GUIDANCE FOR THE ACCREDITATION OF GENERIC METHODS & METHODS OF FLEXIBLE SCOPE OF DETERMINATION OF METALS IN FOODS

ESYD G-METALS

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Prepared by: The Quality Manager of ESYD
Approved by: The Head of ESYD

Quality Manager of ESYD **Head of ESYD**

1. Scope

Taking into account the principles of analytical chemistry, it is possible to obtain accreditation for a generic method of determination of metals in food with an accreditation field which includes specific metals in the general category of "food, provided the following requirements are met:

Either in the method text or in another appropriate laboratory document the following should be specified:

1.1.Method Scope
The scope of the method should cover the following

i. The method's aim
ii. The analytes to be determined by the method
iii. The form in which the analytes are to be determined e.g. total mercury and/or methyl - mercury
iv. The matrixes for which the specific analytes will be determined
v. Application range of the method (concentration range for which the method will be applied per analyte and per substrate)
vi. Interferences or restrictions to method application
vii. Technique used

1.2 Technical Requirements

1.2.1. The instrumental analysis technique employed for the metals' determination e.g. by atomic absorption spectrometry and graphite furnace technique (GFAAS)

1.2.2 Food categories in which the method is applied e.g. fish products, meat, cereals, vegetables, infant formula etc.

1.2.3. Metals determined and the corresponding technical.

1.2.4. The pre-treatment per food category and , where appropriate , relevant variations (e.g. wet digestion with specific acids), specifying if necessary the quantity of sample required , the amount and type of reagents , time and digestionconditions , etc. The specific requirements for sample preparation as described in section G2.2.1 Reg . EK333 / 2007 *mutis mutandis* should also be taken into account/consideration

1.2.5. The instrument settings per category of metals

1.2.6. Suitability checks and calibration

The working range of the calibration curve, and the the mathematical formula of the curve and the goodness-of-fit of the data to the curve should be described

2. Method validation- requirements

2.1. The validation process should include the following

- Evaluation of method trueness
- Evaluation of precision
- Evaluation of the calibration curve

- Determination of limit of detection and limit of quantification limit.

2.2. Full validation and assessment of trueness and precision should be carried out for each metal in at least one foodstuff from each food category as grouped below, for foodstuffs of the same category indicative ruggedness tests should be performed in order to prove that the performance of the method does not differ/vary significantly.

2.3 Legislative requirements

If legal limits and specific performance criteria exist for the method, they should be taken into account during validation. Regulation EK1881 / 2006 (*mutis mutandis*) set legal limits for the metals lead, cadmium, mercury and inorganic tin. The applied methods of determination of these metals must meet the performance criteria of paragraph G3.3.1 Table 5 of the Table 5 of Regulation EK333 / 2007 (*mutis mutandis*). For arsenic, Legislative limits for arsenic and copper are defined in EU Regulation 2015/1006 (amending Regulation EK1881 /2006), and in Regulation EK396 2005, respectively .

2.4 Food categories which must be taken into account during validation are at a minimum the following:

A) Food of plant origin:

1. Food with high water content (fruits , vegetables , mushrooms , fruit and vegetable juices , wine)
2. Cereals, legumes and their products
3. Cocoa, chocolate and their products
4. Fats, oils , food with a high content of vegetable oil

B) Food of animal origin

1. Meat and fish
2. Milk, milk powder
3. infant formulae and follow-on formulae
4. Dairy products
5. Honey and food with high sugar content

For processed products that are classified into one of the above mentioned categories based on their composition indicative ruggedness tests should be performed in order to prove that the performance of the method does not differ/vary significantly. Complete validation should be performed for foods which are not classified in the above categories of foods

2.5 Concentration levels at which validation is performed should reflect the levels defined by the legal limits by metal and food category. If for a particular food a maximum permitted limit exists for a specific metal, validation will be performed on that specific food for the concentration level of the legislative limit
(e.g. lead in offal at a concentration of 0.50 mg / kg), regardless of the food selected for extensive validation in different/several concentration levels. Iif there are different legislative limit sin different foods of the same category representative levels should be selected for a foodstuff that is representative of the food category
(.e.g validation of the method for determining cadmium at levels of 0.05, 0,3and 0,8 mg /kg in samples of fish / meat is considered satisfactory and covers the MRL of 0.05 , 0.1, 0.2, 0.5, and 1 mg / kg in various meat or fish products) .

In any case, the validation should be performed on at least two levels of concentration per food category.

2.6 Detection and quantification limit

If there are different levels in various foods of the same category the most representative food of the category food and / or the one with the lowest legal limit should be selected. There are several calculative approaches in the literature used to determine the limit of detection and limit of quantification such as blank samples or samples containing the analyte at low concentrations. For the calculation of the detection and quantitation limits alternative processes may be used. In any case there should be a confirmation of the determined detection or quantification limits using spiked samples. Furthermore, the criteria for the detection and quantification limits set out in Regulation EK333 / 2007, as amended and in force, should be fulfilled.

3. Uncertainty

Uncertainty is calculated separately for each analyte, in each food sub-category and for each concentration level where validation is performed. Where a limited number of fully validated methods of analysis exist, alternatively, a 'fitness-for-purpose' approach may be used to assess the suitability of the method of analysis. Methods suitable for official control must produce results with standard measurement uncertainties lower than the maximum standard measurement uncertainty specified in paragraph G3.3.2 of EC Regulation 333/2007, as amended and in force.

4. Expression of results

The results shall be expressed in the same units and with the same number of significant figures as the maximum levels laid down in legislation. If the analytical results are to be corrected for recovery, recovery values must be reported. Additionally, the expanded measurement uncertainty will be reported using a coverage factor of 2 which gives a confidence interval of approximately 95 %.

5. Internal quality control

All stages of the analytical procedure should be applied to the Internal control samples used
The choice of the internal control scheme followed (type of substrate / substrates, analyte concentration, control frequency, etc.) depends on the scope of the method and is documented by the laboratory.
The long term internal control data can be used for a more accurate assessment of random and systematic errors of an analytical procedure

6. External quality control

The laboratory must select interlaboratory proficiency testing organizers and a participation frequency of each analyte so as to satisfy the requirements of ESYD PDI.

7. Flexible Scope

Taking into consideration the "Guideline for the accreditation of laboratories based on a flexible scope " ESYD - KO EVEL / 01 / 00 / 28-7-11 it is possible to grant accreditation based on flexible scope to a metals' determination method, if the following requirements are met:

7.1 Either in the method description or in another appropriate laboratory document the following should be specified beyond the information listed in paragraphs 1.2.1 to 1.2.5 the scope and limits of the flexible scope (analytes, substrates and techniques) should also be specified.

7.2 Flexibility categories may be the following:

i. Flexibility regarding the test matrix / product (e.g. extending the application field of cadmium determination in fish to fruits)

ii. Flexibility regarding the analyte (e.g., extension of the determination method of lead in fish to determine cadmium in fish).

iii. Flexibility concerning analytical method changes depending on the matrix and the analyte.

7.3. The group of matrices with similar analytical performance is specified (see par.2.3)

7.4. The method describes standard operating procedures which include minimum validation requirements that must be performed in order to add new matrixes or selected analytes to a flexible scope

7.5. It describes the criteria required for a flexible scope validation to be considered successful. If unsuccessful within the flexible scope measures will apply described in section 3.5 of the "Guidelines for the accreditation of laboratories based on flexible scope" ESYD - KO EVEL / 01 / 00 / 07.11.28.

7.6. There is a documented procedure for estimating uncertainty when new matrixes or analytes are added.

7.7. For the remaining requirements for the accreditation of flexible scope methods the "Guideline for the accreditation of laboratories flexible scope» ESYD - KO EVEL / 01 / 00 / 07.11.28 applies.