SLOVENSKI STANDARD
SIST-TS CEN/TS 17455:2020

01-julij-2020

Krma - Metode vzorčenja in analize - Izvedbena merila za validacijo metod v posameznem laboratoriju in v primerjalnem preskusu analiz za določanje mikotoksinov

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 mycotoxins

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

Aliments des animaux - Méthodes d'échantillonnage et d'analyse - Critères de performance des méthodes d'analyse des mycotoxines validées dans un seul laboratoire ou suite à un essai interlaboratoires

Ta slovenski standard je istoveten z: **CEN/TS 17455:2020**

ICS:

65.120	Krmila	Animal feeding stuffs
71.040.50	Fizikalnokemijske analitske metode	Physicochemical methods of analysis

SIST-TS CEN/TS 17455:2020 **en,fr,de**

TECHNICAL SPECIFICATION

SPÉCIFICATION TECHNIQUE

TECHNISCHE SPEZIFIKATION

CEN/TS 17455

April 2020

ICS 65.120; 71.040; 71.040.50

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 mycotoxins

Aliments des animaux - Méthodes d'échantillonnage et d'analyse - Critères de performance des méthodes d'analyse des mycotoxines validées dans un seul laboratoire ou suite à un essai interlaboratoires

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This Technical Specification (CEN/TS) was approved by CEN on 27 January 2020 for provisional application.

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Ref. No. CEN/TS 17455:2020 E

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European foreword

This document (CEN/TS 17455:2020) 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.

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

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Introduction

The European Committee for Standardization (CEN) selects and elaborates methods of analysis for organic contaminants that are to become European Standards. These standards can be used for those contaminants that are subject to regulation. When used for this purpose, the main functions of a standard are to enable feed manufacturers to determine with reasonable certainty whether a consignment may be put on the market and to enable regulatory authorities to determine equitably whether feedstuffs on the market comply with legal or recommended limits.

CEN/TC 327/working group (WG) 5 decided to establish a criteria guide in order to allow benchmarking of methods of analysis for their fitness for purpose [1]. The performance criteria laid down therein are based on published data, collected from official reports on inter-laboratory studies [2] to [12].

Where performance characteristics are absent or limited in availability, the criteria were estimated based on the experiences and opinions of the experts of the CEN working group. The selection criteria could need updating in future revisions of this document, if newer or more accurate data on method performance characteristics become available. This document lists relevant performance parameters and gives information on their definition. It further describes how they can be practically obtained; it indicates guidance values demonstrating fitness for purpose. As a result, these guidance values serve as a benchmark for experienced analytical laboratories.

This document may contain useful information for CEN members, the European Commission, the EFTA secretariat, other governmental agencies or outside bodies. The criteria in this CEN report are used as guidance in the CEN/TC 327/WG 5. In general, method performance criteria are generated for collaborative trials or for single laboratory validation (SLV) studies. The first case describes how a method performs when used by several laboratories, giving greater confidence that the method is applicable and can be implemented with the obtained method performance. The second case only demonstrates how a method performs in a particular laboratory. As a result, SLV should be conducted with care to avoid misinterpretation. This is important as reproducibility (between laboratory variance) can only be derived from collaborative trials. Furthermore, performance parameters such as limit of detection (LOD) and limit of quantification (LOQ) are laboratory specific as the quantitative measurement capability strongly depends on the instrumental conditions used by each laboratory.

This document relates to current EU legislation concerning performance requirements of analytical methods for mycotoxins in food and feed chain, such as Regulation (EC) No 2017/625 on official controls performed to ensure the verification of compliance with feed and food law, animal health and animal welfare rules [13] as well as Regulation (EC) No 401/2006 laying down the methods of sampling and analysis for the official control of the levels of mycotoxins in foodstuffs [14]. The latter is specific for food, however as recent legislation on some mycotoxins does not specify the intended final use, either food or feed, rather the raw commodity (Commission Recommendation 2013/165/EU on the presence of T-2 and HT-2 toxin in cereals and cereal products) performance requirements for analytical methods are in the future anticipated to be equivalent independent of the commodities' destination purpose.

Further this criteria approach document also relates to pre-existing documents such as the ISO 5725-series, CR 13505 [15] and CEN/TR 16059 [16].

1 Scope

This document specifies performance criteria for the selection of single-laboratory validated or collaborative study validated methods of analysis of mycotoxins in feed. The terms and definition of the relevant parameters for method validation are included. The performance requirements and characteristics are provided. This document could 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 mycotoxins.

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.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— IEC Electropedia: available at http://www.electropedia.org/

— ISO Online browsing platform: available at https://www.iso.org/obp/ui

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

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: ISO 5725-1:1994, 3.6, see [17], modified; 2002/657/EEC, see [18], modified]

3.2
applicability
scope of the analytical method; description of the analytes, matrices, and concentration ranges (mass fractions) for which a method of analysis can 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})

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

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

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

[SOURCE: ISO 5725-1:1994, 3.8, see [17], modified; Eurachem 2014, see [19], modified]

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

Note 1 to entry: These conditions include that the materials have a suitable homogeneity and acceptable stability that is documented.

Note 2 to entry: The larger the number of participating laboratories, the greater the confidence that can be placed in the resulting statistical parameters.

Note 3 to entry: Vague terms such as “round-robins”, “intercalibrations”, “ring tests”, etc. should not be used. Studies involving several laboratories with each preparing its own test materials and using its own methods, are sometimes called cooperative studies.

[SOURCE: ISO/IEC 17043:2010, 3.4, see [20], modified]

3.4.1
collaborative study
inter-laboratory comparison aiming at the evaluation of the performance characteristics of a method

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.

Note 3 to entry: The reported results are used to estimate the performance characteristics of the method, such as precision parameters like repeatability (within laboratory) and reproducibility (between laboratories). When necessary and possible, other pertinent characteristics such as systematic error and/or recovery can be reported. These can include bias, recovery, internal quality control parameters, sensitivity, limit of quantification, and applicability.

[SOURCE: 2002/657/EEC, see [18], modified]

3.4.2
proficiency testing
evaluation of participant performance against pre-established criteria by means of inter-laboratory comparisons

Note 1 to entry: In practise the study consists of one or more analyses or measurements by a group of laboratories on one or more homogeneous, stable test samples by the method selected or used by each laboratory.

Note 2 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 3 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 [18], modified; ISO/IEC 17043:2010, 3.7, see [20], modified]

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

Note 1 to entry: The assigned values are usually the reference value (best estimate of the "true value") to a quantity (concentration/mass fraction) in the test material, usually with a stated uncertainty.

3.5
limit of detection
x_{LOD}
minimum amount or concentration of the analyte in a test sample which can be detected reliably but not necessarily quantified, as demonstrated by a collaborative trial or other appropriate validation

Note 1 to entry: Guidelines for the sound generation of LOD values can be found in [23].

Note 2 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.

Note 3 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.

[SOURCE: Thompson et al. 2002, see [21], modified; ISO 24276:2006, 3.1.6, see [22], modified]

3.6
limit of quantification
x_{LOQ}
lowest concentration or amount of the analyte in a test sample which can be quantitatively determined with an acceptable level of precision and accuracy, as demonstrated by a collaborative trial or other appropriate validation

Note 1 to entry: Rules for the sound generation of LOQ values can be found in [23].

Note 2 to entry: Measurements below LOQ are not void of information content. However, they are not of use for method criteria assessment

[SOURCE: Thompson et al. 2002, see [21], modified; ISO 24276:2006, 3.1.7, see [22], modified]

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

[SOURCE: VAM/III/5626/94, see [24]]

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

3.9
precision
closeness of agreement between independent test results obtained under stipulated conditions

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.

[SOURCE: ISO 5725-1:1994, 3.12, see [17]]

3.9.1
repeatability
***RSD*$_r$**
precision under repeatability conditions

[SOURCE: ISO 5725-1:1994, 3.13, see [17]]

3.9.1.1
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 5725-1:1994, 3.14, see [17]]

3.9.1.2
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.

[SOURCE: ISO 5725-1:1994, 3.15, see [17]]

3.9.1.3
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:

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

where:

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

3.9.1.4
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:

$$r = 2\sqrt{2} \times s_r \tag{4}$$

[SOURCE: ISO 5725-1:1994, 3.16, see [17]]

3.9.2
intermediate precision
precision under within-laboratory reproducibility conditions

3.9.2.1
within-laboratory reproducibility conditions
conditions where test results are obtained with the same method on identical test items on different days with preferably different operators using different equipment

3.9.2.2
within-laboratory reproducibility standard deviation
***s*$_{Ri}$**
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