SLOVENSKI STANDARD
SIST-TP CEN/TR 17421:2019

01-december-2019

Krma: metode vzorčenja in analize - Priporočila za organizacijo in vrednotenje medlaboratorijskih primerjalnih shem za multirezidualne analizne metode

Animal feeding stuffs: Methods of sampling and analysis - Recommendations for the organization and evaluation of collaborative studies for multi-analyte methods of analysis

Futtermittel - Probenahme- und Untersuchungsverfahren - Ringversuchsvorgaben für Multi-Analyt-Untersuchungsverfahren

Aliments pour animaux : Méthodes d'échantillonnage et d'analyse - Recommandations pour l'organisation et l'évaluation des études comparatives interlaboratoires utilisant des méthodes d'analyses multianalytes

Ta slovenski standard je istoveten z: **CEN/TR 17421:2019**

ICS:

65.120	Krmila	Animal feeding stuffs

SIST-TP CEN/TR 17421:2019 **en,fr,de**

TECHNICAL REPORT

RAPPORT TECHNIQUE

TECHNISCHER BERICHT

CEN/TR 17421

September 2019

ICS 65.120

English Version

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This Technical Report was approved by CEN on 9 September 2019. It has been drawn up by the Technical Committee CEN/TC 327.

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CEN-CENELEC Management Centre: Rue de la Science 23, B-1040 Brussels

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European foreword

This document (CEN/TR 17421:2019) 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 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of an identical text or by endorsement, at the latest by March 2020, and conflicting national standards shall be withdrawn at the latest by March 2020.

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

One of the important parts of the development of a European standard method of analysis is the collaborative study to validate this method. This study should provide sufficient information whether the method is fit for its intended purpose and on the performance characteristics that can be expected in practice. At the same time the necessary effort for the study organizer and the participating laboratories should be kept at a minimum. This guideline is to provide support to those involved in designing, executing, and evaluating such studies.

General information on how to do this is already described in a number of different documents of which a non-exhaustive list can be found at the end of this document in the Bibliography. CEN/TC 327 recommends that for all collaborative studies executed under the auspices of its working groups the "AOAC guidelines for collaborative study procedures to validate characteristics of a method of analysis" [1] is used as the primary source of information for any issues not dealt with in this document. Other relevant documents have been published by ISO [2], IUPAC [3], and EURACHEM [4].

In addition, this document presents prerequisites related to the acceptance of single-laboratory validation studies, the preparation of the standard operating procedure, and the proper implementation of the analytical method by the participating laboratories to ensure the transferability of the method.

The development of methodologies such as GC-MS, LC-MS, ICP-MS, etc. has made it possible to determine multiple analytes in a single analysis (i.e. same extraction, clean up, and determination procedure). The specificities of such multi-analyte methods need to be taken into account when organizing the collaborative trial in order to minimize the workload required while covering the necessary analyte/matrix/concentrations combinations.

1 Scope

This document provides guidance to study organizers involved in designing, executing and evaluating collaborative studies for multi-analyte methods developed by the various working groups of the CEN/TC 327 “Animal feeding stuffs: Methods of sampling and analysis”. The main goal of such studies is to determine the reproducibility standard deviations for the analytes investigated in the selected matrices. They are calculated from the repeatability and the between-laboratory standard deviations determined from the study data. An additional goal may be the determination of the trueness (whenever possible).

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

3.1
material
certain combination of analyte, matrix and concentration

3.2
matrix
components of the test material other than the analyte

[SOURCE: IUPAC Gold book [5]]

Note 1 to entry: The term “matrix” may stand synonymously for “matrix-category”.

4 Prerequisites to a collaborative study

4.1 General

Collaborative studies for multi-analyte methods require a lot of effort from the study organizer and the participants. They may require extensive personnel and material resources. Every effort should be made to ensure the success of the study.

4.2 Performance characteristics from single-laboratory validation

The fitness-for-purpose of the method of analysis that is to be standardized needs to be demonstrated by a comprehensive and well-designed single-laboratory validation study. This study is to be executed according to the internationally agreed rules, covering relevant combinations of analytes, matrices and concentrations.

A single-laboratory validation study should provide for each analyte the following performance characteristics: selectivity, working range, analytical sensitivity, precision (repeatability and intermediate precision), and trueness. The estimation of such performance characteristics is described in international documents [4] [6]. Only when the low end of the working range approaches very low

levels, and if deemed necessary by the responsible TC working group, the limits of detection and/or quantification should explicitly be determined following accepted guidance [7].

Materials with reliable reference values – covering the concentration range of interest – should be used for the estimation of trueness.

4.3 Standard operating procedure

Before starting a collaborative study a draft of the standard operating procedure (SOP) shall be available. It shall be clear, concise, unambiguous, and complete. It shall be understandable to the members of the working group and the potential participants. The quality of this SOP is crucial to the success of the study.

All relevant details of the analytical method shall be described. Critical steps should be identified, clearly addressed, and acceptable ranges of variation shall be defined. The draft SOP should comply with the structure and format specified in ISO 78-2 [8], including instructions on how to calculate and express test results.

Prior to implementation, the SOP should be reviewed by the responsible TC working group. Suggested comments and improvements – also from participating laboratories of a possible pre-trial – should be implemented in the final version of the SOP.

Any change of technical details in the SOP after finishing the validation study might invalidate the data collected during that study.

5 Design of the collaborative study

5.1 Test materials

5.1.1 Selection of test materials

The adequate selection of test materials (different analyte/matrix/concentration combinations) for the collaborative study is critical as it ultimately defines the applicability and working range of the method. The investigated test materials shall be representative of real samples, in terms of composition, matrix, target species, concentration, and interferences.

It would be ideal if:

— The concentration levels in the test materials covered the working range of the method for each analyte/matrix-category combination as much as possible.

— Several different concentration levels were validated when for one matrix-category a broader concentration range (e.g. larger two orders of magnitude) can occur in practice.

— Materials of a slightly lower and slightly larger concentration were included bracketing concentration levels that are tolerance limits or specification levels.

However, in the case of the validation of multi-analyte methods realistic, yet suitable (minimum of five), numbers of materials are required to minimize the workload.

Typically, an appropriate design should take into account matrix similarities, target analyte concentration ranges (at least around the tolerance and/or legislative limits), matrix interferences and instrumental specificities (e.g. ionization suppression or enhancement in LC-MS/MS).

While all matrices should be represented, the variety of matrices should be given preference over the levels of concentrations in each matrix. This may result in complex test materials. If not all the analytes that the method may be able to detect will appear at the same time in the same real test material then it is not necessary to have all analytes in each validation test material. Preferably, every analyte should

appear at least five times in the validation test materials. If this is not possible, a lower number should be agreed upon by the responsible TC working group.

If the scope of the method includes the determination of levels close to the detection limit, a blank material should be included (when available).

In the case of mycotoxins or plant toxins, test materials with naturally incurred analytes should be used if available. This may also be the case e.g. for feed additives, pesticides or process contaminants. If such incurred test materials are not available at all or not at the required concentration they should be prepared by fortifying ("spiking") an existing material, e.g. analyte-free, missing analytes or with analytes at too low concentrations. The procedure to be followed should be defined by the study organizer and applicability be verified during the single-laboratory validation study. The resulting test material needs to mimic the behaviour of real materials.

The concentration for a given analyte in a test material may be outside of the working range. While the SOP should clearly indicate the working range for each analyte and the test portion size to be used, it should also include provisions for possibly different test portion size or an additional dilution in such cases.

When the SOP contains various modular experimental paths (e.g. different extraction approaches for different matrices), which participating laboratories may select from, the selection of test materials shall be such that for each module a sufficient variety of test materials is included.

5.1.2 Production of test units

The total amount of bulk material to be prepared depends on the number and size of test units required. The test unit size depends on the test portion size, and whether laboratories should be able to repeat the sample preparation. The test unit number depends on the number of test units per laboratory (e.g. blind duplicates), and the anticipated number of participating laboratories.

Once the total number of test units has been determined, an additional 10 % of units should be foreseen as safety margin in order to replace lost or damaged test units (if needed). In case the test materials are sufficiently stable an even larger number of units could be prepared to allow other laboratories to validate the implementation of the method.

The bulk material for the validation study shall be prepared, homogenized and split into test units in accordance with generally accepted rules. Repeated comminution and mixing with appropriate equipment which does not contaminate the test materials (e.g. avoid metal abrasion when heavy metals are to be determined) should be performed to minimize heterogeneity.

The test units – marked with unambiguous identifiers – should be shipped to the laboratories under appropriate conditions in clean containers and robust packaging. It shall be ensured that no substance from packing material or utilities for test unit marking will contaminate the test material.

5.1.3 Characterization of test units

According to ISO 13528 [9], reference values of every measurand (analyte/matrix combination) may be derived from:

— formulation (amounts used in a solution or other mixture of ingredients with known analyte content);

— the certified value(s) of a certified reference material;

— direct comparison of the material with certified reference materials;

— measurement by a reference laboratory or reference method; or

— consensus of expert laboratories.