMs also provide an important milestone in the development of robust analytical processes which are composed of screening and confirmatory procedures. They are a cost-attractive tool for validation and quality control of test kits and sensors. The International Organization of Legal Metrology set out guidelines with a view to facilitating a wider use of CRMs for legal metrological control also in the field of food safety. The availability of CRMs is still quite limited and does not fulfil the demands of the laboratories. Therefore, it is essential to encourage worldwide the production of new CRMs and the accreditation of reference material producers under the ISO Guide 34:2009 "General requirements for the competence of reference material producers".

## CONCLUSIONS

The significance of food control measures to the success of the global food trading system and the development of effective food control systems is highlighted. Analytical techniques and analytical systems made important contributions to the food industry, with new tools for investigation and analysis for food safety and quality control. There are also many emerging techniques available that can provide traceability information useful for food authentication purposes. Especially when used in combination with the aid of chemometrics, these techniques can provide extremely powerful tools.

The objectives of ensuring an acceptable level of food quality and safety can be pursued by implementing quality assurance measures along the entire food chain. Quality control and quality assurance systems are established to guarantee the robustness of analytical processes. Finally, the role of metrological comparability as the prerequisite for reliability of measurement results is highlighted.

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