Regulations in Food Testing

Chemical analysis of food is a pre-requisite for safeguarding correct labeling of food and protection of consumers against adulteration and misbranding of food. Of course, such tasks can only be achieved together with suitable food legislation, increased controls by food authorities, continuous studies by food safety agencies and universities to improve knowledge about food (processing), and by enhanced responsibility of the food industry. The latter is supported by food quality management systems like HACCP to prevent and / or control chemical, microbiological and physical hazards within the food supply chain. Such quality assurance procedures require chemical analysis throughout the whole food processing chain, starting from the raw materials and up to the final, labeled food product [1]. For ensuring that food complies to a certain minimum standard very often obligatory quality standards are applied in defining the ingredients and what the food must contain at minimum, including the nutritional composition [2, 3, 4]. Sometimes those standards also include the analytical procedures to be used. Examples for such encompassing quality standards are the US standards of identity or the Codex Alimentarius standards [2, 3].

Nutritional data on packaged food are necessary for helping consumers to choose food in accordance to their individual dietary needs and for reducing diet-related diseases [6, 7, 8, 34]. Accordingly, labeling regulations describe in detail the requirements for nutritional labeling (nutrients, amounts and caloric values) on food packages [5, 6]. To ensure a consistent nutrient declaration, the food manufacturer needs to perform additional testing for nutrients like sugars, organic acids, sugar alcohols, fat and fatty acids, protein and sodium as well as for vitamins and minerals [5].

All of these above described measures for ensuring food safety, provision of wholesome food, and consumer protection against adulteration and misbranding require reliable data obtained by chemical analysis of food. Reliable analytical results are also essential to facilitate international food trade.

Use of Quality Management Systems in Laboratories and Lab Accreditation

The reliability of chemical data depends significantly on how these data have been measured. The implementation of a quality management system for laboratories is an approved measure for safeguarding that the technical equipment and analytical methods are fit for purpose and reproducible [9] and that the staff is suitably qualified and experienced for their tasks.

Consequently, lab accreditation according to an international recognized system is a pre-condition for elaboration of reliable and internationally accepted analytical data [9].

The most important standard for laboratories is the international DIN ISO standard 17025 'General requirements for the competence of testing and calibration laboratories' [9, 10] which is also one of the most important standards for the worldwide globalization of trade. It addresses the technical competence of laboratories to carry out specific tests and is used worldwide by laboratory accreditation bodies as core requirement [9, 10]. The DIN ISO 17025 Standard comprises five elements that are 'Scope', 'Normative References', 'Terms and Definitions', 'Management Requirements' and 'Technical Requirements' [9]. The section 'Technical requirements' includes factors which determines the correctness and reliability of the tests and calibrations performed in laboratory like human factors, accommodation and environmental conditions, test and calibration methods, method validation, equipment, measurement traceability, sampling the handling of test and calibration items [9].

EU – Regulation 882/2004 - Consequences for food testing lab Accreditation

Within the European Union the official laboratories (i.e. those laboratories carrying out the analysis of samples taken during official controls by the competent food authorities of each member state) must be accredited according to DIN ISO 17025 as requested by EU Regulation 882/2004 [11]. The accreditation, however, may relate to individual tests or a group of tests.

Contract or third party laboratories need to be accredited according to this standard only if the results obtained by those laboratories are legally defensible data i.e that these data are used or are intended to be used in the event of a dispute between the competent food authorities and a food operator / manufacturer. However, as an indirect consequence of this regulation the European food market no longer accepts contract labs for food testing which are not accredited [10].

Accreditation in the USA as requested by the Food Safety Modernization Act

In 2011, the USA enacted the Food Safety Modernization Act (FSMA) [12]. It is an enormous reform program to improve food safety and to avoid intentional as well as non-intentional food adulteration within the whole supply chain also involving food testing labs. The FSMA is divided in four main parts. Part 2 of the FSMA ('Improving capacity to detect food safety problems') deals with the detection of food safety problems throughout the food supply chain. Section 202 includes rules to ensure that food testing laboratories (encompassing independent private laboratories, foreign laboratories, and laboratories operated by Federal, State, and local government agencies) meet prescribed standards for quality.

The FDA must establish criteria for laboratory accreditation and develop standards that laboratories must meet to be accredited. FDA missed the statutory deadline in 2013, but FDA officials have indicated a working group is developing a draft proposal and a proposed rule should be published by early 2016.

Correspondingly, accreditation bodies for lab accreditation need to established by the FDA [12]. Publicly available registry of accreditation bodies shall also be built up by the FDA [12]. According to Section 202 of the FSMA the FDA standards that must be met by accredited labs will include among others that

- appropriate sampling techniques are used,
- analytical procedures must be fit for purpose,
- reports of analyses must be certified,
- an internal quality system must be established and maintained,
- complaint evaluation and response procedures must exist, and
- technicians are qualified by training and experience.

These requirements are more or less the requests described by the DIN ISO Standard 17025 for lab accreditation and therefore already quite common for many labs throughout the world. It is planned that the FDA-accredited labs must be used by food manufacturers/ operators at least for

- any specific legal or regulatory testing requirement when addressing an identified or suspected food safety problem;
- any testing required by FDA to address an identified or suspected food safety problem;
- any testing to support admission of an imported food; and
- any testing under an import alert that requires successful consecutive tests.

A key requirement of section 202 is that accredited labs must provide the results of such tests directly to the FDA [12]. Reasons for this requirement may be that due to the other provisions of the FSMA, the number of necessary food samples will increase significantly. On the other hand the FDA does not have the necessary resources to deal with such a sudden rise of food samples to be tested. The FDA, therefore, makes the food manufacturer/operator responsible that the samples are tested and 'outsourced' to third parties, such as contract testing laboratories [14].

The use of official methods in food analysis

In food analysis, it is especially the complexity of the food matrix which has the largest impact on the performance and reliability of the analytical methods and procedures used [5]. The food matrix consists mainly of chemical compounds like protein, carbohydrate and lipids affecting significantly the performance of analytical methods. For example, high-fat or high-sugar foods can cause different types of interferences compared to low-fat or low-sugar food [5].
The application of extraction steps and digestion procedures, although, being a pre-condition for getting accurate analytical results, is often not only time-consuming, but sometimes also bears the risk of artifact formation [15]. Therefore, analytical methods for food analysis always need to take into account the characteristics and composition of the specific food matrix.

Several non-profit (scientific) organizations, such as the Association of Analytical Communities (AOAC), develop, standardize, and endorse official methods for food analysis. Such official methods play an important role in the analysis of foodstuffs, to ensure that food meets the legal requirements. As a consequence there are e.g. in the USA legal provisions requesting the use of a specific analytical method [16]. Furthermore, such official methods allow for comparability of results between different laboratories that follow the same procedure and for evaluating results obtained using new or more rapid procedures [5].

Official methods by WHO/FAO and Codex Alimentarius

The Codex Alimentarius Commission established in 1962 by the Food and Agricultural Organization (FAO) and the World Health Organization (WHO) develops international standards and safety practice for foods and agricultural products (the so-called Codex Alimentarius).

The Codex Alimentarius includes general requirements, code of practices and standards [17]. The Codex Commodity standards include methods for analysis of the respective commodity. Codex Volume 13 includes a list of official methods for analysis and sampling. The methods are sorted according to the specific commodity for which they can be applied (e.g. cereals, fats and oils, infant formulas etc.). There are also some methods which can be used for all kind of food exemplified by the method for detecting and quantification of the sweetener Cyclamate [18]. Codex recommends the use of the listed methods in application of DIN ISO 17025 [18].

Codex methods are elaborated by international organizations occupying themselves with a food or a group of foods and selected by the Codex Commission for Analysis [19]. Within this selection preference is given to those test methods that meet the criteria by the Codex Commission for Analysis for accuracy, precision, selectivity, limit of detection, sensitivity etc. The selection of methods also takes into account practicability and applicability under normal laboratory conditions. Consequently, preference will also be given to methods which have applicability for routine use and which are applicable uniformly to various groups of commodities. [19]. It should be mentioned that the majority of the analytical methods cited in Codex standards are those of the AOAC [22]. This is due to the fact that AOAC has had official observer status in Codex Alimentarius since its foundation and has given input on the development of Codex standards [22].

The Joint FAO/WHO Expert Committee on Food Additives (JECFA) sets standards for purity of food additives. It provides a compendium of food additive specifications comprised of four volumes [20]. The first three volumes are the food additive specifications themselves in alphabetical order while the last volume includes the revised and updated analytical methods, test procedures and laboratory solutions used by and referenced in the specifications. JECFA specifications include guidance on the analytical methods that should be used for testing according to the JECFA specification. Wherever possible, this is done by reference to the fourth volume of the compendium which includes the revised and updated analytical methods [20]. Otherwise details of the test procedures are described in the individual specifications monographs.

As JECFA specifications have been elaborated for a worldwide use, the referenced methods require the use of apparatus and equipment that is available in most laboratories [20]. According to JECFA, methods involving more recently developed techniques or equipment will not normally be quoted until such techniques are accepted internationally and are generally available at reasonable cost. Taking the advances in analytical chemistry in consideration JECFA is reviewing the analytical methods from time to time. In principle, it is possible to deviate from the JECFA methods, however, provided that the use of such other method or the modification to a JECFA method gives results of equivalent accuracy and specificity to those referenced in the respective JECFA specification [20].

Official methods for food analysis in the US

The FDA has established food definitions and standards which are published in 21 CFR 100-169 [2, 21] including standards of identity and quality. The standards of identity have been set for a wide variety of food products establishing specifically the ingredients a food must contain at minimum levels for expensive ingredients as well as at maximum levels for inexpensive ingredients (like e.g. water). Those standards mostly specify official analytical methods which are to be used for analysis. These methods have been elaborated by the international scientific organizations like the AOAC or by US organizations like e.g. the AACC (American Association of Cereal Chemists) or the AOCS (American Oil Chemists Society).

The Compendium Official Methods of Analysis of the AOAC International contains over 3000 methods adopted by the AOAC appropriate for a wide variety of food products and other materials [2, 22]. 21 CFR Section 2.19 defines the AOAC methods as "official methods" which are to be utilized by the FDA in case there is no analytical method described in a regulation [22, 23]. Accordingly, the FDA, and the Food Safety and Inspection Service (FSIS) of the United States Department of Agriculture (USDA) are using the AOAC methods to check if the food complies to the specific legal requirements, like nutritional labeling information on foods, presence or absence of undesirable residues or residue levels [21]. The AOAC reviews, selects and also develops methods. Once a method is selected by the AOAC it undergoes single lab validation and a full collaborative study involving 8–10 laboratories [22]. After a successful completion and approval by the AOAC Official Methods Board, the AOAC method will be published in AOAC Compendium and Journal [22].

Food chemicals codex (FCC) is a compendium containing standards for identification and purity for known food additives and chemicals (including provision of the respective analytical methods) used in food products either in the United States or internationally. Although the FCC standards have been developed in cooperation with the FDA and industry in the United States and elsewhere, the FCC does not provide information about the regulatory status of a food additives and chemicals [24]. However, some countries other than the US (e.g. Australia, Canada, New Zealand) recognize the FCC standards as legal requirements for food additives [24]. The FCC monographs include general information about the use of the particular food additive, chemical data, minimum standards for identity, purity, and quality of food additive and validated testing methods for verifying the purity and quality of the quoted food additive.

Official methods for food analysis in the EU

Existing official methods within the EU can be found e.g. in the German official collection of food analysis methods according § 64 to the German Food and Feed Act (so called Official Collection of § 64 LFGB Methods) including more than 1300 analytical procedures for food analysis [25, 26]. Experts from food control, science and the food industry develop analytical procedures and assess performance, reliability, and comparability of such methods. They decide for which field of application the methods are to be used. Before a method is included in the Official Collection, the methods are statistically tested by a number of labs in course of a inter-laboratory comparison test and are standardized [26].

In Germany, the Official Collection of § 64 LFGB Methods can be applied without any further justification [25, 26]. However, if a lab wants to use a different method instead, such deviation needs to be justified.

In EU legislation, official methods are often specified in case of product specific regulations to ensure that a certain standard of food quality can be maintained. An example is 'Regulation 2548/91 on the characteristics of olive oil and olive-residue oil and on the relevant methods of analysis' which contains a number of official methods [27].

In EU food legislation, however, the traditional approach for food analyses which foresees the use of such official or routine methods is more and more pushed aside by the so called performance criteria approach.

The Performance Criteria approach within the EU

As a result of advances in analytical chemistry the concept of routine methods and reference methods is more and more superseded by the so-called Performance Criteria approach, in which performance criteria for the analytical method and procedures for the validation of screening and confirmatory methods are established [11, 28].

Such change of paradigms is considered in EU Regulation 882/2004 [28]. This Regulation describes the principle requirements for sampling and analytical methods as conducted by the official laboratories of the EU member states. Laboratories involved in the analysis of official samples need to work in accordance with internationally approved procedures (e.g. DIN, CEN, ISO, IUPAC, Codex Alimentarius) or criteria-based performance standards and use methods of analysis that have been validated in accordance with e.g. IUPAC Harmonised Guidelines [11].

This has already been reflected in the EU Regulations 401/2006 [29], 333/2007 [30], 1882/2006 [31] for sampling and analytical methods for contaminants like nitrate, heavy metals, benz(a)pyrene and 3-MCPD. While these regulations describe in detail the sampling procedures (requirements for the personnel, number of samples per lot, precautions to be taken etc.), the regulation does not include (strong) recommendation for analytical methods, but specific requirements for the performance criteria like precision, recovery rate, measurement uncertainty etc. are defined and must be fulfilled by the used validated analytical methods. Also requirements regarding the laboratory quality management system to be applied are included.

For pharmacologically-active residues of authorized and non-authorized veterinarian medicinal products, e.g. certain antibiotics (like chloramphenicol) or substances having an anabolic effect (e.g. Clenbuterol), analytical methods can be selected by the respective lab but also need to be validated and to fulfill the performance criteria as given by EU Decision 2002/657 [32, 33]. The lab itself must comply with the quality norms as requested by EU Regulation 882/2004. However, the National Reference Laboratories develop and validate suitable analytical methods which can be used for analysis of veterinary drugs residues in food as described by the relevant EU legislation [33].

The above described measures aim to ensure the reliability of analytical results for food analysis. In the following compilation, analytical applications for food analysis will be described which may contribute to the reliability of analytical data by simplifying the sample preparation procedures.

References and useful links

Food legislation is a very dynamic legislation. Therefore, food regulations change frequently or are often amended. The given links to food legislation may not in all cases provide the most up-to-date version of the regulation. The most up-to-date legislation is to be found on the respective government websites.

[1] "Regulation (EU) No 852/2004 of the European Parliament and of the Council on hygiene of foodstuffs", Official Journal of the European Communities, L139/1, 2004 and subsequent amendments. http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=CELEX:32004R0852:EN:NOT

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[5] "Introduction to food analysis" Nielsen, S.S. in: "Food Analysis, Springer US, USA, 4th Edition, 2010, 5-13.

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[18] "Recommended methods of sampling and analysis, part A and B", Codex Standard 234-1999, Codex Alimentarius, Secretariat of the Joint FAO/WHO Food Standards Programme, FAO, Rome, 2011.

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[21] ""Food labelling", Curtis P. and Dunlap, W. in "Guide to food laws and regulations" Blackwell Publishing, USA, 2005, 85-111.

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European Union:
Eurlex:
http://eur-lex.europa.eu/de/index.htm.
This website allows you to search for the Official Journal of the EU and to get access to the text of the regulations (also accessible as consolidated versions)
European Commission:
http://ec.europa.eu/food/food/index_en.htm
European Food safety Agency
http://ec.europa.eu/food/efsa_en.htm

USA:
FDA (Food) homepage
http://www.fda.gov/Food/default.htm
US-DA homepage. The US-DA is responsible for safety of food derived from agriculture
http://www.usda.gov/wps/portal/usda/usdahome?navid=FOOD_SAFETY