SLOVENSKI STANDARD
SIST EN 13526:2002

01-maj-2002

Emisije nepremiļ nih virov - Doloļ evanje masne koncentracije celotnega organskega ogljika v plinasti fazi v odpadnih plinih procesov, ki uporabljajo topila - Kontinuirana metoda plamenske ionizacijske detekcije

Stationary source emissions - Determination of the mass concentration of total gaseous organic carbon in flue gases from solvent using processes - Continuous flame ionisation detector method

Emissionen aus stationären Quellen - Bestimmung der Massenkonzentration des gesamten gasförmigen organisch gebundenen Kohlenstoffs in Abgasen von Prozessen, bei denen Lösungsmittel eingesetzt werden - Kontinuierliches Verfahren unter Verwendung eines Flammenionisationsdetektors

Emissions de sources fixes - Détermination de la concentration massique en carbone organique total a de fortes concentrations dans les effluents gazeux - Méthode du détecteur continu a ionisation de flamme

Ta slovenski standard je istoveten z: **EN 13526:2001**

<u>**ICS:**</u>

13.040.40	Emisije nepremiļ nih virov	Stationary source emissions

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EUROPEAN STANDARD

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EUROPÄISCHE NORM

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English version

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Emissionen aus stationären Quellen - Bestimmung der Massenkonzentration des gesamten gasförmigen organisch gebundenen Kohlenstoffs in Abgasen von Prozessen, bei denen Lösungsmittel eingesetzt werden - Kontinuierliches Verfahren unter Verwendung eines Flammenionisationsdetektors

This European Standard was approved by CEN on 29 September 2001.

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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 Management Centre has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.

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Foreword

This European Standard 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 May 2002, and conflicting national standards shall be withdrawn at the latest by May 2002.

This European Standard has been prepared under a mandate given to CEN by the European Commission and European Free Trade Association.

The annex B is normative. The annexes A, C, D, E and F are informative.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

1 Scope

This European Standard specifies a set of minimum performance requirements for an instrument using flame ionisation detection, together with procedures for its calibration and operation, for the measurement of the mass concentration of total gaseous organic carbon (TOC) in flue gases.

This European Standard is suitable for the measurement of gaseous or vapour phase TOC emissions from Solvent Using Processes.

NOTE 1 See Council Directive 1999/13/EEC.

The results obtained using this standard are expressed in milligrams per cubic metre as total carbon (mg/m^3). This standard is suitable for the measurement of concentrations from 20 mg/m^3 to 500 mg/m^3 but can be used at lower concentrations.

NOTE 2 By its nature a flame ionisation detector (FID) can also be used to measure higher concentrations.

The method specified in this European Standard can be used as a reference method or, with suitable minimum operational requirements, for continuous monitoring. It can also be used for the calibration of automated measuring systems. An indication of the uncertainty of the measurement is shown in annex A.

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 (including amendments).

EN 13649	Stationary source emissions - Determination of the mass concentration of individual gaseous organic compounds – Activated carbon and solvent desorption method.
ISO 5725-1	Accuracy (trueness and precision) of measurement methods and results Part 1: General principles and definitions.
ISO 6879	Air quality - Performance characteristics and related concepts for air quality measuring methods.
ISO 9169	Air Quality – Determination of performance characteristics of measurement methods.

3 Terms and definitions

For the purpose of this CEN Standard, the following definitions apply.

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

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

3.3
detection limit
minimum concentration of a substance which produces an observable response, as detailed in annex B and referred to in ISO 9169

3.4
dilution gas
gas used to dilute sampled flue gas to prevent water condensation

3.5
flame ionisation detector (FID)
instrument using flame ionisation detection

3.6
flue gas
product from a combustion or incineration process containing gaseous and particulate components

3.7
fuel gas
gas of known composition used to maintain the flame of the FID

3.8
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 (mg/m^3)

3.9
range
set of values for a measurand for which the error of a measuring instrument is intended to lie within specified limits

3.10
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

3.11
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

3.12
span gas
gas used to adjust and check one point on the calibration curve

3.13
total organic carbon (TOC)
by convention the total gaseous organic carbon which is measured by the FID and expressed as mg/m^3

3.14
zero gas
gas used to adjust and check the zero point on a calibration curve

4 Principle

4.1 General

There are two elements to the extractive TOC analytical system described in this standard. They are the FID and the associated sampling device.

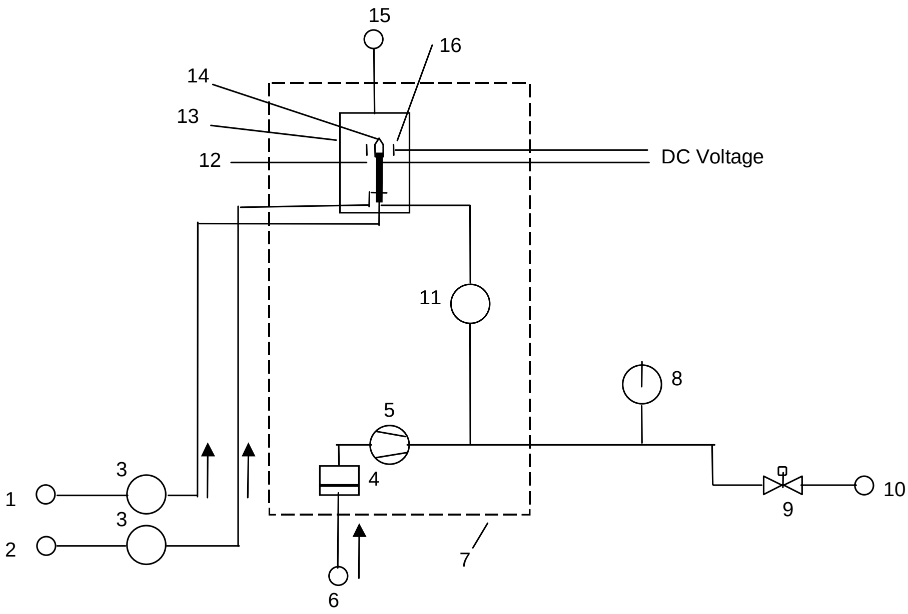
4.2 Flame ionisation detector

The measurement principle is the determination of an ionisation current resulting from the combustion of organic compounds in a hydrogen flame. This current 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 main advantage of the FID is that it responds strongly to organic carbon containing components and less to inorganic flue gas components (such as CO, CO_2, NO, H_2O).

FIDs require a fuel gas and combustion air.



1. Fuel gas
2. Combustion air
3. Flow regulator
4. Fine dust filter
5. Sampling gas pump
6. Sample gas
7. Heated housing
8. Pressure gauge
9. Back-pressure regulator
10. By-pass
11. Meter
12. Nozzle
13. Combustion chamber
14. Flame
15. Gas outlet
16. Electrode

Figure 1: Diagram of the FID measuring principle (example)

A number of different instrument configurations exist. Figure 1 indicates 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 span gas for which the response factor, defined in this standard, has been set at 1.

4.3 Sampling and sampling device

The following principles shall be followed during sampling:

- 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;
- the sampling device is equipped with a filter to remove fine particles which could clog the burner. Condensation downstream of the filter shall be avoided, two methods can be used:
  - a line heated up to the inlet of the analyser,

- a suitable dynamic dilution device.

5 The apparatus and gases

5.1 Apparatus

5.1.1 The FID

The FID shall be shown by the manufacturer to comply with the minimum performance requirements defined in Table 1.

NOTE When used in a continuous mode the instrument should be subject to a periodic functional test which will be specified in subsequent European Standards.

Table 1 - Minimum performance requirements of FIDs - without sampling equipment

Performance characteristics (see Note 1)	Minimum performance requirements
Minimum measuring range	0 mg/m^3 to 50 mg/m^3, 150 mg/m^3 or 500 mg/m^3
Detection limit	5 % of the emission limit value
Response time (0 % to 90 %)	less than 1 min
Linearity deviation	permissible deviation 5 % of emission limit
Range of response factors (see Note 2)	permissible range
Methane	0,9 to 1,2
Aliphatic hydrocarbons (see Note 3)	0,9 to 1,1
Aromatic hydrocarbons (see Note 4)	0,8 to 1,1
Aliphatic alcohols (see Note 5)	0,7 to 1,0
Esters (see Note 6)	0,7 to 1,0
Ketones (see Note 7)	0,7 to 1,0
Organic acids (see Note 8)	0,5 to 1,0
Effect of oxygen (see Note 9)	permissible interference: 5 % of emission limit

NOTE 1 The methods for determining instrument characteristics are given in annex B.
NOTE 2 Examples of typical response factors are given in annex C.
NOTE 3 For the purpose of this standard the aliphatic hydrocarbons may be represented by two of ethane, butane, hexane, heptane, octane or cyclohexane.
NOTE 4 For the purpose of this standard the aromatic hydrocarbons may be represented by benzene and toluene.
NOTE 5 For the purpose of this standard aliphatic alcohols may be represented by two of methanol, ethanol or propanol.
NOTE 6 For the purpose of this standard esters may be represented by ethyl acetate, or isobutyl acetate.
NOTE 7 For the purpose of this standard ketones may be represented by acetone.
NOTE 8 For the purpose of this standard organic acids may be represented by acetic acid.
NOTE 9 For flue gases with an oxygen content of less than 18 % the effect of oxygen shall be determined as shown in annex B.

5.1.2 Sampling device

The sampling device shall be designed to take account of the flue gas characteristics:

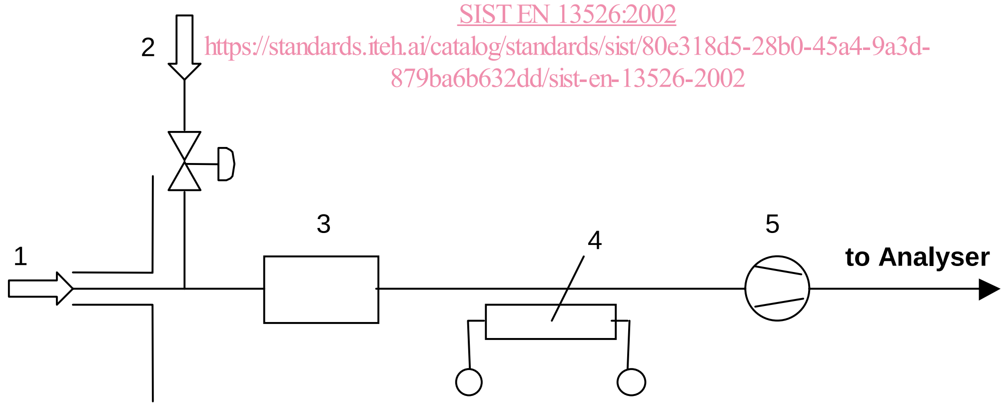
- it shall be made of a material that is chemically and physically inert to the constituents of the flue gas under analysis;

NOTE Stainless steel, polytetrafluoroethylene or polypropylenefluoride are well proven construction materials.

- it shall be designed to ensure a sample residence time less than 1 minute;

- it shall be constructed to avoid condensation in the sampling line, several alternative methods are suitable:
  - heated sampling line. The line shall be heated throughout and where measurements are taken in hot gases the temperature of the coolest point shall be at least 20 °C above the flue gas temperature and shall not exceed 200 °C. An example is shown in Figure 2;
  - dynamic dilution. A suitable device is shown in EN 13649.

- the sampling line shall include a filtering device (upstream) to trap all particles liable to impair the operation of the apparatus.



1 Gas sampling probe
2 Span and zero gas supply
3 Heated particulate filter (can be in-stack or ex-stack)
4 Heating jacket or heating bondage
5 Heated sampling pump

Figure 2 - Example of a sample device used for TOC sampling