



# SLOVENSKI STANDARD

## SIST EN 12619:2013

01-april-2013

Nadomešča:

SIST EN 12619:2000

SIST EN 13526:2002

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**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

**Ta slovenski standard je istoveten z: EN 12619:2013**

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**ICS:**

13.040.40      Emisije nepremičnih virov      Stationary source emissions

**SIST EN 12619:2013**

**en,fr,de**

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EUROPEAN STANDARD  
NORME EUROPÉENNE  
EUROPÄISCHE NORM

**EN 12619**

January 2013

ICS 13.040.40

Supersedes EN 12619:1999, EN 13526:2001

English Version

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This European Standard was approved by CEN on 24 November 2012.

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This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the CEN-CENELEC Management Centre has the same status as the official versions.

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## Foreword

This document (EN 12619:2013) 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of an identical text or by endorsement, at the latest by July 2013, and conflicting national standards shall be withdrawn at the latest by July 2013.

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.

This document supersedes EN 12619:1999 and EN 13526:2001.

The list of the most significant technical changes that have been made in this new edition is to be found in Annex E.

According to the CEN/CENELEC Internal Regulations, the national standards organisations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

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## 1 Scope

This European Standard specifies a flame ionisation detector (FID) method. It is intended for use as a standard reference method for the measurement of the mass concentration of gaseous and vaporous organic substances (expressed as TVOC) in stationary source emissions (e.g. emissions from waste incinerators and solvent using processes, emission measurements according to 2010/75/EU) in the concentration range up to 1 000 mg/m<sup>3</sup>.

This European Standard specifies the requirements for an instrument using flame ionisation detection, together with procedures for its operation. The results obtained using this standard are expressed in milligrams per cubic metre (mg/m<sup>3</sup>) as total carbon (TVOC).

This European Standard is not applicable for permanently installed automated measuring systems (AMS).

Alternative methods to this method may be used provided that the user can demonstrate equivalence, based on the principles of CEN/TS 14793.

## 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 15267-1, *Air quality — Certification of automated measuring systems — Part 1: General principles*

EN 15267-2, *Air quality — Certification of automated measuring systems — Part 2: Initial assessment of the AMS manufacturer's quality management system and post certification surveillance for the manufacturing process*

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 9169, *Air quality — Definition and determination of performance characteristics of an automatic measuring system (ISO 9169)*

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

## 3 Terms and definitions

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

### 3.1

#### **combustion air**

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

### 3.2

#### **detection limit**

minimum concentration of a substance which produces an observable response, which is two times the standard deviation at zero

**3.3****dilution gas**

gas used to dilute sampled flue gas to prevent water condensation

**3.4****flame ionisation detector (FID)**

instrument using flame ionisation detection

**3.5****flue gas**

product from a combustion, incineration or solvent process containing gaseous and/or particulate compounds

**3.6****fuel gas**

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

**3.7****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 ( $\text{mg}/\text{m}^3$ ) as total carbon

**3.8****residence time**

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

**3.9****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

**3.10****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

**3.11****span gas**

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

**3.12****total volatile organic carbon (TVOC)**

total volatile organic compounds which are measured by the FID, expressed in milligrams per cubic metre ( $\text{mg}/\text{m}^3$ ) as total carbon

**3.13****zero gas**

test gas used to check and adjust the zero point on a calibration curve

**3.14****uncertainty**

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

[SOURCE: ENV 13005]

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## 4 The principle of the technique

### 4.1 Flame ionisation detector (FID)

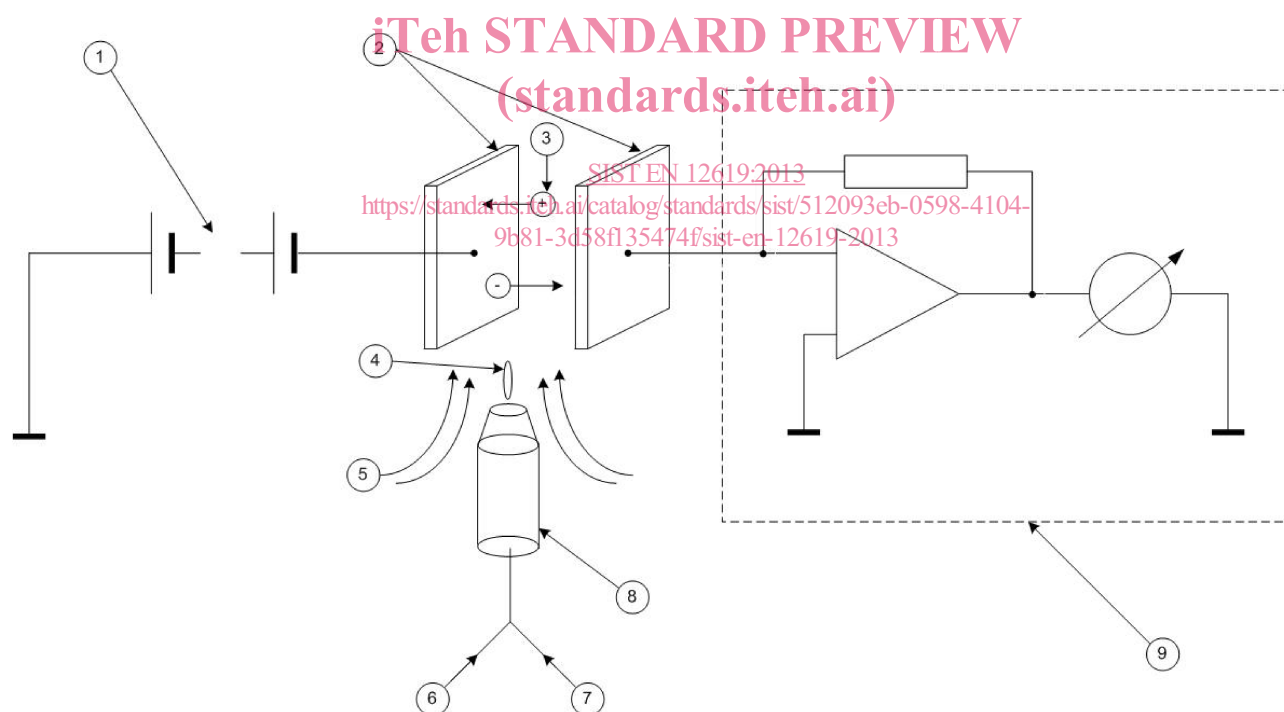
The measurement technique utilised by the flame ionisation detector (FID) is the ionisation of organically bound carbon atoms in a hydrogen flame. The ionisation current measured by the FID depends on the number of C-atoms of organic compounds burning in the fuel gas flame, the form of bonding (straight 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<sub>2</sub>, NO, H<sub>2</sub>O).

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 ionisation 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<sub>3</sub>H<sub>8</sub>) for which the response factor, defined in this standard, has been set at 1,00. The final value will be expressed as TVOC 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 | 6 fuel gas              |
| 2 electrodes           | 7 sample gas            |
| 3 ions                 | 8 jet                   |
| 4 flame                | 9 amplifier and readout |
| 5 combustion air       |                         |

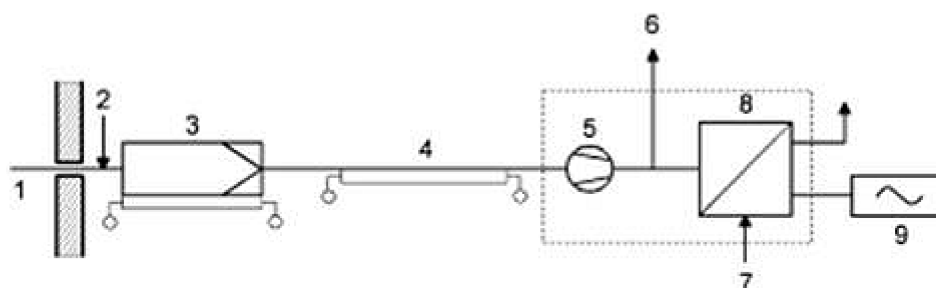
Figure 1 — Principle of FID



## 4.2 Sampling and sampling device

Sampling is the process of extracting from the flue gas a partial volume flow which is 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, 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 Requirements for apparatus and gases

### 5.1 Requirements for the measurement system

The sampling system 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 that it is chemically and physically inert to the constituents of the flue gas under analysis.
- 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. It will 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 Sampling lines in PTFE have a maximum temperature of 200 °C. All instruments that are certified are tested at 180 °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.

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

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The checks in Table 1 shall be carried out with at least the specified frequency.

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

Check	Minimum frequency	Requirement	Clause
Response time	once for each measurement series	$\leq 200$ s	A.2
Repeatability standard deviation at zero point	once a year	$\leq 2$ %	A.4
Repeatability standard deviation at span point	once a year	$\leq 2$ %	A.5
Lack of fit	once a year and after repair <sup>a</sup> of the instrument	$\leq 2$ %	A.3
Effect of oxygen	at least once a year and after repair (can be carried out by the manufacturer)	$\leq 2$ %	A.6
Other interference checks <sup>b</sup>	at least once a year and after repair (can be carried out by the manufacturer)	$\leq 2$ %	A.7
Response factor determination	as specified by the manufacturer and is relevant for single compound measuring tasks		A.8 and Annex B
Sampling system check	once for each measurement series		6.2.2
Leakage check	once for each measurement series		6.2.2
Zero drift	at the beginning and end of the measuring period and at least once a day	$< 5$ %	6.2.3
Span drift	at the beginning and end of the measuring period and at least once a day	$< 5$ %	6.2.3
Regular maintenance of the analyser	as required by the manufacturer		
Cleaning or changing of sampling line and particulate filters <sup>c</sup>	once for each measurement series, if needed		

<sup>a</sup> A repair that might affect the performance of the instrument.

<sup>b</sup> Only those interferences shall be checked which have proven to be relevant during instrument performance testing.

<sup>c</sup> 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.

## 5.2 Operational gases

### 5.2.1 General

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

The use of combustion air or fuel gas whose concentration in TVOC is lower than 0,2 mg/m<sup>3</sup> (as carbon) or of purity 99,998 % shall be used. This avoids any risk of influence of this gas on the result of the measurement.

### 5.2.2 Fuel gases

The fuel gas shall be specified by the equipment manufacturer and according to the certification to EN 15267-1, EN 15267-2 and EN 15267-3. Commonly used gases include:

- hydrogen;
- hydrogen/helium mixture;

— hydrogen/nitrogen mixture.

NOTE The fuel gas pipe will be made from a suitable material for the environment, e.g. stainless steel, copper or PTFE.

### 5.2.3 Zero gas

The TVOC concentration ( $\text{mg}/\text{m}^3$  as carbon) of zero gas shall be lower than  $0,2 \text{ mg}/\text{m}^3$  of carbon or purity 99,998 % shall be used. This avoids any risk of influence of this gas on the result of the measurement. This can be synthetic air or cleaned ambient air.

If a problem occurs at the zero point e.g. negative values, this may be an indication of the effect of oxygen. In this case, it is recommended the zero gas may be replaced with one containing a similar oxygen concentration to the process (nitrogen/oxygen mixture or synthetic air). The worst case for this effect is when a process is running with approximately 10 % excess oxygen. Refer to Annex B for more information on the use and effects of an FID instrument.

### 5.2.4 Span gas

The span gas shall be propane in synthetic air or in a nitrogen/oxygen mixture. It shall have a known concentration of TVOC and the expanded uncertainty on the analytical certificate of the span gas shall be less than or equal to 2 % for TVOC.

When the analyser is used for regulatory purposes, the span gas shall have a known concentration of approximately the half-hourly ELV or 50 % to 90 % of the selected range of the analyser.

The span gas will be propane with the same oxygen concentration in the complementary gas in the zero and in the span gas to take into account the same level of influence at the zero point and the span point during the adjustment of the analyser.

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## 6 Measurement procedure

### 6.1 General

The planning and sampling strategy shall follow the requirements of EN 15259.

The following text describes the routine operation procedures required by the standard; a more detailed procedure for determining the instrument performance characteristics is given in Annex A.

### 6.2 Adjustments and checks

#### 6.2.1 Instrument adjustment

The FID shall be set up according to the manufacturer's instructions in order to ensure that the instrument is correctly adjusted as specified in Table 1. At the beginning of the measuring period, zero and span gases are supplied to the analyser directly, without passing through the sampling system. Adjustments are made until the correct zero and span gas values are given by the data sampling system:

- adjust the zero value;
- adjust the span;
- finally, re-check zero to see if there are no significant changes (zero deviation lower than two times the repeatability at zero). Repeat the adjustment procedure until this is achieved.