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English Version

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

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

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

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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

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

where

$c_{f, \text{meas}}$ 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