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SLOVENSKI STANDARD
oSIST prEN 14884:2021

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Kakovost zraka - Emisije nepremičnih virov - Določevanje celotnega živega srebra: avtomatski merilni sistemi

Air quality - Stationary source emissions - Determination of total mercury: automated measuring systems

Luftbeschaffenheit - Emissionen aus stationären Quellen - Bestimmung der Gesamtquecksilber-Konzentration: Automatische Messeinrichtungen

Qualité de l'air - Emissions de sources fixes - Détermination de la concentration en mercure total : systèmes automatiques de mesure

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13.040.40	Emisije nepremičnih virov	Stationary source emissions

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English Version

Air quality - Stationary source emissions - Determination of total mercury: automated measuring systems

Qualité de l'air - Emissions de sources fixes - Détermination de la concentration en mercure total : systèmes automatiques de mesure

Luftbeschaffenheit - Emissionen aus stationären Quellen - Bestimmung der Gesamtquecksilber- Konzentration: Automatische Messeinrichtungen

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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 14884:2021) has been prepared by Technical Committee CEN/TC 264 "Air Quality", the secretariat of which is held by DIN.

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This document will supersede EN 14884:2005.

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prEN 14884:2021 (E)

Introduction

This document describes the quality assurance procedures related to automated measuring systems (AMS) for the determination of total mercury in waste gas, in order to meet the uncertainty requirements on measured values given by regulations, e.g. EU Directives [1], national or other legislation.

This document is derived from EN 14181, which specifies general procedures for establishing quality assurance levels (QAL) for automated measuring systems (AMS) installed on industrial plants for the determination of the flue gas components and other flue gas parameters. It amends EN 14181 and provides guidance specific to total mercury measurements. It is only applicable in conjunction with EN 14181.

The calibration and validation of mercury AMS that measure the total vapour phase mercury content is based on parallel measurements with the manual method described in EN 13211. The species of mercury (elemental Hg^0 and oxidized Hg^{2+}) and the physical occurrence (gaseous, dust-bound or within droplets) can vary significantly depending on the type of process to be monitored and this is taken into account when implementing the SRM.

Annex C provides details of significant technical changes between this European Standard and the previous edition.

1 Scope

This document specifies requirements for the calibration and validation (QAL2), the ongoing quality assurance during operation (QAL3) and the annual surveillance test (AST) of automated measuring systems (AMS) used for monitoring total mercury emissions from stationary sources to demonstrate compliance with an emission limit value (ELV). This document is derived from EN 14181 and is only applicable in conjunction with EN 14181. This standard also specifies type testing (QAL1) requirements in Annex B that will be incorporated into EN 15267-3 when that standard is next revised.

This document is applicable by direct correlation with the standard reference method (SRM) described in EN 13211.

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 13211, *Air quality - Stationary source emissions - Manual method of determination of the concentration of total mercury*

EN 14181:2014, *Stationary source emissions - Quality assurance of automated measuring systems*

EN 15267-3, *Air quality - Certification of automated measuring systems - Part 3: Performance criteria and test procedures for automated measuring systems for monitoring emissions from stationary sources*

EN 17255-1, *Stationary source emissions - Data acquisition and handling systems - Part 1: Specification of requirements for the handling and reporting of data*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in EN 13211 and EN 14181 apply.

4 Symbols and abbreviations

4.1 Symbols

a	intercept of the calibration function
$\hat{a}$	best estimate of a
b	slope of the calibration function
$\hat{b}$	best estimate of b
D_i	difference between SRM value, y_i and calibrated AMS value $\hat{y}_i$
$\bar{D}$	average of D_i
E	emission limit value
h	absolute water vapour content (by volume)
i	counter
N	number of paired samples in parallel measurements
k_v	test value for variability

o	oxygen content in dry gas (by volume)
p	gauge pressure
s_D	standard deviation of the differences D_i in parallel measurements
t	Celsius temperature
t_{90}	response time
t_{cycle}	total measurement cycle time of AMS with pre-concentration
t_{sample}	sample period of AMS with pre-concentration
U_{max}	maximum permissible expanded uncertainty
x_i	ith AMS measured signal at AMS measuring conditions
$\bar{x}$	average of AMS measured signals
y_i	ith SRM measured value
$\bar{y}$	average of SRM measured values
$y_{i,s}$	ith SRM measured value at standard conditions
y_s	SRM measured value at standard conditions
$y_{s,min}$	lowest SRM measured value at standard conditions
$y_{s,max}$	highest SRM measured value at standard conditions
$\hat{y}_i$	best estimate for the "true value", calculated from the AMS measured signal x_i by means of the calibration function
$\hat{y}_{i,s}$	best estimate for the "true value", calculated from the AMS measured signal x_i by means of the calibration function at standard conditions
Δy_{max}	difference between the maximum and minimum SRM measured value at standard conditions
σ_0	standard deviation associated with the uncertainty derived from requirements of legislation

4.2 Abbreviations

AMS	automated measuring system
AST	annual surveillance test
DAHS	data acquisition and handling system
ELV	emission limit value
QAL	quality assurance level
QAL1	first quality assurance level
QAL2	second quality assurance level
QAL3	third quality assurance level
SRM	standard reference method

5 Principle

The AMS measures total gaseous mercury, both elemental and oxidized, requiring a converter to reduce oxidized Hg into elemental Hg prior to measurement of total Hg. Mercury compounds are reactive and can be adsorbed onto particulate deposits within the sampling system. Therefore, the sample is often diluted with nitrogen or air in order to aid sample transport and reduce cross-interferences from other gas components within the flue gas matrix. The Hg AMS must be capable of measuring the total concentration in μg/m³ and reporting the undiluted vapour phase Hg, regardless of speciation.

NOTE   AMS often report Hg concentration on a wet basis, i.e. the water vapour from the process sample is retained within the sample. The water content is then required in order to correct the Hg concentration to dry reference conditions. If the water content is not measured by any of the installed AMS, the use of a calculated water content may be acceptable as described in CEN/TS 17286 [2].

The general principles of quality assurance of AMS are laid down in EN 14181. These are applied within this standard with the amendments specific to mercury AMS being specified in the following sections.

In this context, an AMS is any system that continuously samples the mercury content of flue gas. This may be a system that continuously analyses for mercury, typically producing a one-minute average concentration that is based on discrete or time-integrated data sampling. Alternatively, this may be a system that pre-concentrates the mercury in the flue gas, prior to analysis, within a gold accumulator for example, with a measurement cycle of typically 2 to 10 min duration. Dual accumulators are then typically used to provide continuous sampling and analysis. Mercury specific requirements relating to QAL1 type testing are specified in Annex B noting that these provisions will be incorporated into EN 15267-3 when that standard is next revised.

Long-term sampling systems involve continuous, repetitive flue gas sampling using paired sorbent traps, located within the flue gas duct, for mercury capture, with subsequent trap analysis of the time-integrated samples. Long term sampling is typically from one day to two weeks sampling duration. The type testing, functional tests and general quality assurance requirements, applicable to long-term sampling systems, are specified in CEN/TS 17286 [2]. However, these systems also require QAL2 calibration according to EN 14181 and this standard, except for the functional tests (6.2 and 8.2 of EN 14181:2014). Alternative functional tests and quality assurance procedures are defined in CEN TS 17286 [2].

6 Calibration and validation of the AMS (QAL2)

6.1 General

The AMS shall be calibrated and validated in accordance with EN 14181 with the modifications specified in 6.2 to 6.8 of this standard. Unless otherwise specified by regulation, the maximum permissible uncertainty is defined as 40 % of the daily ELV. EN 14181 specifies that the short term ELV, i.e. the daily ELV is used for quality assurance purposes.

However, for mercury a lower long term ELV, e.g. an annual ELV, may be specified. If the long term ELV is less than 50 % of the short term ELV, then the long-term ELV shall be used instead of the daily ELV, for all quality assurance assessments.

NOTE   A multiple of the long term ELV may be used for quality assurance purposes if this is agreed with the competent authority, for example, if this is required due to more variable mercury emissions during QAL2 testing.

NOTE   Annex A shows an example of the application of QAL2 for an AMS.

6.2 Functional test

6.2.1 General

Functional tests are performed to ensure that the AMS is working according to the specifications and to check the active measurement components of the AMS to ensure that they are not unduly influenced by contamination. The functional tests shall be carried out in accordance with EN 14181:2014 Annex A with the modifications specified below. Both elemental and oxidized reference materials may be used for the functional tests, noting that only oxidized reference material (e.g. $HgCl_2$) is used for the converter efficiency test. The manufacturers' operating instructions for the reference material generators shall be followed, ensuring that a sufficient flow of reference material is used to avoid simultaneous entrainment of flue gas into the probe. The uncertainty of the reference materials shall be assessed and reported by the test laboratory. The functional test shall be performed by an experienced testing laboratory, which has been recognized by the competent authority. The independent reference material generators shall be traceable to national standards.

All functional tests shall be performed by passing gaseous reference material through the entire AMS, including the filter, dilution system (if applicable), sample line, and the oxidized mercury conversion system. In the case of older AMS, when it is not possible to introduce reference material upstream of the sample filter, then elements of the sampling system may need to be bypassed in which case the test laboratory shall report a non-conformance with this standard.

In the case of oxidized mercury, a reference material containing water vapour is typically specified in order to minimize mercury losses and hold-up within the sampling system. Care should be taken to volume correct the reference mercury concentration to take account of the injected water vapour concentration which should ideally be held constant. If mercury chloride solutions are used within an oxidized mercury generator supplied by the test laboratory then the stability of those solutions should be checked by the test laboratory.

6.2.2 Zero and span check (EN 14181 Annex A.7)

Zero and span checks shall be performed by passing gaseous reference material through the entire AMS. The selected span concentration shall be no higher than 200 % of the daily ELV. The agreement between the measured span result and the span gas concentration shall be better than 5,0 % of the daily ELV. If the first span check is outside of this tolerance then the AMS shall be adjusted according to the QAL1 span adjustment procedure and re-tested. The results of these adjustments shall be reported by the test laboratory.

If the zero point is used to establish the calibration function according to EN 14181:2014, 6.4.3 procedure b), or procedure c), the zero test shall be used to prove that the AMS gives a reading at or below detection limit (as demonstrated in QAL1) at a zero concentration. The test results shall be presented in the QAL2 calibration report.

If the span point is used to establish the calibration function according to EN 14181:2014, 6.4.3 procedure c), the span reference material expanded uncertainty shall be within ± 5 % of the reference concentration, or ± 0,25 ug/m³, whichever is higher. The test results shall be presented in the QAL2 calibration report. In this case, the span gas concentration shall also be measured by the SRM, i.e. by passing the independent mercury generator output through the SRM sampling train. This is achieved by splitting the mercury generator output between the AMS and the SRM to obtain simultaneous measurements but, if that is not possible due to insufficient generator flow rate, then the generator output may be switched from the AMS to the SRM to obtain consecutive measurements.

6.2.3 Linearity test (EN 14181 Annex A.8 and Annex B)

Linearity tests shall be performed by passing gaseous reference materials through the entire AMS.

EN 14181 requires that the concentration range specified for the linearity test shall extend to at least the daily ELV. However, the measuring range of the instrument shall also capture the peak concentrations emitted by the process and so the highest concentration specified for the linearity test may be much higher than the short term ELV.

If the anticipated peak process concentrations are higher than the daily ELV, then additional concentration levels may be specified provided that these additional concentration levels pass the residual test based on the daily ELV.

6.2.4 Response time (EN 14181 Annex A.11)

Response time tests shall be performed by passing reference material through the entire AMS.

The response time pass criterion for AMS with pre-concentration is increased by the cycle time of the AMS. For example, if the response time criterion for AMS without pre-concentration is 400 s and the cycle time of the AMS with pre-concentration is 620 s, then the response time criterion becomes 1020 s (17 min).

6.2.5 Cycle time for AMS with pre-concentration

AMS with mercury pre-concentration are characterized by a total measurement cycle time, t_{cycle}, and a sample period, t_{sample}. The ratio t_{sample} / t_{cycle} shall be larger than 90 %. However, a ratio t_{sample} / t_{cycle} less than 90 % may be used, if there are no rapid changes in the mass concentration of mercury, subject to approval by the competent authority.

The maximum length of the measurement cycle shall not be longer than the shortest ELV averaging period, e.g. 30 min, for these AMS.

6.2.6 Converter efficiency

The AMS convertor efficiency shall be tested to confirm that oxidized Hg is converted to elemental Hg. Oxidized reference material (e.g. $HgCl_2$) shall be introduced into the probe, upstream of the filter, to test for losses of Hg across the sampling arrangement. The converter efficiency is defined as the measured Hg concentration divided by the expected reference Hg concentration multiplied by 100 %. The conversion efficiency shall be ≥ 90 %. Special care should be taken, when handling oxidized Hg reference gases, as surface reactions in the sample tubing can result in longer response times compared to elemental Hg reference gases.

6.3 Parallel measurements with the SRM

According to EN 14181, QAL 2 at least 15 parallel measurements shall be performed with the AMS and SRM in order to calibrate and validate the AMS by use of an independent method. The measurements shall be performed at normal operating conditions of the plant as required by EN 14181.

For AMS with pre-concentration of Hg, the SRM measurement shall start at the beginning of a new AMS total measurement cycle and shall be performed for a complete number of measurement cycles, in addition to complying with the sample time requirements defined by EN 14181.

The SRM shall be applied in accordance with EN 13211. Alternative methods that are demonstrated to be equivalent to the SRM, according to EN 14793, may be approved by the competent authority and these shall be applied in accordance with the relevant European or ISO standards.

Reducing the number of measurements and increasing the individual SRM sampling time can lead to a better quantification of very low mercury concentrations. If most of the SRM measured results are expected to be below the maximum permissible uncertainty, the sampling duration shall be extended to a maximum of 2 h per test. The number of parallel measurements may then be reduced to a minimum of