SLOVENSKI STANDARD
SIST EN 17163:2019

01-april-2019

Vlaknine, papir, karton in lepenka - Določevanje primarnih aromatičnih aminov (PAA) v vodnem ekstraktu z metodo LC-MS

Pulp, paper and board - Determination of primary aromatic amines (PAA) in a water extract by a LC-MS method

Papier, Pappe und Faserstoff - Bestimmung von primären aromatischen Aminen in Wasserextrakten mittels LC-MS

Pâtes, papier et carton - Détermination des amines aromatiques primaires (AAP) dans un extrait aqueux par méthode LC-MS

Ta slovenski standard je istoveten z: **EN 17163:2019**

ICS:

85.040	Vlaknine	Pulps
85.060	Papir, karton in lepenka	Paper and board

SIST EN 17163:2019 **en,fr,de**

EUROPEAN STANDARD

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English Version

Pulp, paper and board - Determination of primary aromatic amines (PAA) in a water extract by a LC-MS method

Pâtes, papier et carton - Détermination des amines aromatiques primaires (AAP) dans un extrait aqueux par méthode LC-MS

Papier, Pappe und Faserstoff - Bestimmung von primären aromatischen Aminen in Wasserextrakten mittels LC-MS

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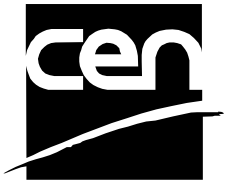

EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

CEN-CENELEC Management Centre: Rue de la Science 23, B-1040 Brussels

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European foreword

This document (EN 17163:2019) has been prepared by Technical Committee CEN/TC 172 “Pulp, paper and board”, the secretariat of which is held by DIN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by August 2019, and conflicting national standards shall be withdrawn at the latest by August 2019.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN shall not be held responsible for identifying any or all such patent rights.

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Introduction

Primary aromatic amines (PAAs) can occur in paper and board materials as residual basic chemicals for the production of azodyes and azo pigments. Furthermore, hydrolysis products of isocyanates especially from paper laminates may be a possible source.

A draft of a European Standard EN 13130-XX concerning the “Determination of Primary Aromatic Amines (PAAs) in Food Simulants” has been prepared by the standardization committee CEN/TC 194/SC1/TG9.

When preparing this standard it has been assessed by the laboratories participating that the photometric screening method described in part A of the draft of standard EN 13130-XX is not applicable for the examination of water extracts of paper and board materials due to interferences of extractable paper components which caused irreproducible test results.

Due to the advantages of the MS/MS-detection regarding selectivity and sensitivity the determination of primary aromatic amines by means of LC-MS/MS was found to be capable for water extracts of paper and board materials when preparing this standard.

1 Scope

This document describes two representative methods for the determination of the extractable amount of specific primary aromatic amines (PAA) in a water extract of paper, board and pulp samples by means of HPLC with MS/MS detection which basically differ concerning the choice of the mobile and stationary phases. Deviating from this standard further methods may be applicable if it can be shown that comparable results can be achieved. A validation should be carried out by each laboratory.

It is applicable for the determination of the 22 primary aromatic amines (PAA) mentioned in the annex of Directive 2002/61/EC of 19th July 2002 amending Council Directive 76/769/EEC relating to restrictions on the market and use of certain dangerous substances and preparations (azocolourants) which are classified as carcinogenic categories 1A and 1B according to the CLP regulation and aniline.

The method described by this standard should be also applicable for the determination of further primary aromatic amines (PAA). A validation for every further analyte has to be done by the laboratory using this method.

The extractable amount of a primary aromatic amine (PAA) is expressed in mg PAA per litre water extract. This method is suitable for the determination of PAA with a working range of about 0,001 mg/l – 0,020 mg/l water extract.

Deviating from this standard further methods may be applicable if it can be shown that comparable results can be achieved. A validation should be carried out by each laboratory.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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.

EN 645, *Paper and board intended to come into contact with foodstuffs — Preparation of a cold water extract*

EN 647, *Paper and board intended to come into contact with foodstuffs — Preparation of a hot water extract*

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- IEC Electropedia: available at http://www.electropedia.org/
- ISO Online browsing platform: available at http://www.iso.org/obp

4 Principle

4.1 General

The primary aromatic amines (PAA) are extracted with water according to EN 645 or EN 647. The corresponding extracts can generally be used for the HPLC-MS quantification without further preparation steps.

4.2 Interferences

As some of the PAAs are sensitive to light, it has to be ensured that the samples are protected from light at all stages of treatment.

5 Apparatus

5.1 An instrument or item of apparatus is listed only if it is special, or made to a particular specification, usual laboratory glassware and equipment which is assumed to be available.

5.2 High performance liquid chromatograph with a **MS-detector** and **electro spray ionization**

6 Reagents

6.1 General

All reagents shall be of recognized analytical quality unless otherwise stated.

6.2 Acetonitrile; [75-05-8]; LC-MS-grade

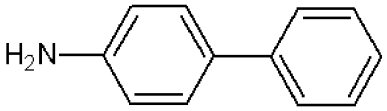
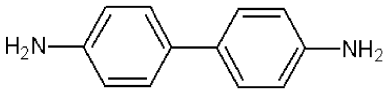
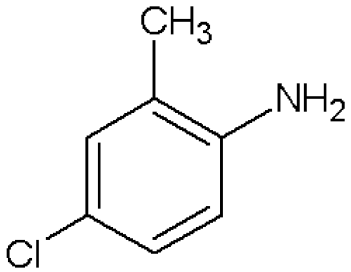
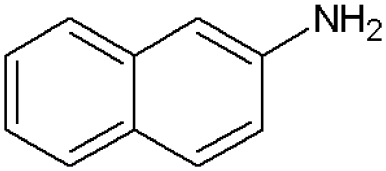
6.3 Deionized water

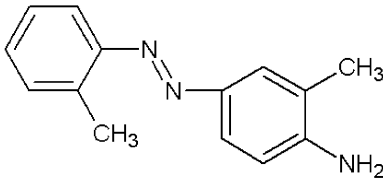
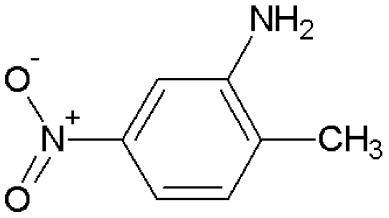
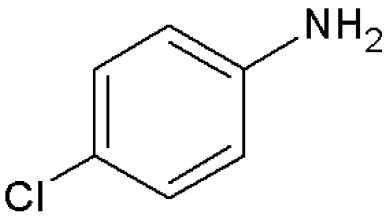
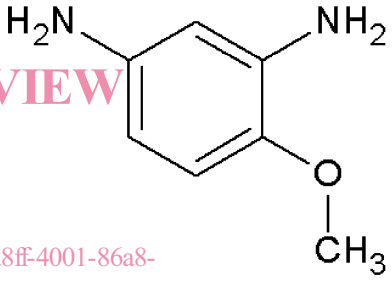
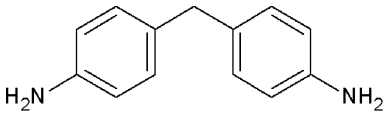
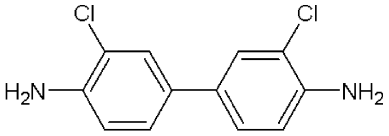
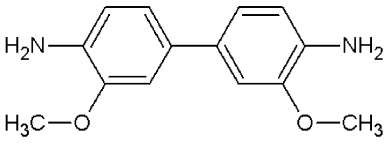
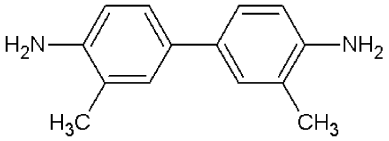
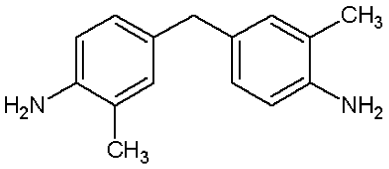
6.4 Pentafluoropropionic acid; [422-61-0] (for system-example 1)

6.5 Formic acid; [64-18-6] (for system-example 2)

6.6 Reference substances

Table 1 — Reference Substances

No.	Name	Abbreviation	CAS No.	Structure	MW
1	4-Aminobiphenyl	4-ABP	92–67–1		169,2
2	Benzidine	BNZ	92–87–5		184,2
3	4-Chloro-o-toluidine	4-CoT	95–69–2		141,6
4	2-Naphthylamine	2-NA	91–59–8		143,2

No.	Name	Abbreviation	CAS No.	Structure	MW
5	o-Aminoazotoluene	o-AAT	97–56–3		225,3
6	2-Amino-4-nitrotoluene	2-ANT	99–55–8		152,2
7	4-Chloroaniline	4-CA	106–47–8		127,6
8	2,4-Diaminoanisole	2,4-DAA	615–05–4		138,2
9	4,4`-Diaminodiphenylmethane	4,4'-DADPM	101–77–9		198,3
10	3,3`-Dichlorobenzidine	3,3`-DCB	91–94–1		253,1
11	o-Dimethoxybenzidine	o-DMB	119–90–4		244,3
12	3,3`-Dimethylbenzidine	3,3`-DMB	119–93–7		212,3
13	3,3`-Dimethyl-4,4`-diaminodiphenylmethane	3,3`-D-4,4`-DADPM	838–88–0		226,3