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Foodstuffs — Determination of elements and their chemical species — General considerations and specific requirements

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National foreword

This British Standard is the UK implementation of EN 13804:2013. It supersedes BS EN 13804:2002 which is withdrawn.

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A list of organizations represented on this committee can be obtained on request to its secretary.

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Contents

Page

Foreword

This document (EN 13804:2013) has been prepared by Technical Committee CEN/TC 275 “Food analysis - Horizontal methods”, the secretariat of which is held by DIN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by September 2013, and conflicting national standards shall be withdrawn at the latest by September 2013.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

This document supersedes EN 13804:2002.

Compared to EN 13804:2002, the following changes were made:

a) the Scope was updated;

b) former Clause 3, "Terms and definitions" was deleted;

c) former Clause 4, "Performance characteristics" was deleted;

d) former Clause 5, "Sampling" was deleted;

e) former Clause 6, "Sample preparation" was updated and wording is now more precise and appears in the new Clause 2;

f) Subclause 2.3.2, "Storage for speciation purposes" was added;

g) Subclause 2.4.2, Table 2 on "Examples of sample preparation procedures for some foodstuffs" was thoroughly updated;

h) Subclause 2.4.3, "Drying and lyophilisation" was added;

i) Former Subclause 7.1, "Reagents" was revised and introduced in 3.1;

j) Former Clause 8, "Laboratory Quality standards" was completely revised and appears now as Clause 4, "Performance requirements and characteristics";

k) Former Clause 9, "Expression of results" was revised and appears now as Clause 5, "Interpretation and expression of results";

l) Former Clause 10, "Test report" was specified and appears now as Clause 6;

m) the Bibliographic references were completed;

n) the whole document was editorially revised.

According to the CEN/CENELEC Internal Regulations, the national standards organisations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

Introduction

The Working Group, CEN/TC 275/WG 10 "Elements and their chemical species", selects and elaborates methods of analysis of elements and their chemical species in foodstuffs.

There are many methods of analysis for the determination of elements in foodstuffs which have been validated and published; the analyst is often required to make a choice between several established methods all of which purport to be applicable to the same analyte/matrix combination. The Working Group decided to establish specific criteria to guide the analyst in the selection between several methods of analysis. As a general rule, analysts should give preference to methods of analysis which comply with the provisions given in Clauses 1 and 2 of the annex to [1], with the Decision in [2] and with the General Principles for Methods of Analysis of the Codex Alimentarius Commission (CAC), as defined in the CAC Procedural Manual and further developed in the ”criteria approach“ to methods of analysis developed by the Codex Committee of Methods of Analysis and Sampling (CCMAS).

The performance criteria laid down in this European Standard are based on published data or collected from official reports on European interlaboratory studies. When such performance characteristics are not available, the criteria were established based on the experience and opinions of the experts of CEN/TC 275/WG 10.

The criteria included in this European Standard have also been used as guidance in the Working Group 10 for the selection of specific methods of analysis of trace elements to be standardised.

In addition, the Working Group 10 also decided to provide some general information on sample handling and sample preparation, laboratory organisation and equipment.

This document does not deal with sampling issues. It provides an overview of the processes involved from receipt of the laboratory sample to the end result.

General sample preparation procedures are specified, as well as examples for some foodstuffs.

1 Scope

This European Standard specifies performance criteria for the selection of methods of analysis of elements and their chemical species in foodstuffs and contains performance requirements and characteristics, guidelines for laboratory set-up, sample preparation and test reports.

2 Sample preparation

2.1 General

Food samples are commonly treated in the same way as is done before consumption (washed, peeled, removal of non-edible parts). Apart from the treatments that are just mentioned and that are clarified in some more detail in 2.4.1 and 2.4.2, samples should not undergo further processing (i.e. samples should not be boiled, steamed, fried, heated or the like). Extra treatments, like drying, may be performed to prevent the sample from decaying. In particular, for speciation analyses, the potential loss or change of analytes shall be considered carefully though. Finally the sample needs to be homogenised (for example by grinding) to obtain a homogenous test portion. Contamination and uncontrolled loss of liquid, e.g. during operations such as grinding and cutting, shall be avoided to the greatest extent possible.

2.2 Reagents, apparatus and equipment

No reagents and chemicals other than water shall be used in sample preparation. Water with the quality of drinking water may be used for washing some samples, e.g. vegetables. However, depending on the purpose of the analysis and the element to be determined, it may be necessary to use purified water (see 3.1). Apparatuses and equipment should preferably be designated for trace element analysis.

Typical kitchen utensils (e.g. plastic dishes, plastic salad spinner, and coffee grinder) may also be used for sample preparation steps as long as they do not affect both the sample and the elements to be determined.

Any equipment coming into direct contact with the sample shall, if necessary, be cleaned with detergent and hot water. Equipment cleaned with detergent, and containers for storage of homogenised samples, should be treated with diluted nitric acid (approximately 6 % by volume) followed by purified water, if appropriate.

Contamination problems may arise when stainless steel or iron equipment is used for analysis of elements such as chromium, molybdenum, nickel and iron. Special apparatus shall be used in such cases, e.g. titanium or ceramic knives, agate mortar or ball mill for size reduction and homogenisation. Ceramic materials show a high degree of mechanical and chemical stability, but they might have varying compositions (e.g. Al-oxides, Zr-oxides).

Homogenisers shall be chosen carefully as not to cause losses of volatile elements (e.g. by heating up the sample during mixing) or to cause contamination of the elements to be determined or modification of species (e.g. due to warm-up by friction) in the case of speciation analyses.

2.3 Storage

2.3.1 General

Both laboratory samples and test samples shall be stored in such a way that the composition and sample mass does not change as a result of, for instance, drying out, evaporative loss, spoilage or decay. The test sample should be prepared as quickly as possible after arrival of the laboratory sample.

Test samples may be stored in a refrigerator or frozen depending on the nature of the material and the expected storage time. Suitable storage material may be made of glass, plastic containers closed with a screw cap or plastic bags. Dry products may be stored at room temperature.

2.3.2 Storage for speciation purposes

Some considerations shall be kept in mind when storing samples for speciation purposes. Parameters with a strong influence in speciation analysis are (see Table 1):

a) Temperature: Storage temperature shall be low enough to prevent microbial activity resulting in reactions, e.g. methylation and biodegradation (see Table 1).

b) pH: The pH of the media may strongly affect the stability of the inorganic species. Samples intended for species analysis shall not be changed in their acidity for preservation purposes.

c) Light: Light may cause instability of organometallic compounds by photodegrading. When analysing organometallic compounds storage shall be done in dark or in opaque containers.

d) Storage time: Generally, storage should be kept as short as possible.

Table 1— Examples of storage conditions for samples intended for speciation purposes

Element (species)	Storage conditions
Hg (Hg(II), methylmercury)	Keep samples at 4 °C or lower in darkness. Dilution shall be done only immediately before the analysis.
Sn (butylated organotin compounds)	PVC containers shall not be used to avoid contamination by organotin compounds. Store samples in borosilicate glass bottles in the dark, at least at 4 °C or lower.
Cr (Cr (III) and Cr (VI))	Only borosilicate or quartz glass should be used for handling and storage. Some glassware may cause contamination with chromium. Keep samples at 4 °C or lower. The pH has different effect on the stability of Cr (III) and Cr (VI).
Se (Se (IV), Se (VI) and methylated compounds)	Avoid the use of poly tetra fluoro ethylene containers [3]. Keep at -20 °C or lower to avoid any change of the inorganic species. Acid media (pH 1,5 to 2) avoid oxidation of Se (IV) to Se (VI).
As (As (III), As (V), and organic As-species)	Keep samples at 4 °C or lower, in darkness. Avoid the use of glassware as this may cause contamination with arsenate.

2.4 Procedures for sample preparation

2.4.1 General

For some trace elements sampling preparation procedures are laid down in EU legislation. Commission Regulation (EC) No 1881/2006 [4] defines the foodstuffs to which the EU maximum levels apply. Further sample preparation methods for official control of the levels of these elements are laid down in Regulation (EC) No 333/2007 [5]. Other specific legislation may also apply to trace elements.

Without prejudice to the specific provisions of EU legislation for official control, in food analysis only the edible part should be investigated and the results should refer to this part. Parts which are usually not intended for consumption should be removed, e.g. outer leaves, shell, skin and bones. Surface contamination like soil should be thoroughly removed by rinsing with drinking water or purified water. For preparation of the test portion a sufficient and representative amount should be available from the edible part of the laboratory sample. Please note that the edible part can differ between countries. The prepared and analysed part of the test portion has to be described in the test report (e.g. potatoes peeled or potatoes washed and unpeeled).

Rinse samples with water before cutting to avoid leaching from cut surfaces. After rinsing with drinking water samples shall be rinsed with purified water. Excess water should be physically removed, e.g. by shaking, using a sieve or a strainer (e.g. for leafy vegetables). Removing excess water by wiping with a soft tissue paper can cause random sample contamination and should not be used for elemental trace analysis. The washing step can be omitted for samples designated as “ready to eat”.

2.4.2 Examples of sample preparation for some foodstuffs

There is a huge variety of foodstuffs on the European market. Preparation practices vary between products as well as between geographical areas. It is therefore not possible to cover every possible preparation procedure in this standard. Examples of preparation procedures for some foodstuffs are given in Table 2. It is advisable to check if there is specific legislation or, e.g., traditions that could give some guidance in their preparation.

Table 2 — Examples of sample preparation procedures for some foodstuffs

Foodstuff	Sample preparation
Milk	Shake well.
Cheese	Remove waxed, oiled or coloured rind. Edible rind shall not be removed.
Eggs	Remove shell.
Muscle meat of warm blooded animals, liver and kidney	Remove parts of the samples which are not to be consumed, e.g. bones, tendons, connective tissues and fat. Homogenise the whole kidney, because some elements may not be equally distributed in kidney tissue.
Sausages	Remove non-edible parts, e.g. synthetic skin.
Fresh Fish	Rinse the fish with water. During cutting care should be taken not to damage the abdominal wall in order to avoid contamination of the muscle with the gut content Homogenise only the muscles, without skin and bones. Fish species which are normally intended for eating with bones and skin (scales), e.g. sardines, anchovies, sprats and whitebait, shall be homogenised whole. This shall be clearly noted in the analytical protocol.
Canned fish	Separate fish from the liquid phase of the product if possible. When the complete content of the can is intended for consumption, homogenise the entire content of the can.
Crustacean and molluscan shellfish	In crustaceans the visible digestive tract shall be removed prior to analysis. The edible part prepared from the laboratory sample should be free from remaining shell fragments or parts of exoskeleton. If the edible part of cephalopods is investigated, all parts of intestines (hepatopancreas) shall be quantitatively removed. For crabs only the muscle meat from appendages shall be used.
Cereals	The grain shall be separated from the chaff. If necessary the sample should be ground to a particle size low enough to obtain a homogenous portion for analysis.
Legume, oil seeds, nuts	Remove damaged or dirty parts. Remove shells from nuts. Legumes can be peeled if it is customary.
Potatoes	Remove sprouts and rinse with water to remove soil. Peel and wash with water.
Leafy vegetables	Remove dirty, withered, rotten and decaying parts and stalks, or wash if practical.
Shoot vegetables	Remove dirty, withered, rotten and decaying leaves. Remove stalks if they are not intended for eating. Rinse the sample. Samples where the peelings are not intended for eating (e.g. onions, kohlrabi) shall be peeled.
Root vegetables	Remove tops, tips, dirty and decaying parts and rinse with water. Samples where the peelings are not intended for eating (e.g. red beets) shall be scraped or peeled prior to rinsing.
Processed vegetables	No specific preparation is necessary. Products in liquid where the liquid is not intended for consumption should be drained, e.g. using a strainer. Products in liquid where the liquid is intended for consumption should be homogenised together with the liquid.
Mushrooms	Remove any rotten and dirty parts. Remove the skin of the cap if appropriate, and rinse with water, if necessary.
Fruit and fruiting vegetables	Remove stalks, sepals, petals, dirty and rotten parts. Rinse the sample and remove the seeds if necessary. Fruits which are not intended for eating with peel should not be rinsed but peeled and the seeds should be removed (e.g. pumpkin, melon).
Processed food (canned food, frozen food)	Remove the sauce, brine or other packing medium which is normally not intended to be eaten, by draining. Include the sauce/juice when intended to be eaten.
Dried food	Generally dried food, e.g. fruits, are intended for direct consumption, therefore selection of the edible part is required only.
Wine and alcoholic drinks	Remove any seal and clean the top of the bottle or container carefully with a moist tissue before it is opened. Wine with a visible precipitate shall be decanted prior to taking a test sample or test portion. Degas all CO_2 containing drinks using an ultrasonic bath before any test sample or test portion is taken.
Mineral water and beverages	Degas using an ultrasonic bath before any test sample or test portion is taken.

2.4.3 Drying and lyophilisation

Liquids may be removed, either by thermal drying (evaporation of moisture and alcohol), or by lyophilisation (sublimation of water). Care shall be taken to avoid losses of analyte or change of species, e.g. by lower drying temperature. Frozen products may be freeze-dried. Make sure the product to be freeze-dried is prepared into sufficiently thin layers. Drying is highly recommended for samples containing alcohol that shall be removed prior to decomposition to avoid uncontrolled reactions.

2.4.4 Homogenisation

After preparation according to Table 2, the samples shall be homogenised by suitable apparatus (see 3.3). For speciation analyses, please check that no species are modified during homogenisation.

Frozen foodstuffs may be homogenised before thawing. Take care to avoid loss of liquid during thawing. Uncontrolled loss of liquid may be caused by e.g. excessive grinding and exposure to elevated temperatures. Liquid nitrogen or carbon dioxide snow may also be used indirectly to aid for freezing and grinding of samples but should not come into contact with the sample.

Homogenisation of dry or semi-dry foodstuffs (e.g. tea leaves, herbs, corn, rice) may be simplified by adding a known amount of purified water (see 3.1).

3 Specific requirements for analysis of trace elements

3.1 Reagents

The concentration of trace elements in water shall be low enough not to affect the results of the determination. Purified water can be produced by, e.g. double distillation, distillation in quartz apparatus, de-ionisation, or sub-boiling distillation.

Chemicals of analytical grade are generally not sufficiently pure for determination of trace concentrations of elements. It is recommended to use high purity chemicals. Alternatively further purification of mineral acids (nitric or hydrochloric acid) in the laboratory by sub-boiling distillation procedures can be used.

3.2 Calibration materials and reference materials

The standard substances used for calibration should be accompanied by a certificate that makes it traceable to its origin. Reference materials (RM) used for the purpose of internal quality control should be, whenever possible, traceable to certified reference materials (CRM).

3.3 Containers, glassware and equipment

3.3.1 Special requirements concerning composition

In general, apparatus, containers and equipment made of high purity materials shall be used in order to avoid contamination with trace elements. Flasks, volumetric flasks, beakers, vessels, dishes, crucibles or containers shall be made out of fluoropolymers [e.g. polytetrafluoroethylene (PTFE), perfluoroalkoxyfluorocarbons (PFA)], quartz or polyolefines (e.g. polyethylene, polypropylene). Polyolefines may contain catalysts that might affect the determination of trace elements.

After pre-cleaning with diluted acid (e.g. nitric acid diluted approximately 6 % by volume), contamination from these materials is unlikely. But every type of material shall be checked to ensure that the concentration of elements to be determined has not been affected. However, the possibility of adsorption on the walls should be taken into account when the vessels are repeatedly used or show scratched surfaces.

Glassware apparatus are in principle not recommended for use in the determination of the total amount of trace elements owing to the possibility of release of substance from and adsorption on the glass wall.

For determination of iodine only glass vessels are recommended in order to avoid absorption and release of iodine during use.

In speciation analysis the use of dark borosilicate glass containers is recommended for mercury and tin species. For the determination of arsenic or chromium species glass containers should be avoided, because migration from glassware, e.g. arsenate from vials or glass pipettes, may affect samples and solutions.

3.3.2 Cleaning

Labware used in the analysis need to be cleaned in such a way that any contamination is removed prior to use. These cleaning procedures often contain the following steps: rinse with drinking water, treatment with a detergent solution (not containing any elements of interest), rinse again with drinking water and leave it overnight or at a longer time period in diluted nitric acid. Finally rinse with purified water before use.

Avoid the use of chromic sulphuric acid for cleaning of glassware.

For example, steaming of vessels of high chemical stability (quartz, fluoropolymers, glassware) with nitric acid is known to be an effective method for cleaning and regularly used in elemental trace analysis.

3.4 Apparatus

Measurement apparatus should, where possible, be set up in rooms other than that in which sample preparation is carried out.

3.5 Laboratory

The use of chemicals and substances for other analytical determinations or for general cleaning purposes, containing large quantities of one or more of the elements to be determined shall be avoided in rooms where trace element analysis is conducted.

Make sure that the incoming air has a low content of dust and, if possible, a higher pressure than outside of the laboratory. If necessary, use dust-free laminar airflow benches or fume hoods.

Regularly cleaning of the laboratory to remove dirt and dust is beneficial in order to avoid contaminations of equipment and samples.

4 Performance requirements and characteristics

4.1 General

Appropriate performance criteria for Pb, Cd, Hg and Sn are laid down in Regulation (EC) No 333/2007 [5]. A more detailed procedure for the identification of relevant analytical methods is described by Codex [6].

4.2 Limits of detection and quantification

The limit of detection (LOD) and limit of quantification (LOQ) are calculated from the variation of:

a) the analyte signal from blank solutions which either do not contain the analyte, or contain a quantity that is near zero, or;

b) a sample matrix that does not contain the analyte; in cases where it is not possible to find such a food matrix a simulation by major matrix elements may be used.

The blanks shall go through the whole analytical procedure including decomposition, dilution and determination. LOD is calculated by using the factor 3 x SD and LOQ the factor 6 or 10 times the SD [5].

The variation of the analyte signal in order to determine LOD/LOQ may be calculated in different ways.

c) Use the standard deviation (SD) for the mean of a sufficient number of blanks. The number of blanks should be ≥ 20 and be obtained on different days and on the basis of different calibrations. It should be emphasised that blank solutions consisting of water or diluted acid do not represent food matrices in general, but may represent foodstuffs with a low content of mineral matrix e.g. spirits and sugar. The LOD will vary depending on sample mass and dilution volume.

d) Spike the analyte free sample at concentrations of the expected LOD up to 10 x of LOD. In this case 10 different additions in equidistant concentrations are made, alternatively 4 additions in triplicate determination can be used [7]. The LOD/LOQ may differ between matrices, e.g. milk, meat, fish, vegetables, fruit etc. depending on type and amount of matrix and sensitivity of the method of determination on the matrix components. It is common to choose a substitute foodstuff for a certain class of matrix and determine LOD/LOQ for this substitute. The standard deviation of the blank (σ) is derived from the standard deviation of the intercept of the calibration curve, see [8].

In case these methods are not appropriate, other variations for determination of the detection and quantification limits are permitted, e.g. standard deviation of the background noise of the chromatogram at the retention time of the analyte in case of speciation analysis.

4.3 Method bias and recovery

In trace element analysis where the total amount of the analyte is determined after destruction of the organic matrix the method trueness is established by means of CRMs. The trueness is usually expressed as bias. The result from a CRM usually reflects the whole analytical process after homogenisation. A significant bias shall generally not be compensated for since it indicates methodological problems which should be identified and remedied. There are several procedures available for the calculation of bias [9], [10], [11].

Recovery is a performance characteristic that is usually used in methods containing an extraction or preconcentration step. Methods which determine the total amount of elements are not affected by factors e.g. extraction efficiency, and recovery therefore does not provide much valuable information. Influences caused by contamination or loss of elements during the procedure are random or systematic and may not be corrected by applying a factor. In these cases recovery is not relevant and correction should not be applied.

During extraction of chemical species of elements recovery can be corrected in those cases, where an incomplete extraction of the target species was shown. The analytical result shall be corrected for recovery only for speciation analyses and the level of recovery shall be reported (for more details, see [12]).

4.4 Measurement uncertainty

The measurement uncertainty should cover all factors, influencing the uncertainty of the final result. Therefore all steps of the procedure, including e.g. sample preparation, homogenisation, decomposition and determination shall be included in calculation and estimation of the measurement uncertainty.

With respect to the maximum levels of contaminants in foodstuff as described in EC regulation No 1881/2006 [4], it is recommended to calculate a maximum standard measurement uncertainty in order to obtain an objective decision whether a maximum level is exceeded or not. In this case the calculation can be based on the ‘fitness-for-purpose’ approach in Regulation (EC) No 333/2007 [5], representing a maximum measurement uncertainty with respect to the suitability of a method, according to Formula 1.

$$u_{\max} = \sqrt{\left(\frac{C}{b}\right)^2 + (\alpha \cdot C)^2} \quad (1)$$

where

u_{max} is the maximum standard uncertainty (µg/kg);

C is the concentration of interest (µg/kg);

α is a numeric factor depending on the value of C. The values to be used are given in Table 3;

b = 10 for concentrations less than 100 µg/kg;
= 20 for concentrations equal or above 100 µg/kg;

The factor b takes into account, that the LOD should be less than one tenth of the maximum level, respectively one fifth for concentrations less than 100 µg/kg.

Table 3 — Numeric values to be used for constant α in Formula (1) depending on the concentration of interest

C µg/kg	α
≤ 50	0,20
51 to 500	0,18
501 to 1 000	0,15
1 001 to 10 000	0,12
> 10 000	0,10

The expanded maximum measurement uncertainty U_{max} is calculated with Formula (2):

$$U_{max} = 2 \cdot u_{max} \quad (2)$$

representing a confidence level of approximately 95 %.

A method used for official control shall produce results with a lower maximum measurement uncertainty as calculated by the formula above. It shall be kept in mind that U_{max} only indicates what the maximum uncertainty associated to a measurement can be in compliance with the European Legislation on contaminants. A proper evaluation of the uncertainty of the method used shall be performed to check if it fulfils the requirement or not.

4.5 Analytical Quality Assurance (AQA)

4.5.1 General

Laboratories shall demonstrate that they are under control and are operating proficiently. Laboratories should have a documentation system for analytical quality assurance based on the requirements in EN ISO/IEC 17025 [13].

4.5.2 Analytical Quality Control (AQC)

This is an integrated part of AQA and serves to safeguard that the laboratory has appropriate procedures for monitoring the validity of the test results. This includes regular use of RM or CRM, and participation in proficiency tests (PT). It also includes judgement of test results and deciding whether reanalysis with the same or a different method may be called for and what may be needed to safeguard the results.

4.5.3 Internal Quality Control

Food control laboratories should be able to demonstrate that they have internal quality control procedures in place. Examples of those are given in EN ISO/IEC 17025 [13] and in [8].

The internal quality control consists of:

a) Blank samples, serving for the control of contamination of the acids and vessels used;

b) Reference Materials (RM's), representing the accuracy of the method and procedure;

c) Samples analysed in two independent repeats, serving as indicator of contamination, sufficient homogeneity of the sample or losses during the analytical procedure.

It is an important factor of the internal quality control, to run all the samples from a) to c) through the complete analytical procedure (e.g. decomposition, dilution etc.).

A deeper insight into Internal Quality Control can be gathered in [8].

4.5.4 Use of CRMs

A CRM is usually analysed in parallel to the test sample and should be treated identically. The purpose of the analysis of the CRM is primarily to get an estimation of the method bias. It is therefore important that the CRM should be as similar to the test sample as possible, with regards to the matrix and analyte level. The result of the CRM analysis should be evaluated with respect to:

a) water content of the material (certified values are often referred to dried material);

b) confidence interval (or similar variation parameters) of the CRM and

c) measurement uncertainty of the method used.

4.5.5 Use of RMs

When the use of RMs is preferred the RMs should be, whenever possible, traceable to CRMs. This can be achieved by analysis of a CRM, with similar matrix and analyte concentration, in direct sequence, e.g. Std (Standard Substance) – CRM – RM – Blank – RM – CRM – Std. Check, using an appropriate statistical procedure, if there is a significant difference between the two sets of results. Note, however, that acceptable difference does not transform the RM into a CRM.

4.5.6 Proficiency tests

By taking part in proficiency tests (PT), a laboratory gets an independent evaluation of its competence in a specific area of analysis. It is important that the PT test item is as similar as possible to the test samples, with regards to the matrix and analyte level. Many accreditation bodies require laboratories to take part in PT in order to achieve, and maintain, accreditation. However, PT programmes are not available for every analyte or sample matrix.

5 Interpretation and expression of results

All final results should be reported including the measurement uncertainty previously established if required.

For official control purposes specific provisions for interpretation and expression of results have been laid down in Regulation (EC) No 333/2007 [5].

For other analyses, it is recommended to report the expanded measurement uncertainty with the final result, in case:

a) a certain limit is exceeded;

b) to compare the final result with results from other laboratories.

In Regulation (EC) No 333/2007 [5] it is required to always report the results with the expanded measurement uncertainty for official control purposes, and not only in case of suspicion of exceeded maximum limits (MLs).

A final result below the limit of quantification should be reported as “< LOQ (mg/kg)” or “less than LOQ (mg/kg)”, where LOQ is a numerical value, e.g, < LOQ (0,010 mg/kg) or < 0,010 mg/kg (LOQ).

It is not recommended to report results as e.g. “< LOD”, “n.d.”, “n.b” or equal.

Numeric value and units of expression of results should be in accordance with the appropriate regulation and the requirement of the precision (e.g. if the limits are set in mg/kg, results should be expressed in the same units and with the same number of significant figures).

For some foodstuffs it is frequently required to express concentration results as mass of the element on sample dry mass basis. In this case information on the water content of the sample analysed should be provided, as well as the humidity determination technique used.

6 Test report

The test report should follow EN ISO/IEC 17025 [13]. Usually, the following information is considered to be the minimum for a report, but additional information may be required, depending on the circumstances.

a) all information necessary for the complete identification of the sample;

b) description of the test portion analysed and prepared part of the sample;

c) the test method used, with reference to this document;

d) the test results obtained and the units in which they are specified;

e) date when the analysis was finished;

f) whether the requirement of the repeatability limit has been fulfilled;

g) all operating details not specified in this document, or regarded as optional, together with details of any incidents occurred when performing the method which may have influenced the test results.

Bibliography

[1] Regulation (EC) No 882/2004 of the European parliament and of the council of 29 April 2004 on official controls performed to ensure the verification of compliance with feed and food law, animal health and animal welfare rules (OJ L 191, 28.5.2004, p. 1).

[2] Council Directive 96/23/EC of 29 April 1996 on measures to monitor certain substances and residues thereof in live animals and animal products (OJ L 125, 23.5.1996, p. 10-32).

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