SLOVENSKI STANDARD

SIST-TP CEN/TR 15645-1:2008

01-april-2008

Papir, karton in lepenka v neposrednem stiku z živili - Umerjanje za preskus vonja - 1. del: Vonj

Paper and board intended to come into contact with foodstuffs - Calibration of the odour test - Part 1: Odour

Papier und Pappe vorgesehen für den Kontakt mit Lebensmitteln - Kalibrierung der Geruchsprüfung - Teil 1: Geruch und Aroma

Papiers et cartons destinés en entrer en contact avec les denrées alimentaires - Etalonnage de l'essai de flaveur atypique - Partie 1: Odeur

Ta slovenski standard je istoveten z: CEN/TR 15645-1:2008

ICS:

67.250

85.060

SIST-TP CEN/TR 15645-1:2008 **en,fr,de**

TECHNICAL REPORT

RAPPORT TECHNIQUE

TECHNISCHER BERICHT

CEN/TR 15645-1

January 2008

ICS 67.250; 85.060

English Version

Paper and board intended to come into contact with foodstuffs - Calibration of the odour test - Part 1: Odour

Papiers et cartons destinés en entrer en contact avec les denrées alimentaires - Etalonnage de l'essai de flaveur atypique - Partie 1: Odeur

Papier und Pappe vorgesehen für den Kontakt mit Lebensmitteln - Kalibrierung der Geschmacksprüfung - Teil 1: Geruch und Aroma

This Technical Report was approved by CEN on 13 August 2007. It has been drawn up by the Technical Committee CEN/TC 172.

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Ref. No. CEN/TR 15645-1:2008: E

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Foreword

This document (CEN/TR 15645-1:2008) has been prepared by Technical Committee CEN/TC 172 “Pulp, paper and board”, the secretariat of which is held by DIN.

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Introduction

Paper and board, intended to be in contact with food, may have characteristic odour that can migrate via the airspace to the food packed in it. The purpose of testing the odour of paper and board is to establish whether the material to be tested possesses an inherent odour when kept at room temperature.

In order to gain reliable results from the sensory evaluation, the performance of a sensory panel assessing the odour of paper and board needs to be validated. This can be implemented through a training procedure by using spiked calibration samples prepared according to the given instructions.

This document consists of:

— protocol to prepare the calibration samples (spiked paper) for sensory evaluation of odour;

— description of the training procedure for a sensory panel in the use of the calibration samples;

— instructions for sensory evaluation of calibration samples before and after training.

This guide is meant to be used in connection with the European Standard EN 1230-1. The guidance given in this document is only a recommendation. Please note that the calibration samples, the preparation of which is described, can be also applied in other ways than described in this document.

This guide has been devised and collaboratively tested in the context of the EU research project CALIBSENSORY (Growth programme, Measurement and Testing activity, GRD2-2000-30015) and it is the sole responsibility of its authors. It does in no way represent the views of the Commission or its services. Published results of the project are available at http://www1.kcl.fi/euproj/calib.html .

1 Scope

This Technical Report specifies a written protocol to prepare calibration samples for assessment of odour released by a paper or board sample, and how to train the panel in the use of these calibration samples.

The general outline of the testing procedure consists of sensory assessment of the odour samples without formal training by a selected panel, followed by training of the panel, and finally sensory assessment of the odour samples after training by the same sensory panel.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 5496, *Sensory analysis — Methodology — Initiation and training of assessors in the detection and recognition of odours*

ISO 6658, *Sensory analysis — Methodology — General guidance*

ISO 8586-1, *Sensory analysis — General guidance for the selection, training and monitoring of assessors — Part 1: Selected assessors*

3 Terms and definitions

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

3.1
assessor
any person taking part in a sensory test

[see ISO 5492:1992]

3.2
base paper
paper that has minimal number of sensory properties

3.3
calibration procedure
protocol of calibration samples and written instructions to train selected assessors with calibration samples i.e. calibrate the panel

3.4
calibration sample
base paper that is spiked with spiking compounds at a certain concentration

3.5
control sample
a hidden reference sample served blind coded to the assessors among the calibration samples, and prepared according to the procedure of the calibration samples but without the spiking compound

3.6
odour
organoleptic attribute perceptible by the olfactory organ on sniffing certain volatile substances

[ISO 5492:1992]

3.7
multicomparison test
test where the assessor is asked to give a rating of the intensity of the difference in taste or odour between test portions for analysis and a known reference sample

3.8
reference sample
calibration sample without any spiking compound

NOTE This is presented to the assessors labelled as "Reference" and served to the assessors before the calibration samples.

3.9
selected assessor
assessor chosen for his/her ability to perform a sensory test

[see ISO 5492:1992]

3.10
spiking compound
a volatile chemical compound having a specific odour

NOTE In this case hexanal is the selected spiking compound.

3.11
spiking method
a method for spiking the base paper with spiking compounds

4 Principle

A training process validation for a sensory panel has to be conducted with freshly made calibration samples, prepared by spiking an odourless base paper with different concentrations of hexanal. The panel performance in evaluating the odour intensity of hexanal is determined before and after a formal training step. The effectiveness of training can then be determined.

The test procedure consists of 3 steps:

a) first step: assessment of calibration samples before training;

b) second step: training procedure;

c) third step: assessment of calibration samples after training.

Detailed instructions for general test procedure, sample preparation and sensory evaluation are provided within this Technical Report, and must be carefully followed to ensure validity of results.

5 Materials and reagents

5.1 General

Odourless base paper is used as a carrier for different concentrations of the spiking compound diluted with the solvent. In this Technical Report, hexanal is used as the spiking compound and triacetin as the solvent.

5.2 Base paper

The base paper used in the odour test shall be suitable for food use. The paper shall respect the following criteria:

— odourless

— porous.

One example for the base paper is coffee filter paper made of bleached chemical pulp, including wet strength resin and grammage of approximately 52 g/m^2.

5.3 Spiking compound

Hexanal (CAS number 66-25-1, 98 %) is used as the spiking agent in the odour test. The odour of hexanal is described as being green, fruity, bitter and grassy. Hexanal is often found in rancid fats (in foods) and in fibre-based materials.

5.4 Solvent

Triacetin (CAS number 102-76-1) shall be used as a solvent for diluting the spiking compound. It is assessed to be almost odourless, and related to its other characteristics, suitable for this purpose.

6 Equipment

6.1 General

All equipment used shall be free from odour and only in use for sensory analysis (see ISO 6658). Special care is required to ensure that lids, seals and inserts do not become a source of odour.

Equipment and glass flasks shall be cleaned between sessions. Any glassware used shall be washed with minimum-odour cleaning agents and dried by placement in oven at a temperature > 100 °C and left to evaporate for at least 2 h. All other equipment shall be washed with minimum-odour cleaning agents and left to dry at room temperature.

6.2 Micro-syringes

Micro-syringes in capacity from 20 µl to 100 µl.

6.3 Glass flasks

Glass flasks in 500 ml in capacity. The shape of the flask is not as essential as the volume. Brown glass bottles with lids of polytetrafluorethylen or Erlenmeyer flasks with narrow neck and conical ground joint and stopper may be used.

The flasks (if needed) are covered by aluminium foil for preventing the influence of the appearance of the sample. Each sample is served to each assessor in a separate flask, therefore 5 flasks are required for each assessors at each session.

6.4 Aluminium foil

Aluminium foil used for wrapping the glass flasks (if needed) shall be free from odour.

7 Preparation of calibration samples

7.1 General

Prepare for each five sessions five samples: the reference sample plus four samples for assessment. These are summarised, along with their concentrations, in table 1.

Table 1 — Calibration samples and concentrations of hexanal (µg hexanal/paper sample)

sample	µg/sample
reference	0
N 0 (control sample)	0
N 1	0,04
N 2	0,80
N 3	8,0

For the three spiked samples, base paper should be spiked at various concentrations of hexanal diluted in triacetin, the concentrations being 0,04 µg, 0,8 µg and 8 µg hexanal/sample. The reference (given reference) and control samples (hidden reference) shall be odour flasks with base paper and triacetin but without hexanal.

For each of the five samples, the number of prepared test portions (flasks) has to be equal to the number of assessors on the panel, the recommended panel size varying between 10 and 12. The flasks should be covered with aluminium foil and coded with three-digit random numbers prior to preparing the individual test portions. These have to be presented individually to each assessor. Examples for blinding codes and experimental designs for each session are presented in the annexes.

7.2 Preparation of the spiking solutions

7.2.1 General

The following sets of stock solutions will be needed. Stock solution K can be used over three consecutive days only. The other stock solution K 2 and the batch solutions for each sample have to be freshly made each day.

7.2.2 Stock solution K (800 mg/l)

Dissolve 80 mg (100 µl) of hexanal in about 50 ml of triacetin, then fill up with triacetin to make total volume of 100 ml.

Stock solution K has to be stored at + 4 °C, and can be used for three consecutive days.

7.2.3 Solution K 2 (40 mg/l)

Dilute 1 ml of stock solution K in 20 ml of triacetin (i.e. 1 ml K/20 ml triacetin).

Stock solution K 2 has to be stored at + 4 °C and shall be prepared daily.

7.2.4 Spiking solutions

To create batch solutions for the spiked samples, dilute solution K 2 as presented in Table 2.

Table 2 — Preparation of batch solutions for spiking the calibration samples

sample	K 2 ml	triacetin ml	description
N1	0,1	9,9	to create a batch of N1 solution, dilute 0.1 ml of K 2 stock solution with 9.9 ml triacetin
N2	1	4	to create a batch of N2 solution, dilute 1 ml of K 2 stock solution with 4 ml triacetin
	K, ml		
N3	1	9	to create a batch of N3 solution, dilute 1 ml of K stock solution with 9 ml triacetin

Sample batches for N1 to N3 shall be prepared daily.

The reference and control samples, also presented to the assessors during the sensory assessment, will be prepared using triacetin without any added hexanal.

7.3 Preparation of calibration samples

The sample flasks should be prepared in a completely odourfree space (see ISO 6658). To prepare the calibration samples the following scheme has to be followed:

1) Reference test paper must first be prepared by cutting into squares of 1 dm² (10 cm × 10 cm) cm. For each session, five pieces of test paper (each 1 dm²) **for each assessor** (one for each sample) will be needed.

2) Reference test paper (1 dm²) shall be folded in double thickness and bent in accordion style. Place the bent paper into the flask.

3) Sample solution has to be pipetted onto the test paper. The amount of solution per paper is 100 µl, and should be pipetted in four drops, 25 µl each, to guarantee even distribution of the spiking solution on the paper. Close the flask immediately.

4) Store the flasks for 3 h at (22 ± 3) ºC in the dark, wrapped in aluminium foil (or use brown coloured bottles) to avoid the possibility of any chemical reactions prior to the sensory assessments.