SLOVENSKI STANDARD
oSIST prEN 12619:2011

01-maj-2011

Emisije nepremičnih virov - Določevanje masnih koncentracij celotnega organskega ogljika v plinasti fazi - Kontinuirana metoda plamenske ionizacijske detekcije

Stationary source emissions - Determination of the mass concentration of total gaseous organic carbon - Continuous flame ionisation detector method

Emissionen aus stationären Quellen - Bestimmung der Massenkonzentration des gesamten gasförmigen organisch gebundenen Kohlenstoffs - Kontinuierliches Verfahren mit dem Flammenionisationsdetektor

Emissions de sources fixes - Détermination de la concentration massique en carbone organique total - Méthode du détecteur continu à ionisation de flamme

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13.040.40 Emisije nepremičnih virov Stationary source emissions

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English Version

Stationary source emissions - Determination of the mass concentration of total gaseous organic carbon - Continuous flame ionisation detector method

Emissions de sources fixes - Détermination de la concentration massique en carbone organique total - Méthode du détecteur continu à ionisation de flamme

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

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Foreword

This document (prEN 12619:2011) has been prepared by Technical Committee CEN/TC 264 “Air quality”, the secretariat of which is held by DIN.

This document is currently submitted to the CEN Enquiry.

This document will supersede EN 13526:2001, EN 12619:1999.

1 Scope

The method specified in this European Standard is designed for use as a standard reference method.

This European Standard specifies a set of minimum performance requirements for an instrument using flame ionization detection, together with procedures for its calibration and operation, for the measurement of the mass concentration of total gaseous organic carbon (TOC) and for solvent emissions total volatile organic compounds (TVOC) in stationary source emissions.

This European Standard is suitable for the measurement of gaseous or vapour phase TOC and TVOC emissions such as emissions from waste incinerators and solvent using processes.

The results obtained using this standard, are expressed in milligrams per cubic metre as total carbon (mg/m^3). This standard is suitable for use in the range 1 mg/m^3 up to at least 1000 mg/m^3.

This European Standard is not applicable for permanently installed AMS. For permanently installed AMS refer to EN 15267-3.

NOTE This method can also be used also for higher mass concentrations TVOC.

2 Normative references

This European Standard incorporates, by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies.

EN 14789, *Stationary source emissions — Determination of volume concentration of oxygen (O_2) — Reference method — Paramagnetism*

EN 14790, *Stationary source emissions — Determination of the water vapour in ducts*

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

ENV 13005, *Guide to the expression of uncertainly in measurement*

EN ISO 14956, *Air Quality — Evaluation of the suitability of a measurement procedure by comparison with a required measurement uncertainty*

CEN/TS 14793, *Stationary source emissions — Interlaboratory validation procedure for an alternative method compared to a reference method*

3 Terms and definitions

For the purposes of this standard, the following definitions apply.

3.1
combustion air
air supply used for the combustion of fuel gas in an instrument using flame ionization detection

3.2
complementary gas
component of a calibration gas which completes a calibration gas mixture

3.3
control gas mixture
gas mixture used to check the minimum performance requirements of the FID during instrument certification

3.4
detection limit
minimum concentration of a substance which produces an observable response, as detailed in A.1.2 and referred to in ISO 7504:1984

3.5
dilution gas
gas used to dilute sampled flue gas to prevent water condensation

3.6
flame ionization detector (FID)
instrument using flame ionisation detection

3.7
flue gas
product from a combustion, incineration or solvent process containing gaseous and/or particulate components

3.8
fuel gas
gas of known composition used to maintain the flame of the FID

3.9
mass concentration of gaseous total organic carbon
quotient of the mass of total organic carbon to the volume of the dry gas under specified reference conditions of temperature and pressure, normally expressed in milligrams per cubic metre as total carbon (mgC/m^3)

3.10
residence time
time period for the sampled gas to be transported from the inlet of the probe to the inlet of the measurement cell

3.11
response factor
dimensionless quotient of the response of the FID with any carbon based compound or compounds to its response to propane, in each case referred to the number of carbon atoms of the molecule

3.12
response time
time which elapses between the moment when a change is produced and the moment when the instrument response reaches a value of 90 % of the final change in instrument response as a consequence of a stepwise

change in the total organic carbon concentration

3.13
span gas
gas used to adjust and check a specific point on a calibration curve

3.14
total organic carbon (TOC)
by convention the total gaseous organic carbon which is measured by the FID and expressed as mgC/m³

3.15
total volatile organic carbon (TVOC)
by convention total volatile organic compounds which are measured by the FID, expressed as mgC/m³ from solvent using process

3.16
zero gas
gas or a gas mixture used to check and adjust the zero point on a calibration curve

3.17
VOC
definition from solvent directive: volatile organic compound (VOC) shall mean any organic compound having at 293,15 K a vapour pressure of 0,01 kPa or more, or having a corresponding volatility under the particular conditions of use. For the purpose of this Directive, the fraction of creosote which exceeds this value of vapour pressure at 293,15 K shall be considered as a VOC

3.18
uncertainty
parameter associated with the result of a measurement, that characterizes the dispersion of the values that could reasonably be attributed to the measurand

3.19
uncertainty budget
calculation table combining all the sources of uncertainty according to ISO 14956 or ENV 13005 in order to calculate the overall uncertainty of the method at a specified value

3.20
overall uncertainty
expanded combined standard uncertainty attached to the measurement result calculated according to ENV 13005

4 The principle of the technique

4.1 Flame ionization detector (FID)

The measurement technique utilized by the FID is the ionization of organically bound carbon atoms in a hydrogen flame. The ionization current measured by the FID depends on the number of C-atoms of organic compounds burning in the fuel gas flame, the form of bonding (straight chain or branched chain) and of bonding partners.

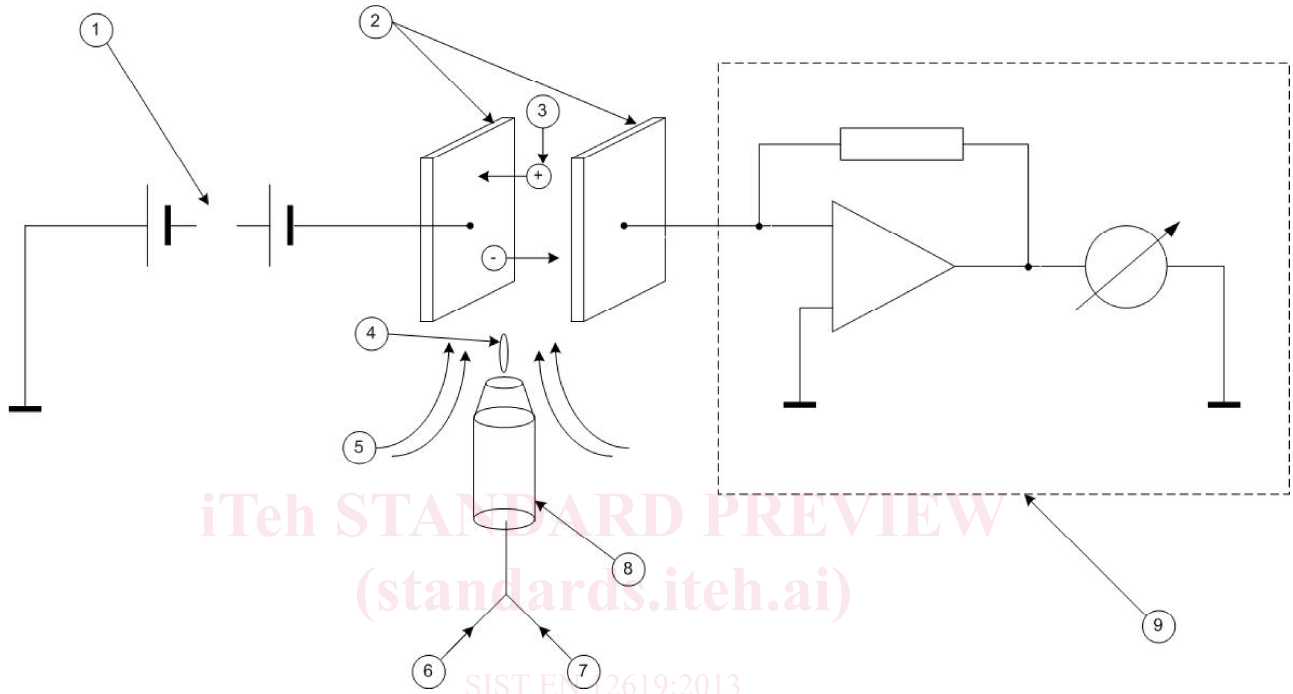
The response factor is a function of the specific design of the detector and the adjusted operating conditions. The advantage of the FID is that it responds to organic carbon compounds and has negligible response to inorganic flue gas compounds (such as CO, CO_2, NO, H_2O).

A number of different instrument configurations exist. Figure 1 is an example of the principle whereby in the detector a sample gas is fed into a hydrogen flame across which a DC electrical potential is placed. The introduction of the sampled gas causes a specific ionization current to flow, which is measured using suitable

equipment. Defined test gases are required to determine the response factors. These can be produced by a number of methods including: static methods (with gas collectors or direct injection) or dynamic methods (e.g. vapour pressure method or certified test gases from compressed gas bottles).

The span of the instrument shall be adjusted with propane (C_3H_8) for which the response factor, defined in this standard, has been set at 1,00. The final value will be expressed as TOC in milligrams per cubic metre.

Refer to Annex B for more information on the use and effects of an FID instrument.



Key

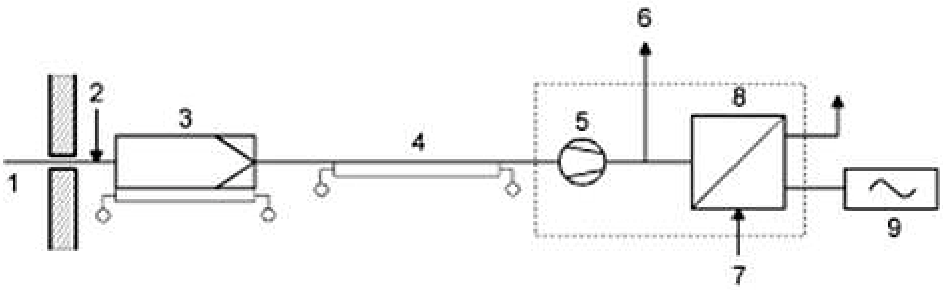
1 polarisation voltage
2 electrodes
3 ions
4 flame
5 combustion air
6 fuel gas
7 sample gas
8 jet
9 amplifier and readout

Figure 1 — Principle of FID

4.2 Sampling and sampling device

Sampling is the process of extracting from a large quantity of flue gas a small portion which is truly representative of the composition of the main gas stream.

A partial flow of the flue gas is directly fed into the FID analyser via the sampling probe, the particle filter and the heated sampling line. An example of the set-up of the measuring system is shown in Figure 2. The sampling device including the filter needed to remove fine particles, which could clog the burner, is heated to avoid sample condensation.



Key

1	sampling probe	6	bypass (optional)
2	zero and span gas inlet	7	test gas inlet for functional tests
3	particle filter (in-stack or out-stack), heated	8	FID
4	sampling line, heated	9	data evaluation system
5	external sample pump (optional), heated		

Figure 2 — Example of the set-up of the measuring system

5 Apparatus and gases

5.1 Sampling device

The sampling device shall meet the following requirements:

— It shall be made of stainless steel, polytetrafluoroethylene or polypropylenefluoride. If an alternative material is used, it shall be proven by validation that it is chemically and physically inert to the constituents of the flue gas under analysis.

NOTE 1 If a material is used in a permanently installed FID AMS with type approval to EN 15267-3, then it can be assumed that the material will be acceptable for use in a portable version of the AMS.

— The design and configuration of the sampling device used shall ensure the residence time of the sample gas within the device is minimised in order to reduce the response time of the measuring system.

NOTE 2 It should be designed to ensure a sample residence time less than 60 s; with long sampling lines or high flow resistance the use of an external pump with bypass is recommended.

— It shall be heated throughout to at least 180 °C.

NOTE 3 Sampling lines in PTFE has a maximum temperature of 200 °C.

— It shall have a heated filtering device upstream of the sampling line to trap all particles liable to impair the operation of the apparatus;

— It shall have an inlet for applying zero and span gases at or close to the probe inlet of the sampling probe, upstream of the filter. This is to check the sampling system including the filter assembly.

5.2 The FID

The FID and sampling system shall comply with the performance requirements of EN 15267-3.

5.3 Periodic and annual checks

The periodic and annual checks in Table 1 shall be carried out.

Table 1 — Minimum frequency of checks for QA/QC during the operation

Check	Minimum frequency
Response time	The response time will be controlled periodical
Repeatability standard deviation at zero point	once a year
Repeatability standard deviation at span point	once a year
Lack of fit	once a year and after repair of the instrument
Effect of oxygen	at least once a year and after repair (can be carried out by the manufacturer)
Other interference checks	once a year and after repair (can be carried out by the manufacturer)
Sampling system and leakage check	once for each measurement series
Cleaning or changing of particulate filters[a] at the sampling inlet and at the monitor inlet	once for each measurement series, if needed
Zero drift	at the beginning and end of the measuring period and at least once a day
Span drift	at the beginning and end of the measuring period and at least once a day
Regular maintenance of the analyser	as required by the manufacturer

[a] The particulate filter shall be changed periodically depending on the dust load at the sampling site. During this filter change the filter housing shall be cleaned.

5.4 Operational gases

A number of operational gases are required when using the standard.

NOTE The use of combustion air or fuel gas whose concentration in TOC is lower than 0,2 mgC/m³ or of purity 99,998 % is recommended and allows to avoid any risk of influence of this gas on the result of the measurement.

5.4.1 Combustion air

The TOC concentration (mgC/m³) of combustion air shall not exceed 1 % of the emission limit value (ELV) TOC. This can be synthetic air or cleaned ambient air.

5.4.2 Fuel gases

The fuel gas shall be specified by the equipment manufacturer, it may be:

— hydrogen;

— hydrogen/helium mixture;

— hydrogen/nitrogen mixture.

The TOC concentration (mgC/m³) of fuel gas shall not exceed 1 % of the emission limit value (ELV) TOC. This can be synthetic air or cleaned ambient air.

NOTE 1 Gases with a purity of 99,999 % (percent by volume) usually meet this requirement.