SLOVENSKI STANDARD
oSIST prEN 17600:2020

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Papir, karton in lepenka, namenjeni neposrednemu stiku z živili - Ugotavljanje svetlobne obstojnosti papirja, kartona in lepenke, ki vsebujejo optična belila - Analiza s tekočinsko kromatografijo visoke ločljivosti s fluorescenčno detekcijo

Paper and board intended to come into contact with foodstuffs - Determination of the fastness of fluorescent whitened paper and board - Analysis by high-performance liquid chromatography with fluorescence detection

Papier und Pappe für den Kontakt mit Lebensmitteln - Bestimmung der Farbechtheit von optisch aufgehelltem Papier und Pappe - Analyse durch Hochleistungsflüssigkeitschromatographie mit Fluoreszenzdetektion

Papiers et cartons destinés à entrer en contact avec des denrées alimentaires - Détermination de la solidité des papiers et cartons blanchis fluorescents - Analyse par chromatographie liquide à haute performance et détection par fluorescence

Ta slovenski standard je istoveten z: **prEN 17600**

ICS:

67.250	Materiali in predmeti v stiku z živili	Materials and articles in contact with foodstuffs
85.060	Papir, karton in lepenka	Paper and board

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EUROPEAN STANDARD

NORME EUROPÉENNE

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Paper and board intended to come into contact with foodstuffs - Determination of the fastness of fluorescent whitened paper and board - Analysis by high-performance liquid chromatography with fluorescence detection

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

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European foreword

This document (prEN 17600:2020) has been prepared by Technical Committee CEN/TC 172 “Pulp, paper and board”, the secretariat of which is held by DIN.

This document is currently submitted to the CEN Enquiry.

1 Scope

This document describes procedures for the testing of migration of fluorescent whitening agents from paper and board intended to come into contact with foodstuffs. The method is based on liquid chromatography with fluorescence detection for quantification. The document is applicable to four different types of fluorescent whitening agents, diaminostilbene hexasulfonate, diaminostilbene tetrasulfonate, diaminostilbene disulfonate and distyrylbiphenyl.

2 Normative references

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

EN 648, *Paper and board intended to come into contact with foodstuffs — Determination of the fastness of fluorescent whitened paper and board*

EN ISO 186, *Paper and board — Sampling to determine average quality (ISO 186)*

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:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

4 Symbols and abbreviated terms

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

FWA	Fluorescent whitening agent
ACN	Acetonitrile
DAS	Diaminostilbene

5 Principle

A sample is brought into contact with glass fibre papers, which have been saturated with a test fluid and placed under load for a given time. The test fluids are water, acetic acid 3 %, ethanol 10 % and ethanol 95 %. After short or long contact duration, fluorescent whitening agent are extracted from the glass fibre and analysed by liquid chromatography-fluorescence detection. The four different types of fluorescent whitening agent analysed are hexasulfonate, tetrasulfonate, disulfonate and distyrylbiphenyl without specific separation of the fluorescent whitening agent with the same degree of sulfonation.

6 Interferences

FWA are extremely sensitive to light. A short contact with UV light can isomerise and destroy these compounds. Therefore, pay special attention to avoid any contact with light. In order to avoid these interferences, work in significant darkness or use special light without UV. The flasks shall be protected with aluminium foil.

7 Test materials and equipment

7.1 General

The used materials shall be free of FWA.

EXAMPLE Common laboratory glassware could be rinsed with water/acetonitrile 50/50 v/v before use.

7.2 Materials

The following materials shall be used.

7.2.1 Pipette of 100 µl to 1 000 µl.

7.2.2 Volumetric brown glass flasks of 50 and 100 ml.

7.2.3 Teflonlined cap brown glass vial, single use.

7.2.4 25 ml brown glass flask.

7.2.5 Whatmann or MN Glassfibre (Under testing at CTP).

7.2.6 Glass plates, 60 mm × 90 mm.

7.2.7 Polyethylene film, uncoloured and transparent.

7.2.8 Mass, 1 kg.

7.3 Apparatus

The usual laboratory apparatus and, in particular, the following, shall be used.

7.3.1 Balance: Analytical of accurately weighting 0,000 1 g.

7.3.2 High Pressure Liquid Chromatograph.

7.3.3 Fluorescence detector.

7.3.4 Column C18: for example ODS, 250 mm × 4,6 mm, particle size 5µm, 120 Å.

7.3.5 Guard column C18: for example Hypersil ODS.

8 Reagents

Use, as far as available, reagents of analytical quality, or better. Verify by blank determinations.

8.1.1 Water for HPLC-Fluorescence detection.

8.1.2 Acetonitrile CAS n° 75-05-8.

8.1.3 Ammonium acetate CAS n° 631-61-8.

8.1.4 Distilled or deionised water.

8.1.5 Acetic acid 3 % m/v.

8.1.6 Ethanol 95 % v/v.

8.1.7 Ethanol 10 % v/v.

8.1.8 Fluorescent whitening agents (FWAs). An example of different types of fluorescent whitening agents is given in Annex A.

9 Preparation of calibration solutions

9.1 Stock solutions of commercially available individual whitening agent at 1g/L

Prepare stock solutions of individual component by weighing about 100 mg of a whitening agent into 100 ml volumetric flask. Fill the volumetric flask with 50/50 v/v ACN/water, shake to dissolve and dilute to the mark with the solvent. Protect the flask from light with aluminium foil. Prepare one solution for each different types of FWA.

Stock solutions shall be stored protected from light at 4 °C and replaced after 1 week.

9.2 Individual intermediate solution of commercially available whitening agent at 10 mg/L

For each of the 4 stock solutions, transfer 1 ml into a 100 ml volumetric flask and dilute to the mark with the solvent 50/50 v/v ACN/water. Protect the flask with aluminium foil.

The 4 individual intermediate solutions shall be freshly prepared for analysis and stored protected from light at 4 °C.

9.3 Purity of the stock solution

The amount of active compounds shall be known from the supplier of fluorescent whitening agent.

9.4 Calibration standard solutions of fluorescent whitening agent

Five standard solutions shall be prepared by transferring respectively: 0,125 ml; 0,25 ml; 0,5 ml and 1 ml of the 4 individual intermediate solutions in 50 ml volumetric flasks and 0,1 ml of the 4 individual intermediate solutions in a 100 ml volumetric flask and adjust the volume with the solvent. The respective concentrations of the calibration standard solutions are: 10 µg/l; 25 µg/l; 50 µg/l; 100 µg/l and 200 µg/l of each FWA. At the end of the preparation, the calibration standards shall be protected from light.

The calibration solutions shall always be freshly prepared and stored protected from light at 4 °C.

10 Sampling

Sampling shall be carried out in accordance with EN ISO 186.

11 Preparation of sample

Take from the sample under investigation several test pieces 50 mm × 20 mm by cutting or punching so that smooth edges are obtained.

12 Migration procedure

The migration shall be made with the 4 simulants:

— Aqueous simulant: water;

— Alcoholic simulant: ethanol 10 % v/v;

— Fatty simulant: ethanol 95 % v/v;

— Acid simulant: acetic acid 3 % m/v.

WARNING — The simulants listed here are different from the one used in EN 648.

Immerse two sheets of glass fibre paper (7.2.5) in a test fluid. Remove the sheets after saturation and free the sheets from excess fluid by wiping on the rim of the container.

Lay one sheet of glass fibre paper with its smooth side upwards on the glass plate (7.2.6). Lay the test piece immediately on top and cover it with the second saturated sheet of glass fibre paper so that the smooth side of the glass fibre paper is in contact with the test piece again. Place a second glass plate (7.2.6) on top, wrap the total assembly in polyethylene film (7.2.7) to prevent the edges from drying out, load it with a mass of 1 kg (7.2.8) and allow it to stand for a define time at (23 ± 2)° C with protection against direct light penetration. Examples of time contact are given in Table 1.

If test pieces of a grammage > 140 g/m² are to be investigated, an appropriate even number of glass fibre paper layers (7.2.5) is used so that the total of their grammages just exceeds the grammage of the test piece.

Construct the assembly, with each glass fibre paper being individually saturated and wiped, and arranged in such a way that the same number of glass fibre papers are in contact with both sides of the test piece.

Once the time of contact is completed, open the assembly. Lay the glass fibre papers on 3 adjacent glass rods with a diameter of 8 mm to 10 mm, with the side, which was in contact with the test piece upwards, cover them without contact to prevent light penetration and air-dry at ambient temperature.

In parallel, a blank migration is performed, using the same migration procedure but without paper sample in contact with the glass fibre papers.

Example of time of contact are given in Table 1.

Table 1 — Examples of time contact

Procedures A – C	Simulation of the contact
Procedure A 24 h at 23 °C	long duration contact
Procedure B 4 h at 23 °C	medium time contact
Procedure C 10 min. at 23 °C	short time contact

13 Glass fibre paper extraction

For each migration, 2 samples shall be analysed. For each sample, the glass fibre paper in contact with recto and verso are analysed separately. For each glass fibre paper, the following extraction procedure is applied:

— Before to begin the extraction, a brown glass flask is filled with the calibration solution at 50 µg/l and placed without aluminium protection at the place where the extraction is done in order to see if

an isomerisation and degradation can take place. It is important to make the extraction in darkness or under special light without UV.

— The glass fibre paper in contact with the sample is cut in pieces of 1 cm^2 and 2 cm^2. Place the pieces in a brown glass flask of 25 ml and cover with aluminium foil to protect the flask from light. Introduce 20 ml of water/ACN 50/50 v/v. Place the flask 10 min in an ultrasonic bath. Collect the extract in a volumetric flask of 50 ml. Add 20 ml of water/ACN 50/50 v/v to the first flask and place it again in an ultrasonic bath for 10 min. Pool the extract with the first extract in the 50 ml volumetric flask and fill to the mark with the solvent.

— The extracts shall be stored protected from light at 4 °C until being analysed no later than 48 h.

— When all the extractions are finished, the solution for isomerisation check is covered with aluminium foil. The solution is analysed with the extracts.

14 Chromatographic analysis procedure

14.1 HPLC conditions

An example of conditions of analysis is given hereafter.

14.1.1 Chromatographic parameters

a) Elution flow: 0,8 ml/min;

b) Elution solvent:

1) Ammonium acetate at 5 mmol/l; A

2) Acetonitrile / ammonium acetate at 5 mmol/l 90/10 v/v; B

c) Gradient program:

89/11 (A/B), 10 min linear gradient to 68/32 (A/B), 5 min, 1 min linear gradient to 89/11 (A/B), 10 min;

Time of analysis: 26 min;

d) Injection loop: 20 µl.

14.1.2 Fluorescence Detection conditions

— Excitation wavelength: 340 nm;

— Emission wavelength: 440 nm.

14.2 Calibration

The vials shall be protected from light during the analysis.

Inject 20 µl of each of the calibration standard solutions.

Under the analytical conditions as described previously (14.1), the 4 studied whitening agent family presented the following retention time:

AO DAS hexasulfonate	≅ 2 min
AO DAS tetrasulfonate	≅ 4 min
AO DAS disulfonate	≅ 13 min
AO DSBP	≅ 15 min

Calculate the area of each peak detected. Trace the calibration curve for each compound: peak area response versus the accurate concentration of the calibration standard solution injected. From the experimental area data, create a linear regression through the standard points.

14.3 Analysis of the extracts

The vials shall be protected from light during the analysis.

Inject 20 µl of each test sample extracts, 20 µl of the blank extract and 20 µl of the isomerisation witness solution.

If the isomerisation witness solution has a response different from the calibration solution by more than 20 %, the test shall be repeated.

Under the analytical conditions as described previously, Calculate the area of each peak detected.

14.4 Expression of results

Determine the concentration of individual compounds in the blank control sample and in the test samples from the each linear equation of the calibration.

Determine the concentration of individual compounds that have migrated from the sample to the glass fibre for each type of FWA:

$$C_{AOi} = \frac{Cc \times V \times P}{S \times 0,8}$$

where

CAOi	is the concentration of FWA / dm^2 in µg;
Cc	is the concentration of the extract of commercial FWA / l in µg;
P	is the purity of the commercial standard solution (example: 0,2 for 20 % of active compounds);
V	is the volume of extract, 0,050 l;
S	is the surface of paper in contact with the glass fibre, 0,1 dm^2;
0,8	corresponds to the mean recovery of OBA for the extraction step.

EXAMPLE Purity of the commercial standard solution, 0,2 for 20 % of active compounds.