SLOVENSKI STANDARD
kSIST-TS FprCEN/TS 17174:2017

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Krma: metode vzorčenja in analize - Izvedbena merila v posameznem laboratoriju in v primerjalnem preskusu validirane metode analiz za določanje težkih kovin

Animal feeding stuffs: Methods of sampling and analysis - Performance criteria for single laboratory validated and ring-trial validated methods of analysis for the determination of heavy metals

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Futtermittel - Probenahme- und Untersuchungsverfahren - Leistungskriterien für laborintern validierte und Ringversuch validierte Analysemethoden zur Bestimmung von Schwermetallen

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Aliments des animaux : Méthodes d'échantillonnage et d'analyse - Critères de performance pour les méthodes d'analyse validées par un ou plusieurs laboratoires pour le dosage des métaux lourds

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65.120	Krmila	Animal feeding stuffs

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2003-01.Slovenski inštitut za standardizacijo. Razmnoževanje celote ali delov tega standarda ni dovoljeno.

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TECHNICAL SPECIFICATION

SPÉCIFICATION TECHNIQUE

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**FINAL DRAFT
FprCEN/TS 17174**

October 2017

ICS

English Version

Animal feeding stuffs: Methods of sampling and analysis - Performance criteria for single laboratory validated and ring-trial validated methods of analysis for the determination of heavy metals

Aliments des animaux : Méthodes d'échantillonnage et d'analyse - Critères de performance pour les méthodes d'analyse validées par un ou plusieurs laboratoires pour le dosage des métaux lourds

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This draft Technical Specification is submitted to CEN members for Vote. It has been drawn up by the Technical Committee CEN/TC 327.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

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European foreword

This document (FprCEN/TS 17174:2017) has been prepared by Technical Committee CEN/TC 327 "Animal feeding stuffs: Methods of sampling and analysis", the secretariat of which is held by NEN.

This document is currently submitted to the Formal Vote.

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Introduction

The working group, CEN/TC 327 "Animal feeding stuffs" WG 4 "Methods of sampling and analysis" selects and elaborates methods of analysis of elements and their chemical species in feedstuffs.

There are many methods of analysis for the determination of elements in feedstuffs, which have been validated and published. It is often required to make a choice between several established methods applicable to the same measurand (analyte/matrix combination).

The Working Group was mandated by the European Commission [1] 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 the Regulation (EC) No 882/2004 [2], with the Decision in the Directive 2002/32/EC [3], with the Commission Regulation (EC) No 152/2009 [4], or with the General Principles for Methods of Analysis of the Codex Alimentarius Commission (CAC), as defined in the CAC Procedural Manual [5] and further developed in the "criteria approach" to the methods of analysis developed by the Codex Committee of Methods of Analysis and Sampling (CCMAS) [6].

The performance criteria laid down in this document are based on published guidance documents (such as the AOAC guidelines [7]; the IUPAC Harmonized Protocol [8] and the SANCO document [9]) or data collected from official reports on European collaborative studies (e.g. EN 15510 [10] or CEN/TS 15620 [11]). When such performance characteristics are not available, the criteria were established based on the experience and opinions of the CEN/TC 327/WG 4.

The criteria included in this document have also been used as guidance in the Working Group for the selection of specific methods of analysis of trace elements to be further standardized, for the evaluation of on-going collaborative trials and for the review of previously published standards of analytical methods.

NOTE   All the criteria provided in this document refer to concentrations (mass fractions) greater than or equal to 0,1 mg/kg, related to the lowest maximum level (ML) set for mercury in feed.

1 Scope

This document specifies performance criteria for the selection of single-laboratory validated or collaborative-trial validated methods of analysis of elements and their chemical species in feed. The terms and definition of the relevant parameters for method validation are included. The performance requirements and characteristics are provided. This document may serve as a guide:

— to assess the quality of new European Standard methods under validation;

— to review the quality of previous collaborative trials;

— to confirm the extension of the scope of an already published European Standard applied to other analyte concentrations or matrices; or

— to evaluate the fitness-for-purpose of single-validated methods.

The performance criteria can apply to methods dedicated to the determination of heavy metals, trace elements, major elements and minerals.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
accuracy
closeness of agreement between a test result and the accepted reference value

[SOURCE: ISO 5725-1, see [12]]

Note 1 to entry: The term accuracy, when applied to a set of test results, involves a combination of random components and a common systematic error or bias component.

Note 2 to entry: It is assessed by determining trueness and precision.

[SOURCE: 2002/657/EEC, see [13]]

3.2
applicability
scope of the analytical method; description of the analytes, matrices, and concentration ranges (mass fractions) for which a method of analysis may be used satisfactorily to determine compliance with a given standard (i.e. CEN, ISO, CODEX)

Note 1 to entry: In addition to a statement of the range of capability of satisfactory performance for each factor, the statement of applicability (scope) also includes warnings as to known interference by other analytes, or inapplicability to certain matrices and situations.

3.3
bias
difference between the expectation of the test results (x) and an accepted reference value (x_{ref})

[SOURCE: ISO 5725-1, see [12]]

Note 1 to entry: The bias can be expressed in absolute or relative terms (b or $b(\%)$, respectively) as:

$$b = x - x_{\text{ref}} \tag{1}$$

$$b(\%) = \frac{x - x_{\text{ref}}}{x_{\text{ref}}} \times 100 \tag{2}$$

[SOURCE: Eurachem 2014, see [14]]

3.4
inter-laboratory comparison
organisation, performance and evaluation of measurements or tests on the same or similar items by two or more laboratories in accordance with predetermined conditions

[SOURCE: ISO/ISO 17043:2010, see [15]]

Note 1 to entry: The larger the number of participating laboratories, the greater the confidence that can be placed in the resulting statistical parameters.

3.4.1
collaborative trials
inter-laboratory comparison aiming at the evaluation of the performance characteristics of a method – often described as ring-test or ring-trial

[SOURCE: ISO/ISO 17043:2010, see [15]]

Note 1 to entry: It means analysing the same sample by the same method to determine the performance characteristics of the method.

Note 2 to entry: The study covers random measurement error and laboratory bias.

[SOURCE: 2002/657/EEC, see [13]]

Note 3 to entry: The reported results are used to estimate the performance characteristics of the method, such as precision. When necessary and possible, other pertinent characteristics such as systematic error and/or recovery may be reported.

3.4.2
proficiency testing
inter-laboratory comparison aiming at the evaluation of participant performance against pre-established criteria

[SOURCE: ISO/IEC 17043:2010, see [15]]

Note 1 to entry: The reported results are compared with those from other laboratories or with the known or assigned reference value, usually with the objective of evaluating or improving laboratory performance.

Note 2 to entry: Proficiency testing means analysing the same sample allowing laboratories to choose their own methods, provided these methods are used under routine conditions.

[SOURCE: 2002/657/EEC, see [13]]

3.4.3
reference material characterisation
inter-laboratory comparison aiming at the assignment of values to reference materials and assessment of their suitability for use in specific test or measurement procedures

[SOURCE: ISO/IEC 17043:2010, see [15]]

3.5
limit of detection
*x*LOD
concentration (mass fraction) derived from the smallest measure that can be detected with reasonable certainty for a given analytical procedure

[SOURCE: ISO 11843, see [16]]

Note 1 to entry: For analytical systems where the validation range does not include or approach it, the detection limit does not need to be part of a validation.

[SOURCE: IUPAC 2002, see [8]]

Note 2 to entry: The various conceptual approaches to the subject depend on the estimate of precision at or near zero concentration (mass fraction) under repeatability or reproducibility conditions.

3.6
limit of quantification
*x*LOQ
lowest concentration (mass fraction) determined with satisfactory measurement uncertainty

[SOURCE: ISO 11843, see [16]]

Note 1 to entry: It is useful to state a concentration (mass fraction) below which the analytical method cannot operate with an acceptable precision. Such precision is arbitrarily defined as relative standard deviation of 10 %.

Note 2 to entry: Usually the limit is set as a fixed multiple (e.g. 3,3) of the detection limit.

Note 3 to entry: Measurements below LOQ are not devoid of information content and are fit for purpose.

[SOURCE: IUPAC 2002, see [8]]

3.7
linearity
ability (within a given range) of an analytical procedure to obtain test results which are directly proportional to the concentration (mass fraction) of analyte in the sample

3.8
lowest validated level
lowest concentration (mass fraction) investigated in the frame of a collaborative trial or a single-laboratory validation

3.9
measurement uncertainty
parameter associated with the result of a measurement, that characterises the dispersion of the values that could reasonably be attributed to the measurand

[SOURCE: GUM, see [17]]

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Note 1 to entry: Uncertainty should be distinguished from an estimate attached to a test result which characterizes the range of values within which the expectation is asserted to lie. This latter estimate is a measure of precision rather than of accuracy and should be used only when the true value is not defined.

Note 2 to entry: The measurand is a description of the specific quantity intended to be measured. In chemical and biochemical analysis the specification of the measurand requires at least the description of the quantity (e.g. mass fraction or amount of substance concentration), the analyte and where relevant the matrix.

Note 3 to entry: Uncertainty of measurement comprises, in general, many components. Some of these components may be estimated on the basis of the statistical distribution of the results of a series of measurements and can be characterized by standard deviations. Estimates or other components can only be based on experience or other information.

[SOURCE: TAM, see [18]]

3.10
precision
closeness of agreement between independent test results obtained under stipulated conditions

[SOURCE: ISO 5725-1, see [12]]

Note 1 to entry: Precision depends only on the distribution of random errors and does not relate to the true value, conventional true value or specified value.

Note 2 to entry: The measure of precision is usually expressed in terms of imprecision and computed as a standard deviation of the test results. Less precision is reflected by a larger standard deviation.

Note 3 to entry: "Independent test results" means results obtained in a manner not influenced by any previous result on the same or similar test object. Quantitative measure of precision depends critically on the stipulated conditions. Repeatability and reproducibility conditions are particular sets of extreme conditions.

3.10.1
repeatability
precision under repeatability conditions

[SOURCE: ISO 5725-1, see [12]]

3.10.1.1
repeatability conditions
conditions where independent test results are obtained with the same method on identical test items in the same laboratory by the same operator using the same equipment within short intervals of time

[SOURCE: ISO 5725-1, see [12]]

3.10.1.2
repeatability standard deviation
s_r
standard deviation of test results obtained under repeatability conditions

Note 1 to entry: It is a measure of the dispersion of the distribution of test results under repeatability conditions.

Note 2 to entry: Similarly "repeatability variance" and "repeatability coefficient of variation" could be defined and used as measures of the dispersion of the test results under repeatability conditions.

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3.10.1.3
repeatability relative standard deviation
*RSD*r
relative standard deviation of test results obtained under repeatability conditions

Note 1 to entry: The repeatability relative standard deviation (RSD_r) can be expressed as follows:

$$RSD_r\,(\%) = \frac{s_r}{c_{mean}} \times 100 \qquad (3)$$

where

c_{mean} is the mean concentration (mass fraction)

3.10.1.4
repeatability limit
r
value less than or equal to which the absolute difference between two test results obtained under repeatability conditions may be expected to be within a probability of 95 %

Note 1 to entry   The repeatability limit (r) can be expressed as follows:

$$r = 2\sqrt{2} \times s_r \qquad (4)$$

[SOURCE: ISO 3534-1, see [19]]

[SOURCE: ISO 5725-6, see [20]]

3.10.2
within-laboratory reproducibility (intermediate precision)
precision under within-laboratory reproducibility conditions

3.10.2.1
within-laboratory reproducibility conditions
conditions where test results are obtained with the same method on identical test items on different days with different operators using different equipment

[SOURCE: ISO 5725-1, see [12]]

3.10.2.2
within-laboratory reproducibility standard deviation
*s*WLR
standard deviation of test results obtained under within-laboratory reproducibility conditions

Note 1 to entry   It is a measure of the dispersion of the distribution of test results under within-laboratory reproducibility conditions

3.10.2.3
within-laboratory reproducibility relative standard deviation
*RSD*WLR
relative standard deviation of test results obtained under within-laboratory reproducibility conditions

Note 1 to entry: The within-laboratory reproducibility relative standard deviation (RSD_{WLR}) can be expressed as follows: