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Validation and interpretation of analytical methods, migration testing and analytical data for materials and articles in contact with food - Part 1: General considerations

Validation et interprétation des méthodes d'analyse, essais de migrations et données analytiques des matériaux et objets en contact avec les denrées alimentaires - Partie 1 : Considérations générales

Validierung und Interpretation analytischer Verfahren, Migrationsprüfung und analytischer Daten von Werkstoffen und Bedarfsgegenständen in Kontakt mit Lebensmitteln - Teil 1: Allgemeine Betrachtungen

This Technical Report was approved by CEN on 16 January 2006. It has been drawn up by the Technical Committee CEN/TC 194.

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Foreword

This document (CEN/TR 15356-1:2006) has been prepared by CEN /TC 194, "Utensils in contact with food", the secretariat of which is held by BSI.

Introduction

0.1 Requirement for validation of analytical methods for enforcement of Directives

Regulation (EC) No. 1935/2004[1] has laid down the requirements that may be included in specific Directives to protect human health. It allows for specific Directives to set overall migration limits and specific limits on the migration of certain constituents or groups of constituents into foodstuffs.

Commission Directive 90/128/EEC[2] and its subsequent amendments (e.g.[3]) introduced specific migration limits for more than 300 substances. A consolidation of these directives has since been issued as Commission Directive 2002/72/EC[4]. In addition, some substances are subject to a maximum permitted quantity of the residual substance in the material or article. Some substances are subject to group limits. Continuously, additional substances are being evaluated and added to the Directive.

New technical dossiers are being prepared for substances which could eventually be listed in future amendments to Directive 2002/72/EC. Methods of control will be required for the majority of the abovementioned substances.

The two Food Control Directives (European Council Directive 89/397/EEC[5] and Council Directive 93/99/EEC[6]) require that methods used for control purposes must be correctly and fully validated. So far only the methods developed by CEN as parts of EN 13130 have been so validated. Methods developed in the project sponsored by DG Research (SM&T project, MAT1-CT92-0006, "Development of Methods of Analysis for Monomers") have only been validated by two competent laboratories. Most methods from technical dossiers have only limited validation data at best.

This Technical Report considers the background to whether or not acceptable validation of analytical methods could be achieved faster and at less cost. The Technical Report also considers the need for validation of the whole test procedure for enforcement purposes, for compliance purposes, and for the creation of data for risk assessment purposes. It should be noted that the considerations apply to both overall as well as specific migration.

The list of current legislation currently adopted by the Commission is given in Annex A.

The list of current methods adopted by CEN/TC 194/SC 1 is given in Annex B.

0.2 Variability in the migration contact stage

The determination of migration from plastics is quite unlike other measurement tasks in ensuring food safety and quality. Reliable measurements depend upon more than simply having validated analytical methods for measuring chemical concentrations in foods. The Directives allows that, as an alternative to the analysis of foodstuff itself, migration testing can be carried out with food simulants applied under conditions which simulate actual use of the plastic material or article with food. This introduces many potential sources of variability in the final migration value. These are discussed in Clause 8.

0.3 Quality of data submitted for risk assessment purposes

Migration data is usually an important part of the petition submitted for a risk assessment carried out by the Scientific Committee on Food (since 2003, by the European Food Safety Authority, EFSA). For new substances it is unlikely that a fully validated method in food simulants will exist. A single laboratory (in-house) system of validation is required as part of the demonstration that the data submitted is of adequate quality. For example, validation of a method's intended use, the determination of accuracy and precision, usually involves replicate analyses of appropriate matrices

spiked with known amounts of the additive at concentrations similar to those encountered in the migration studies and determination of the percentage recovery of the spiked additive.

Where data are supplied to other authorities, e.g. the US-FDA, the data has to be applicable and acceptable to those authorities.

Even when a validated method exists there is still the need for the laboratory carrying out the test to ensure the migration testing carried out within the laboratory does not suffer from excessive error. The possibility of error may be reduced by taking part in proficiency testing schemes. Proficiency testing schemes aim to assess the competence of laboratories to carry out migration testing. At present there is at least one scheme which is known to operate in this area. This is the Food Analysis Performance Assessment Scheme (FAPAS) operated by the FAPAS Secretariat, Central Science Laboratory, Sand Hutton, York (UK).

Laboratories carrying out these methods will also be able to demonstrate their general competence by being accredited to EN ISO/IEC 17025:2005, which is administered by the appropriate Accreditation Agencies in the European Countries. For overall migration testing, samples of plastics with known overall migration values are available from the IRMM, Geel, Belgium. Spectra and a table of physical properties of the monomers and additives listed in Directives have been published to assist ensuring that substances used for calibration are of adequate and known purity [7], [8].

1 Scope

This Technical Report gives guidance in support of Directives adopted by the European Union in the Food Contact Materials Sector and is intended to aid Food Control Authorities and industry enforce and comply with those Directives.

2 Form of regulations

2.1 General

The EU Directives on food contact plastics, provide for various types of quantitative restrictions i.e. specific migration limits (SML, expressed as mg (of substance) /kg of food), overall migration limit (OML, expressed as mg/kg of food or mg/dm^2 of surface) and maximal quantity of the substance in the finished plastic article referred either to the quantity of article (QM, expressed as mg/kg of article) or to area of the surface in contact with the foodstuffs (QMA, expressed as mg/dm² of surface). The determination of these quantities implies various procedural steps e.g. sampling, migration tests with different experimental conditions (OML, SML) or extraction (QM, QMA) as well the usual multi-step analytical determination. Each of these steps is subject to a certain variability and an overall variability will affect the value found by one laboratory (repeatability) or by more than one laboratories (reproducibility). In the past at the level of the Standing Committee for Foodstuffs a discussion took place on the method of analysis for vinyl chloride. The Commission proposed then that the variability should be expressed as "Reproducibility" but the majority of Member States were in favour of the "Repeatability". Therefore the Commission services decided to avoid any further scientific discussion on this issue and decided to propose a new term, "Analytical Tolerance" which shall comprise the variability due to all the above-mentioned procedural steps. Until now no Member States objected to this choice and no fundamental problems were raised from its application. Three options have been chosen by the Commission services as regards the various existing quantitative restrictions:

a) restrictions affected by a specified analytical tolerance,

b) restrictions affected by an unspecified analytical tolerance, and

c) restrictions not affected by any analytical tolerance.

The three options and their background are explained in 2.2, 2.3 and 2.4.

2.2 Restriction and specified analytical tolerance

This case applies to the overall migration limit, where the value of the OML in fatty simulants (60 mg/kg (ppm) or 10 mg/dm^2) is accompanied by an analytical tolerance of 20 mg/kg (ppm) (or 3 mg/dm^2). In this case the variability should be added to the limit value and, only if the value found is greater than 80 mg/kg (ppm) (=60+20) or 13 mg/dm^2 (=10+3), the article is considered not in compliance with the Directive. The choice to increase the OML by the value of the tolerance was due to the variability of the analysis.

NOTE This approach has the disadvantage that as the variability of sampling and analytical procedures becomes less, the overall limit becomes, effectively greater. However it is possible to change the value of the analytical tolerance by an amendment of the Plastics Directive. For example, as practical experience was gained and as both standardised methods and certified reference materials became available it became clear that many laboratories struggled to meet the analytical tolerance value of 1 mg/dm^2 set for tests using volatile simulants. Consequently, Commission Directive 2001/62/EC was issued which, based on expert judgement rather than any statistical evaluation of the available results, raised this tolerance figure to 2 mg/dm^2. The same problem would exist if an EN rather than a Directive establishes the value of the variability. If no value is specified, this issue is no longer harmonised and this should also be considered as disadvantage. The Member States and professional organisations requested, at unanimity, that an analytical tolerance should be fixed.