SLOVENSKI STANDARD
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Analize živil - Merila za validacijo metod za določanje mikotoksinov v enem laboratoriju

Food analysis - Performance criteria for single laboratory validated methods of analysis for the determination of mycotoxins

Untersuchung von Lebensmitteln - Leistungskriterien für Einzel-labor validierte Verfahren zur Bestimmung von Mykotoxinen

Analyse des produits alimentaires - Critères de performance des méthodes validées monolaboratoires d'analyse des mycotoxines

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Food analysis - Performance criteria for single laboratory validated methods of analysis for the determination of mycotoxins

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This Technical Report was approved by CEN on 19 June 2010. It has been drawn up by the Technical Committee CEN/TC 275.

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Foreword

This document (CEN/TR 16059:2010) has been prepared by Technical Committee CEN/TC 275 “Food analysis - Horizontal methods”, the secretariat of which is held by DIN.

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Introduction

Within the European Union, regulatory and scientific interest in mycotoxins has undergone a development in the last decade from autonomous national activity towards more EU-driven activity with a structural and network character. Harmonized EU limits now exist for several dozens of mycotoxin-food combinations, and this number will further grow in the coming years. The direct or indirect influence of European organizations and programmes on the EU mycotoxin regulatory developments has become significant. For example the position of CEN with regards to mycotoxin regulations has been strengthened as a consequence of the European Commission's "Mandate for standardization addressed to CEN in the field of methods of analysis for mycotoxins in food" [1]. The new mandate falls within the framework of regulation EC no 882/2004 [2]. This regulation stipulates that methods for sampling and analysis used in the context of official control shall comply with relevant Community rules or, if no such rules exist, with internationally recognized rules or protocols, for example those that CEN has accepted. The view of the European Commission on CEN standards is clear: "*The establishment of standardized methods of analysis is of utmost importance to guarantee a uniform application and control of the European legislation in all Member States. Standardized methods of analysis are an indispensable element in guaranteeing a high level of food safety*". In the annex of the mandate a number of methods of analysis, for which standardized procedures are necessary, have been specifically mentioned. In addition to these tasks, a special task has been indicated: *The preparation of a review of updates and extended performance criteria for methods of analysis of mycotoxins*.

The objective is to produce a complementary version of CEN report, CR 13505, *Food analysis — Biotoxins — Criteria of analytical methods of mycotoxins* [3], which has been the basis for stipulating performance criteria for mycotoxin methods in current EU legislation.

Complementary to the currently accepted approach, where method performance statistics are obtained from formal interlaboratory validation studies as defined within CR 13505, the concept presented in this document has single-laboratory validation as the primary source of method performance statistics.

The highly preferred option, by default, is for all standards developed through CEN/TC 275/WG 5 "Biotoxins" to contain method performance data generated from formal interlaboratory validation studies undertaken to either the Harmonised IUPAC Protocol (1995) or ISO 5725:1994. However, it is recognised that in some circumstances the interlaboratory validation study approach is impracticable. Additional method performance criteria should be developed in order to aid laboratories undertaking official food or feed control and also to help guide CEN/TC 275/WG 5 when considering the standardisation of methods supported by single-laboratory validation data. This document is not intended to replace CR 13505:1999. It will be complementary.

Some of the criteria in this document could have influence on the conclusions regarding the acceptability of validation studies that have already been performed and that were the basis for recent CEN standards on various mycotoxins. Therefore a comparison was made using criteria for single-laboratory validation instead of criteria for interlaboratory validated methods. Validation parameters such as RSD_{WLR}, RSD_r, and trueness/recovery were considered. A large majority of these CEN standards still show acceptable performance using the criteria written down in this document.

1 Scope

This Technical Report gives criteria for single laboratory validated methods of analysis for the determination of mycotoxins. The criteria and topics covered are accuracy, trueness, recovery, precision, measurement uncertainty, selectivity, applicability, linearity, limit of detection, limit of quantification, sensitivity, ruggedness, specificity. This report also contains information on terms and definitions, validation, standardization procedures and interlaboratory studies by international organizations (e.g. AOAC, CEN, ISO, IUPAC, IDF). Confirmatory methods and screening methods are described. The validation criteria specified for mycotoxins in general are given.

2 Terms and definitions

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

2.1
accuracy
closeness of agreement between a test result and the accepted reference value

NOTE 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.

[ISO 5725-1:1994, see [4]]

2.2
trueness
closeness of agreement between the average value obtained from a large series of test results and an accepted reference value

[ISO 5725-1:1994, see [4]]

2.3
recovery

$$\text{recovery} = \frac{c_{\text{f, meas}} - c_{\text{unf, meas}}}{c_{\text{known}}} \times 100\ \% \qquad (1)$$

where

$c_{\text{f, meas}}$ is the measured concentration (mass fraction) in fortified material;

$c_{\text{unf, meas}}$ is the measured concentration (mass fraction) in unfortified material;

c_{known} is the known increment in concentration (mass fraction)

NOTE 1 The amount added should be a substantial fraction of, or more than, the amount present in the unfortified material. Ideally, the unfortified material should contain no measurable level of the analyte under test.

NOTE 2 A true or assigned value is known only in cases of spiked or fortified materials, certified reference materials, or by analysis by another (presumably unbiased) method. The concentration (mass fraction) in the unfortified material is obtained by direct analysis or by the method of standard additions. In other cases, there is no direct measure of bias, and consensus values derived from the collaborative study itself often can be used for the reference point.

[CR 13505:1999, see [3]]

2.4
precision
closeness of agreement between independent test results obtained under stipulated conditions

NOTE 1 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 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 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.

[ISO 5725-1:1994, see [4]]

2.5
repeatability
precision under repeatability conditions

[ISO 5725-1:1994, see [4]]

2.6
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

[ISO 5725-1:1994, see [4]]

2.7
within-laboratory reproducibility
precision under within-laboratory reproducibility conditions

2.8
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

[ISO 5725-1:1994, see [4]]

2.9
reproducibility
precision under reproducibility conditions

NOTE 1 It is a measure of the dispersion of the distribution of test results under reproducibility conditions.

NOTE 2 Similarly "reproducibility variance" and "reproducibility coefficient of variation" could be defined and used as measures of the dispersion of the test results under reproducibility conditions.

[ISO 5725-1:1994, see [4]]

2.10
reproducibility conditions
conditions where test results are obtained with the same method on identical test items in different laboratories with different operators using different equipment

[ISO 5725-1:1994, see [4]]

2.11
Horwitz ratio
HorRat
normalized performance parameter indicating the acceptability of methods of analysis with respect to among-laboratory precision (reproducibility)

NOTE 1 It is the ratio of the observed relative standard deviation among laboratories calculated from the actual performance data, RSD_R (%), to the corresponding predicted relative standard deviation calculated from the Horwitz equation. It is more or less independent of analyte, matrix, method, and time of publication.

NOTE 2 See [5].

2.12
RSD$_r$
relative standard deviation, calculated from results generated under repeatability conditions

$$RSD_r = \frac{S_r}{c_{\text{mean}}} \times 100\ \% \qquad (2)$$

where

S_r is the standard deviation, calculated from results generated under repeatability conditions;

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

[Based on CR 13505:1999, see [3]]

2.13
RSD$_{WLR}$
relative standard deviation, calculated from results generated under within-laboratory reproducibility conditions (WLR)

$$RSD_{WLR} = \frac{S_{WLR}}{c_{\text{mean}}} \times 100\ \% \qquad (3)$$

where

S_{WLR} is the standard deviation, calculated from results generated under within-laboratory reproducibility conditions;

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

2.14
RSD$_R$
relative standard deviation, calculated from results generated under reproducibility conditions

$$RSD_R = \frac{S_R}{c_{\text{mean}}} \times 100\ \% \qquad (4)$$

where

S_R is the standard deviation, calculated from results generated under reproducibility conditions;

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

[Based on CR 13505:1999, see [3]]

2.15
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

NOTE 1 See [6].