SLOVENSKI STANDARD
SIST EN 15980:2011

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Kakovost zraka - Določevanje usedanja benzo[a]antracena, benzo[b]fluorantena, benzo[j]fluorantena, benzo[k]fluorantena, benzo[a]pirena, dibenzo[a,h]antracena in indeno[1,2,3-cd]pirena

Air quality - Determination of the deposition of benz[a]anthracene, benzo[b]fluoranthene, benzo[j]fluoranthene, benzo[k]fluoranthene, benzo[a]pyrene, dibenz[a,h]anthracene and indeno[1,2,3-cd]pyrene

Luftqualität - Bestimmung der Deposition von Benz[a]anthracen, Benzo[b]fluoranthen, Benzo[j]fluoranthen, Benzo[k]fluoranthen, Benzo[a]pyren, Dibenz[a,h]anthracen und Indeno[1,2,3-cd]pyren

Qualité de l'air - Détermination du benzo[a]anthracène, benzo[b]fluoranthène, benzo[j]fluoranthène, benzo[k]fluoranthène, benzo[a]pyrène, dibenzo[a,h]anthracène et indeno[1,2,3-cd]pyrène dans les dépôts atmosphériques

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Air quality - Determination of the deposition of benz[a]anthracene, benzo[b]fluoranthene, benzo[j]fluoranthene, benzo[k]fluoranthene, benzo[a]pyrene, dibenz[a,h]anthracene and indeno[1,2,3-cd]pyrene

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Foreword

This document (EN 15980:2011) has been prepared by Technical Committee CEN/TC 264 “Air quality”, 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of an identical text or by endorsement, at the latest by November 2011, and conflicting national standards shall be withdrawn at the latest by November 2011.

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

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Introduction

Directive 2004/107/EC [2] requires the measurement of the "total or bulk deposition" of benz[*a*]anthracene (BaA), benzo[*b*]fluoranthene (BbF), benzo[*j*]fluoranthene (BjF), benzo[*k*]fluoranthene (BkF), benzo[*a*]pyrene (BaP), dibenz[*a,h*]anthracene (DBahA) and indeno[1,2,3-*cd*]pyrene (INP) at background sites, one every 100 000 km². Wet-only sampling may be used instead of bulk sampling if it can be shown that the difference between their results is within 10 %. Where appropriate, monitoring shall be coordinated with the European Monitoring and Evaluation Programme (EMEP).

The Directive uses "total deposition" and "bulk deposition" as synonyms and defines it as the total mass of pollutants which is transferred from the atmosphere to surfaces (e.g. soil, vegetation, water, buildings) in a given area within a given time. The considered polycyclic aromatic hydrocarbons (PAH) are predominantly bound to particles. The mass of PAH bound to non sedimenting particles is thought to be low compared to the mass of the compounds bound to sedimenting wet and dry particles. Consequently the sum of the deposition of sedimenting wet and dry particles (defined as "bulk deposition" in this European Standard) and total deposition has been considered as substantially equivalent for the purposes of this document. At sites far enough from obstacles like buildings and trees the influence of specific surface characteristics on the deposition of PAH can be neglected.

This document describes the measurement of the deposition of PAH using collectors designed for bulk and wet-only deposition. The validation field test demonstrated that the funnel-bottle bulk collector provided the most reliable and robust results. Therefore this collector type has been chosen as standard collector.

1 Scope

This document specifies a method for the determination of the bulk deposition of benz[*a*]anthracene (BaA), benzo[*b*]fluoranthene (BbF), benzo[*j*]fluoranthene (BjF), benzo[*k*]fluoranthene (BkF), benzo[*a*]pyrene (BaP), dibenz[*a,h*]anthracene (DBahA) and indeno[1,2,3-*cd*]pyrene (INP), which can be used in the framework of Directive 2004/107/EC. This European Standard specifies performance requirements with which the method has to comply in order to meet the data quality objectives given in this Directive.

This document specifies methods for sampling, sample preparation and analysis using gas chromatography with mass spectroscopic detection (GC/MS) or high performance liquid chromatography with fluorescence detection (HPLC/FLD). The funnel-bottle bulk collector is used as the standard collector.

The method is applicable for deposition measurements in

- rural and remote areas,
- industrial areas,
- urban areas.

The standard is applicable in the range from a few ng/($m^2 \cdot$ d) to a few hundred ng/($m^2 \cdot$ d).

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.

EN 15549:2008, *Air quality — Standard method for the measurement of the concentration of benzo[a]pyrene in ambient air*

3 Terms and definitions

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

3.1
analysis
all operations carried out after sample preparation to determine the amount or concentration of the polycyclic aromatic hydrocarbons of interest present in the sample

3.2
bulk collector
equipment to collect bulk deposition, permanently open to the atmosphere

NOTE In this document, two types of bulk collectors are described: a funnel-bottle bulk collector and a cylindrical gauge (Bergerhoff collector).

3.3
bulk deposition
sum of the deposition of sedimenting wet and dry particles

NOTE Bulk deposition does not contain gases and non-sedimenting particles. The bulk deposition of the polycyclic aromatic hydrocarbons is considered as substantially equivalent to total deposition.

3.4
detection limit
lowest deposition rate of an analyte detectable using the specified procedure

3.5
expanded uncertainty
quantity defining an interval about the result of a measurement that can be expected to encompass a large fraction of the distribution of values that could reasonably be attributed to the measurand

[ISO/IEC Guide 98-3:2008 [9]]

3.6
field blank
artificial sample (e.g. distilled water) transported to the sampling site, mounted in the sampling unit, but not left exposed to ambient air, returned to the laboratory and worked up in the same way as the deposition sample

3.7
laboratory blank
artificial sample (e.g. distilled water) worked up in the same way as the deposition sample in the laboratory

3.8
precipitation
rain, snow, sleet, graupel, hail and dew

3.9
sample extraction
sample dissolution process to obtain a solution containing the analyte of interest in an organic solvent

3.10
sample preparation
all operations carried out on a sample, after transportation and storage, to prepare it for analysis

NOTE Such operations are, e.g. extraction, purification, evaporation, solvent exchange.

3.11
standard uncertainty
uncertainty of the results of measurements expressed as a standard deviation

[ISO/IEC Guide 98-3:2008 [9]]

3.12
uncertainty (of measurement)
measurement uncertainty
parameter associated with the result of a measurement that characterises the dispersion of the values that could reasonably be attributed to the measurement

[ISO/IEC Guide 98-3:2008 [9]]

3.13
wet-only collector
equipment to collect wet deposition, consisting of a funnel-bottle combination, open to the atmosphere only during precipitation events

4 Symbols and abbreviations

4.1 Symbols

A	area of the cross-section of the collector opening;
$\bar{c}$	mean value of the concentration of a compound in a solution;
$\bar{c}_i$	mean value of the concentration of a compound, obtained by laboratory i;
c_j	concentration of a compound in the solution j;
$d_{(1,j)}$	deposition in collector 1;
$d_{(2,j)}$	deposition in collector 2;
D_{a}	deposition rate of compound a;
$D_{\mathrm{L,a}}$	detection limit of compound a;
$D_{\mathrm{M\,,a}}$	minimal detectable mass of compound a;
k	coverage factor;
m_{a}	mass of compound a;
$m_{\mathrm{a,d}}$	detected mass of compound a;
$\overline{m}_{\mathrm{a}}$	mean of laboratory blank value;
$m_{\mathrm{a},i}$	individual laboratory blank value for compound a;
n	number of measurements;
rss	residual sum of squares from the fit;
s_i	within-laboratory standard deviation;
s_{L}	between-laboratory standard deviation;
s_{d}	between-collector standard deviation;
s_{R}	reproducibility standard deviation;
s_{ri}	standard deviation of repeated measurements of laboratory i;
S_{m}	mass of surrogate standard, added to the sample;
S_{d}	mass of surrogate standard detected in the sample extract;
$S_{\mathrm{lb,a}}$	standard deviation of laboratory blank value for compound a;
t	sampling duration;
$t_{\mathrm{n-1;0,95}}$	Student factor for n measurements and a 95 % confidence interval;

$u_{bc,j}$	between-collector uncertainty for collector type j;
u_{D}	uncertainty of measured depositions;
u_{M}	standard uncertainty of the deposition measurement;
$U_{\mathrm{p}}(y)$	expanded uncertainty of the measured value y with a coverage probability p;
$w_{bc,j}$	relative between-collector uncertainty for collector type j;
w_{fit}	random relative uncertainty of the fits.

4.2 Abbreviations

BaA	Benz[*a*]anthracene
BaP	Benzo[*a*]pyrene
BeP	Benzo[*e*]pyrene
BbF	Benzo[*b*]fluoranthene
BjF	Benzo[*j*]fluoranthene
BkF	Benzo[*k*]fluoranthene
CHR	Chrysene
CRM	Certified reference material
CPP	Cyclopenta[*cd*]pyrene
DAD	Diode array detection
DBacA	Dibenz[*a,c*]anthracene
DBahA	Dibenz[*a,h*]anthracene
DBajA	Dibenz[*a,j*]anthracene
DCM	Dichloromethane
EMEP	Co-operative Programme for Monitoring and Evaluation of the Long-range Transmission of Air pollutants in Europe (European Monitoring and Evaluation Programme)
FLD	Fluorescence detection
GC	Gas chromatography
HPLC	High performance liquid chromatography
INP	Indeno[1,2,3-*cd*]pyrene
MS	Mass spectrometry
PAH	Polycyclic aromatic hydrocarbons

PTFE	Polytetrafluoroethylene
QA/QC	Quality Assurance/Quality Control
SOP	Standard operating procedure
SPE	Solid phase extraction
TRI	Triphenylene
WMO/GAW	World Meteorological Organization / Global Atmosphere Watch

5 Principle

Deposition samples for the determination of specified PAH compounds are taken in funnel-bottle bulk collectors for a measured period of time (see Clause 7). Samples are prepared using liquid-liquid or solid phase extraction with organic solvent (see 8.2). Identification and quantification of the substances are performed by either GC/MS or HPLC/FLD (see 8.3).

6 Apparatus, reagents and materials

6.1 Reagents

High purity reagents:

- solvents: e.g. acetone, acetonitrile, cyclohexane, dichloromethane (DCM), methanol, toluene, water;
- solid substances: NaCl, anhydrous Na_2SO_4.

6.2 Materials

- Ordinary laboratory materials;
- wiping material, e.g. glass wool, quartz wool or pre-extracted cotton wool.

6.3 Sampling equipment

Funnel-bottle bulk collectors consist of a cylindrical funnel and a sample collection vessel (see A.2). The vertical section of the funnel shall be at least as high as its diameter. The collection vessels shall have a cylindrical vertical section of sufficient height to avoid sampling losses resulting from splashing. The collector is permanently open to the atmosphere. The collector shall be equipped with a device for keeping birds off.

The area of the cross-section of the funnel opening and the size of the sample collection vessel shall be adjusted to the precipitation amount which is expected in the chosen sampling period in order to avoid overflow. Typical sampling periods vary between one week and one month. The area of the cross-section of the funnel opening shall be large enough to provide sufficient sample for analysis at a minimum precipitation height of 1 mm per week.

NOTE 1 The funnel diameter should be at least 15 cm.

The funnel opening shall be at least 1,5 m above ground. For areas that receive high snowfall accumulations, the collector may be raised onto a platform above the snow.

NOTE 2 An extra large and deep cylindrical collector may be used for snow sampling (see [1]).

Funnel and sample collection vessel shall be made of glass. If connectors, which come into contact with sampled material, are not made of glass, only PTFE or other fluoroelastomers are permissible. All parts shall be able to be easily cleaned.

6.4 Laboratory equipment

6.4.1 General

Ordinary laboratory apparatus and the items given in 6.4.2 and 6.4.3 are required.

6.4.2 Apparatus for sample extraction

Liquid-liquid or solid phase extraction apparatus (see B.2.2.2 and B.2.2.3).

6.4.3 Apparatus for sample analysis

6.4.3.1 HPLC/FLD apparatus

Liquid chromatograph fitted with injection system, a reverse phase column suitable for PAH analysis, a temperature controlled oven, a pump system and an FLD. Furthermore a system for solvent degassing (internal or external) is required.

NOTE If the amounts of PAH are high, also the HPLC/DAD technique can be used.

6.4.3.2 GC/MS apparatus

Gas chromatograph with split/splitless injector or on-column injector, a capillary column suitable for PAH analysis, and a mass selective detector.

7 Sampling

7.1 General

The validation field test was carried out using funnel-bottle bulk collectors, cylindrical gauges and wet-only collectors (see Annex A). The results showed that the deposition rates of PAH measured with the funnel-bottle bulk collector were in most cases higher than those measured with the cylindrical gauges or the wet-only collector. The between-collector uncertainty in general was the lowest for the funnel-bottle bulk collector (see D.4.2). Therefore the funnel-bottle bulk collector shall be used as the standard collector for the determination of the deposition of BaA, BbF, BjF, BkF, BaP, DBahA and INP.

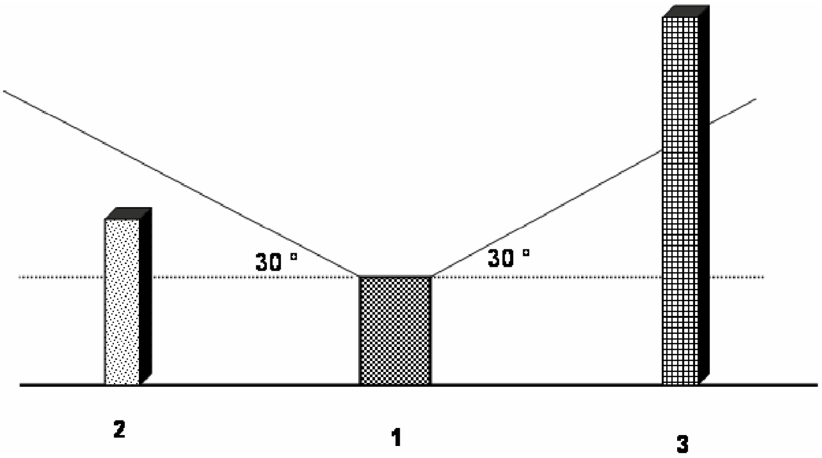
NOTE When the deposition rates were high and the climatic conditions moderate the results of cylindrical gauges were similar to those obtained with the funnel-bottle bulk collectors. When the precipitation was high and the climatic conditions were cold the results of wet-only and funnel bottle bulk collectors were similar. Therefore in special cases, if equivalence with the funnel-bottle bulk collector can be shown different types of collectors like wet-only collectors and cylindrical gauges can be used to measure the deposition of PAH.

7.2 Sampling location

Macroscale and microscale siting shall follow the guidelines of Directive 2004/107/EC [2], which are harmonized with the guidelines from EMEP [1] and WMO/GAW [3] for deposition measurements in rural areas.

The collector shall as far as possible not be exposed in areas where strong winds occur like shores, cliffs and top of hills, but it shall also not be sheltered by tall trees or buildings. The flow around the collector should be unrestricted, without any obstructions affecting the airflow in the vicinity of the collector. The criteria depend on the site characteristic:

- rural site: as far as practicable no obstacles such as trees, buildings, hedges or topographical features above 30° (see Figure 1) from the rim of the collector, which may give rise to updraughts or downdraughts;
- urban and industrial site: as far as possible away from buildings, trees and other obstacles.



Key

1	collector
2	obstacle, acceptable
3	obstacle, too high

Figure 1 — Position of obstacles which affect sampling

7.3 Sampling requirements

Handle sampling equipment such that contamination is avoided. Avoid contact of skin with the glassware of the exposed side of the collector, e.g., by wearing cotton gloves during mounting and dismounting of the collector. The procedures used in the field test are described in B.3 and B.4.

NOTE 1 In order to avoid breaking of glassware due to freezing 100 ml of a saturated NaCl solution may be added before sampling, if low temperatures are expected.

If materials like leaves or insects clog the pipe of the funnel, remove them carefully with tweezers and rinse them with water. The rinsing water is added to the collected rain water. Also rinse the funnel carefully.

Cleaning of the funnel can be done either at the sampling site or in the laboratory depending on local conditions. Parts of the collector which are transported to the laboratory shall be carefully wrapped, e.g. with aluminium foil.

NOTE 2 The sampled water amount can be determined by weighing the collector before and after exposure. The precipitation rate can be calculated from the water amount, the cross-section of the funnel opening and the exposure time. These data can be compared with meteorological data to check the plausibility of the results.

Store equipment in the field so that contamination is prevented, e.g. wrapped in aluminium foil, and kept it in an as clean and dust-free place as possible.

Clean the funnel between exposures with wiping material moistened with acetone or methanol, followed by rinsing with water. Send the funnel to the laboratory for thorough cleaning, if necessary.

The sampling duration shall not exceed 1 month.

SOPs are given in Annex B.