SLOVENSKI STANDARD
SIST-TS CEN/TS 16429:2013

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Emisije nepremičnih virov - Vzorčenje in določevanje vodikovega klorida v odvodnikih in odvodnikih v zrak - Infrardeča analizna tehnika

Stationary source emissions - Sampling and determination of hydrogen chloride content in ducts and stacks - Infrared analytical technique

Emissionen aus stationären Quellen - Probenahme und Bestimmung von Chlorwasserstoff in Abgaskanälen und -kaminen - Infrarotverfahren

Émissions de sources fixes - Prélèvement et détermination du chlorure d'hydrogène dans les conduits et les cheminées - Technique analytique infrarouge

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13.040.40	Emisije nepremičnih virov	Stationary source emissions

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TECHNICAL SPECIFICATION
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English Version

Stationary source emissions - Sampling and determination of hydrogen chloride content in ducts and stacks - Infrared analytical technique

Émissions de sources fixes - Prélèvement et détermination du chlorure d'hydrogène dans les conduits et les cheminées - Technique analytique infrarouge

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Foreword

This document (CEN/TS 16429:2013) has been prepared by Technical Committee CEN/TC 264 “Air quality”, the secretariat of which is held by DIN.

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1 Scope

This Technical Specification specifies an automatic method for determination of the mass concentration of hydrogen chloride (HCl) in ducts and stacks emitting to atmosphere. It describes the infrared analytical technique, including the sampling and gas conditioning system. The method should fulfil the performance characteristics requirements of this Technical Specification and the expanded uncertainty is less than 20 % relative at the daily Emission Limit Value (ELV).

In order to use an alternative method to this method, it is necessary to demonstrate equivalence according to the Technical Specification CEN/TS 14793. It is necessary that the capability to demonstrate equivalence is officially recognised by the national accreditation body or law.

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

EN 15267-3:2007, *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 ISO 14956:2002, *Air quality — Evaluation of the suitability of a measurement procedure by comparison with a required measurement uncertainty (ISO 14956:2002)*

3 Terms and definitions

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

3.1
adjustment of a measuring system
set of operations carried out on a measuring system so that it provides prescribed indications corresponding to given values of a quantity to be measured

[SOURCE: VIM 3.11]

3.2
ambient temperature
temperature of the air around the measuring system

3.3
drift
difference between two zero (zero drift) or span readings (span drift) at the beginning and at the end of a measuring period

3.4
emission limit value
ELV
emission limit value according to EU Directives on the basis of 30 min, one hour or one day

3.5
influence quantity
quantity that, in a direct measurement, does not affect the quantity that is actually measured, but affects the measurement result

[SOURCE: VIM 2.52, modified]

EXAMPLES

- Ambient temperature;
- atmospheric pressure;
- presence of interfering gases in the flue gas matrix;
- pressure of the gas sample.

3.6
interference

negative or positive effect upon the response of the measuring system, due to a component of the sample that is not the measurand

3.7
lack of fit

systematic deviation within the range of application between the measurement result obtained by applying the calibration function to the observed response of the measuring system measuring test gases and the corresponding accepted value of such test gases

Note 1 to entry: Lack of fit may be a function of the measurement result.

Note 2 to entry: The expression “lack of fit” is often replaced in everyday language by “linearity” or “deviation from linearity”.

3.8
measurand

quantity intended to be measured

[SOURCE: VIM 2.3]

3.9
measuring system

complete set of measuring instruments and other equipment assembled to carry out specified measurements

[SOURCE: VIM 3.2, modified]

3.10
performance characteristic

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

3.11
repeatability in the laboratory

closeness of the agreement between the results of successive measurements of the same measurand carried out under the same conditions of measurement

Note 1 to entry: Repeatability conditions include:

- the same measurement procedure;
- the same laboratory;
- the same measuring system, used under the same conditions;
- the same location;
- repetition over a short period of time.

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

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

[SOURCE: VIM 2.20, modified]

3.12
residence time in the measuring system
time period for the sampled gas to be transported from the inlet of the probe to the inlet of the measurement cell

3.13
response time
duration between the instant when an input quantity value of a measuring instrument or measuring system is subjected to an abrupt change between two specified constant quantity values and the instant when a corresponding indication settles within specified limits around its final steady value

Note 1 to entry: By convention time taken for the output signal to pass from 0 % to 90 % of the final change.

[SOURCE: VIM 4.23, modified]

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

[SOURCE: EN 13284-1:2001, 3.8]

3.15
sampling point
specific position on a sampling line at which a sample is extracted

[SOURCE: EN 13284-1:2001, 3.10]

3.16
span gas
test gas used to adjust and check a specific point on the response line of the measuring system

Note 1 to entry: This concentration is often chosen around 80 % of the upper limit of the range or around the emission limit value.

3.17
uncertainty
parameter associated with the result of a measurement, that characterises the dispersion of the values that could reasonably be attributed to the measurand

3.17.1
standard uncertainty u
uncertainty of the result of a measurement expressed as a standard deviation u

3.17.2
expanded uncertainty U
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

$$U = k \, . \, u$$

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

3.17.3

combined uncertainty u_c

standard uncertainty u_c attached to the measurement result calculated by combination of several standard uncertainties according to GUM

3.18

uncertainty budget

calculation table combining all the sources of uncertainty according to EN ISO 14956 or ENV 13005 in order to calculate the expanded uncertainty of the method at a specified value

4 Principle

4.1 General

This Technical Specification describes a method for the determination of the mass concentration of hydrogen chloride (HCl) in ducts and stacks emitting to atmosphere by means of an automatic analyser using the infrared absorption principle. The specific components and requirements for the sampling system and the infrared analyser are described in Clause 6. A number of performance characteristics with associated minimum performance criteria and an expanded uncertainty of the method are given. Requirements and recommendations for quality assurance and quality control are given for measurements in the field (see Table 1 in 7.3).

4.2 Measuring principle

The HCl concentration is measured with an infrared absorption method. The attenuation of infrared light passing through a sample cell is a measure of the concentration of HCl in the cell, according to the Lambert-Beer law. Not only HCl but also most hetero-atomic molecules absorb infrared light, in particular water and CO_2 have broad bands that can interfere with the measurement of HCl. Different technical solutions have been developed to suppress cross-sensitivity, instability and drift in order to design automatic monitoring systems with acceptable properties. For instance: Gas Filter Correlation, Tunable Diode Laser (TDL) and Fourier Transform Infrared Spectroscopy (FTIR).

Special attention is paid to infrared light absorbing gases such as water vapour, carbon dioxide, nitrous oxide, nitrogen dioxide and also hydrocarbons for some special applications.

Infrared analysers are part of extractive or in-situ systems. Most of them are combined with an extractive sampling system and a gas conditioning system. A representative sample of gas is taken from the stack with a sampling probe and conveyed to the analyser through the sampling line and gas conditioning system. The values from the analyser are recorded and/or stored by means of electronic data processing.

The concentration of HCl is measured in volume/volume units (if the analyser is calibrated using a volume/volume standard). The final results for reporting are expressed in milligrams per cubic meter using standard conversion factors (see Clause 10).

5 Sampling system

5.1 General

A representative volume (see 8.2.1) is extracted from the flue gas for a fixed period of time at a controlled flow rate. A filter removes the dust in the sampled volume before the sample is conditioned and passes to the analyser. Three different sampling and conditioning configurations can be used in order to avoid uncontrolled water vapour condensation in the measuring system. These configurations are:

- configuration 1: removal of water vapour through elimination using a permeation drier;
- configuration 2: maintaining the temperature of the sampling line up to the heated analyser;

— configuration 3: for in situ measurements.

Conditions and layout of the sampling equipment contribute to the expanded uncertainty. In order to minimise this contribution to the expanded uncertainty of the method, performance criteria for the sampling equipment and sampling conditions are given in 5.2 and 7.2.

5.2 Sampling probe

In order to access the representative sampling point(s) of the sampling plane, probes of different lengths and inner diameters may be used. The design and configuration of the probe used shall ensure the residence time of the sample gas within the probe is minimised in order to reduce the response time of the measuring system.

The procedure described in 8.1 shall be used when a lack of homogeneity in the flue gas is suspected.

5.3 Filter

The filter shall be made of an inert material (e.g. ceramic with an appropriate pore size). The particle filter shall be changed or cleaned periodically depending on the dust loading at the sampling site.

Overloading of the particle filter may increase the pressure drop in the sampling line.

5.4 Sampling line

The sampling line shall be heated up to the conditioning system, where required. It shall be made of a suitable corrosion resistant material (e.g. borosilicate glass, ceramic or titanium could be used; PTFE is suitable for flue gas temperatures lower than 200 °C).

5.5 Conditioning system

5.5.1 Permeation drier (configuration 1)

It is important that all parts of the sampling equipment upstream of the analyser are made of materials that do not react with or absorb HCl. The temperature of its components coming into contact with the gas shall be maintained, upstream the permeation system, at a sufficiently high temperature (between 180 °C and 200 °C) to prevent salt formation and condensation in the sampling equipment (in the presence of water vapour (beyond in 15 %) and ammonia (few ppm), risk occurs to have salt even at temperatures of 165 °C -170 °C).

The permeation drier is used before the gas enters the analyser in order to separate water vapour from the flue gas. A dew-point temperature below 4 °C is required at the outlet of the permeation drier.

The concentrations, provided by this sampling configuration, are considered to be given on dry basis. However, the results may be corrected for the remaining water vapour (refer to the table of Annex A in EN 14790:2005).

This configuration shall not be used if the flue gas has an ammonia concentration higher than 1 ppm.

5.5.2 Heated line and heated analyser (configuration 2)

It is important that all parts of the sampling equipment upstream of the analyser are made of materials that do not react with or absorb HCl. The temperature of its components coming into contact with the gas shall be maintained at a sufficiently high temperature (between 180 °C and 200 °C) to prevent salt formation and condensation in the sampling equipment (in the presence of water vapour (beyond in 15 %) and ammonia (few ppm), risk occurs to have salt even at temperatures of 165 °C -170 °C).

If the concentrations are given on wet basis, they shall be corrected so that they are expressed on dry basis. The correction shall be made from the water vapour concentration measured in the flue gas. The uncertainty attached to this correction shall be part of the uncertainty budget.

5.5.3 In situ analysers (configuration 3)

A calibration system, evaluated during the certification process shall be available to determine zero and span drift for the whole system.

5.6 Sample pump

The sample pump shall be capable of operating to the specified flow requirements of the manufacturer of the analyser and pressure conditions required for the sample cell. The pump shall be resistant to corrosion. If an external pump is used it shall be compatible with the requirements of the analyser to which it is connected.

5.7 Secondary filter

The secondary filter is used to separate fine dust, with a pore size less than 5 µm. For example it may be made of glass-fibre, sintered ceramic, stainless steel or PTFE-fibre.

5.8 Flow controller and flow meter

This apparatus sets the required flow. A corrosion resistant material shall be used. The sample flow rate into the analyser shall be maintained within the analyser manufacturer's requirements. A controlled pressure drop across restrictors is usually employed to maintain flow rate control into the infrared analyser.

NOTE No additional flow controller or flow meter is necessary when they are part of the analyser itself.

6 Analyser equipment

6.1 General

The main parts of the analyser are typically:

- source of infrared radiation;
- optics to focus the radiation through the measuring cell to the infrared detector;
- a way of modulating the infrared beam;
- means to select a suitable wavelength or wavelengths to measure the gas;
- a measuring cell that the sample gas enters. In some designs there may be a reference cell;
- an infrared detector; and
- an amplifier and signal processing system to give an electrical output proportional to the HCl concentration.

The standard of construction and vibration/corrosion resistance shall be suited to industrial environments and to the composition of the flue gas.

In Annex A, schematic diagrams are given of several different examples of infrared analysers.

6.2 Pressure and temperature effects

The output signal of the analyser is proportional to the number of HCl molecules present in the absorption cell and depends on the absolute pressure and temperature in the absorption cell. The effects of variations of pressure and temperature in the absorption cell should be taken into account by the manufacturer to give results in reference conditions of 273 K and 101,3 kPa.