SLOVENSKI STANDARD
SIST-TS CEN/TS 17638:2021

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Emisije nepremičnih virov - Določevanje masne koncentracije formaldehida - Ročna metoda

Stationary source emissions - Determination of the mass concentration of formaldehyde - Manual method

Emissionen aus stationären Quellen - Manuelles Verfahren zur Bestimmung der Massenkonzentration von Formaldehyd - Referenzverfahren

Emissions de sources fixes - Détermination de la concentration massique en formaldéhyde - Methode manuelle

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Stationary source emissions - Manual method for the determination of the mass concentration of formaldehyde - Reference method

Emissions de sources fixes - Méthode manuelle pour la détermination de la concentration massique en formaldéhyde - Méthode de référence

Emissionen aus stationären Quellen - Manuelles Verfahren zur Bestimmung der Massenkonzentration von Formaldehyd - Referenzverfahren

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

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European foreword

This document (CEN/TS 17638:2021) has been prepared by Technical Committee CEN/TC 264 “Air quality”, the secretariat of which is held by DIN.

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Introduction

Formaldehyde is a carcinogenic pollutant that is generated in different industrial sectors, like energy industries (combustion plants (e.g. for wood and gas), combustion engines (gas engines and turbines)), chemical industry (e.g. formaldehyde production), food industry (e.g. smoking plants), wood industry (e.g. production of wood-based panels or wood pellets) and thus contained in emissions of these processes.

Currently, no European (EN) or International (ISO) Standard exists for the continuous or periodic measurement of formaldehyde emissions, which are being addressed, e.g. by the European Commission in its implementing decision 2015/2119 [1] establishing best available techniques (BAT) conclusions, under Directive 2010/75/EU [2], for the production of wood-based panels.

Instead, different national methods for formaldehyde measurements are currently applied, e.g. US EPA M316 [3], VDI 3862 Part 4 [4], VDI 3862 Part 6 [5], and FD X43-319 [6], all of them based on sampling in aqueous absorption solutions. Several comparison studies have shown that the equivalence of these methods is not ensured.

This measurement method is specified as a Technical Specification because currently no sufficient validation data are available. A comprehensive validation programme has been developed and will be carried out as soon as the funding is ensured (see Annex C).

1 Scope

This document specifies the reference method for the determination of the concentration of formaldehyde in emissions from stationary sources. Waste gas samples are taken by absorption in water and subsequently analysed by spectrophotometry or HPLC. The method applies to waste gases in which the formaldehyde concentration is 2 mg/m^3 to 60 mg/m^3, on dry basis, at the reference conditions of 273 K and 101,3 kPa.

The specific components and the requirements for the measuring system are described. A number of performance characteristics with associated minimum performance criteria are specified for the measuring system.

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 13284-1:2017, *Stationary source emissions - Determination of low range mass concentration of dust - Part 1: Manual gravimetric method*

EN 15259:2007, *Air quality - Measurement of stationary source emissions - Requirements for measurement sections and sites and for the measurement objective, plan and report*

EN ISO 14956, *Air quality – Evaluation of the suitability of a measurement procedure by comparison with a required measurement uncertainty (ISO 14956)*

ISO/IEC Guide 98-3:2008, *Uncertainty of measurement - Part 3: Guide to the expression of uncertainty in measurement (GUM:1995)*

3 Terms and definitions

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

3.1
absorber
device in which formaldehyde is absorbed into an absorption solution

Note 1 to entry: For formaldehyde absorption wash bottles are used as absorbers.

3.2
limit of quantification
lowest amount of an analyte that is quantifiable with a given confidence level

Note 1 to entry: For a manual method the limit of quantification is usually calculated as ten times the standard deviation of blank measurements provided that the blank value is negligible. This corresponds to a confidence level of 95 %.

3.3
analytical repeatability
closeness of the agreement between the results of successive measurements of the same measure and carried out under the same conditions of measurement

Note 1 to entry: Analytical repeatability conditions include:

— the same measurement procedure;

— the same laboratory;

— the same sampling equipment, used under the same conditions and at the same location;

— repetition over a short period of time.

Note 2 to entry: Analytical repeatability may be expressed quantitatively in terms of the dispersion characteristics of the results.

Note 3 to entry: In this document the analytical repeatability is expressed as a value with a level of confidence of 95 %.

3.4
calibration
set of operations that establish, under specified conditions, the relationship between values of quantities indicated by a measuring method or measuring system, and the corresponding values given by the applicable reference

3.5
certified reference material
CRM
reference material, accompanied by documentation issued by an authoritative body and providing one or more specified property values with associated uncertainties and traceabilities, using valid procedures

[SOURCE: ISO/IEC Guide 99:2007 [7]]

3.6
chemical blank
content of an unexposed sample of the absorption solution, plus reagents that are added to the solution before analysis if necessary

3.7
combined uncertainty
standard uncertainty attached to the measurement result calculated by combination of several standard uncertainties

Note 1 to entry: According to the principles laid down in ISO/IEC Guide 98-3.

3.8
emission limit value
ELV
limit value given in regulations such as EU Directives, ordinances, administrative regulations, permits, licences, authorisations or consents

Note 1 to entry: ELV can be stated as concentration limits expressed as half-hourly, hourly and daily averaged values, or mass flow limits expressed as hourly, daily, weekly, monthly or annually aggregated values.

3.9
expanded uncertainty
quantity defining a level of confidence about the result of a measurement that may be expected to encompass a specific fraction of the distribution of values that could reasonably be attributed to a measurand

$U = k \times uc$

Note 1 to entry: In this document, the expanded uncertainty is calculated with a coverage factor of $k = 2$, and with a level of confidence of 95 %.

Note 2 to entry: The expression overall uncertainty is sometimes used to express the expanded uncertainty.

3.10
field blank
value determined by a specific procedure used to ensure that no significant contamination has occurred during all steps of the measurement and to check that the operator can achieve a quantification level adapted to the task

3.11
isokinetic sampling
sampling at a rate such that the velocity and direction of the gas entering the sampling nozzle are the same as the velocity and direction of the gas in the duct at the measurement point

[SOURCE: EN 13284-1:2017]

3.12
measurand
particular quantity subject to measurement

Note 1 to entry: The measurand is a quantifiable property of the stack gas under test, for example mass concentration of a measured component, temperature, velocity, mass flow, oxygen content and water vapour content.

[SOURCE: EN 15259:2007]

3.13
measurement method
method described in a written procedure containing all the means and procedures required to sample and analyse, namely field of application, principle and/or reactions, definitions, equipment, procedures, presentation of results, other requirements and measurement report

[SOURCE: EN 14793:2017 [8]]

3.14
measurement plane
plane normal to the centreline of the duct at the sampling position

Note 1 to entry: Measurement plane is also known as sampling plane.

[SOURCE: EN 15259:2007]

3.15
measurement point
position in the measurement plane at which the sample stream is extracted or the measurement data are obtained directly

Note 1 to entry: Measurement point is also known as sampling point.

[SOURCE: EN 15259:2007]

3.16
measurement port
opening in the waste gas duct along the measurement line, through which access to the waste gas is gained

Note 1 to entry: Measurement port is also known as sampling port or access port.

[SOURCE: EN 15259:2007]

3.17
measurement series
successive measurements carried out at the same measurement plane and at the same operating conditions of the industrial process

[SOURCE: EN 13284-1:2017]

3.18
performance characteristic
one of the quantities (described by values, tolerances, range, etc.) assigned to equipment in order to define its performance

3.19
reference method
RM
measurement method taken as a reference by convention, which gives the accepted reference value of the measurand

Note 1 to entry: A reference method is fully described.

Note 2 to entry: A reference method can be a manual or an automated method.

Note 3 to entry: Alternative methods can be used if equivalence to the reference method has been demonstrated.

[SOURCE: EN 15259:2007]

3.20
repeatability in the field
closeness of the agreement between the results of simultaneous measurements of the same measurand carried out with two sets of equipment under the same conditions of measurement

Note 1 to entry: These conditions include:

— the same measurement procedure;

— two sets of equipment, the performances of which are fulfilling the requirements of the reference method, used under the same conditions;

— the same location;

— implemented by the same laboratory;

— typically calculated over short periods of time in order to avoid the effect of changes of influence parameters (e.g. 30 min).

Note 2 to entry: Repeatability may be expressed quantitatively in terms of the dispersion characteristics of the results.

Note 3 to entry: In this document the repeatability under field conditions is expressed as a value with a level of confidence of 95 %.

3.21
reproducibility in the field
closeness of the agreement between the results of simultaneous measurements of the same measurand carried out with several sets of equipment under the same conditions of measurement

Note 1 to entry: These conditions include:

— the same measurement procedure;

— several sets of equipment, the performance of which fulfils the requirements of the reference method, used under the same conditions;

— the same location;

— implemented by several laboratories.

Note 2 to entry: Reproducibility may be expressed quantitatively in terms of the dispersion characteristics of the results.

Note 3 to entry: In this document the reproducibility under field conditions is expressed as a value with a level of confidence of 95 %.

3.22
standard uncertainty
uncertainty of the result of a measurement expressed as a standard deviation

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

3.23
uncertainty
parameter, associated with the result of a measurement, that characterizes the dispersion of the values that could reasonably be attributed to the measurand

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

3.24
uncertainty budget
statement of a measurement uncertainty, of the components of that measurement uncertainty, and of their calculation and combination

Note 1 to entry: An uncertainty budget should include the measurement model, estimates, and measurement uncertainties associated with the quantities in the measurement model, covariances, type of applied probability density functions, degrees of freedom, type of evaluation of measurement uncertainty, and any coverage factor.

[SOURCE: ISO/IEC Guide 99:2007 [7]]

3.25
waste gas
any gas leaving a process which is not a product (includes exhaust gas, off-gas and flue-gas)

4 Symbols and abbreviations

AHMT	4-Amino-3-hydrazino-5-mercapto-1,2,4-triazole
CRM	Certified Reference Material
DNPH	2,4-Dinitrophenylhydrazine
HPLC	High Performance Liquid Chromatography
PFA	Perfluoroalkoxy Alkane
PTFE	Polytetrafluoroethylene

5 Principle

A known volume of waste gas is extracted representatively from a duct or a stack during a certain period of time at a controlled flow rate with a heated sampling probe. A heated filter removes the particulate matter in the sampled volume, thereafter the gas stream is passed through a series of wash bottles containing water as absorption solution. The samples are analysed using one of the four analysis methods specified in Annex A.

6 Sampling strategy

6.1 General

The sampling programme shall be established following the advice and requirements of EN 15259. The following points shall be considered when preparing the sampling programme:

— the nature of the plant process, e.g. steady-state or discontinuous;

— the homogeneity of the gas effluents at the sampling sections can be performed either by using an automatic analyser or a surrogate gas (e.g. O_2, CO_2). When droplets are present, it is not necessary to perform a homogeneity test because a grid measurement is performed;

— the expected concentration to be measured and any required averaging period, both of which can influence the measuring and sampling time. Sampling time shall be in accordance with EN 15259 requirements related to the representativeness of the sample;