SLOVENSKI STANDARD
SIST EN 12619:2000

01-januar-2000

Emisije nepremiļ nih virov - Doloļ evanje nizkih masnih koncentracij celotnega organskega ogljika v plinasti fazi v odpadnih plinih - Kontinuirana metoda plamenske ionizacijske detekcije

Stationary source emissions - Determination of the mass concentration of total gaseous organic carbon at low concentrations in flue gases - Continuous flame ionisation detector method

Emissionen aus stationären Quellen - Bestimmung der Massenkonzentration des gesamten gasförmigen organisch gebundenen Kohlenstoffs in geringen Konzentrationen in Abgasen - Kontinuierliches Verfahren unter Verwendung eines Flammenionisationsdetektors

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

Ta slovenski standard je istoveten z: **EN 12619:1999**

ICS:

13.040.40 Emisije nepremiļ nih virov Stationary source emissions

SIST EN 12619:2000 **en**

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 12619

June 1999

ICS 13.030.40; 13.040.40

English version

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This European Standard was approved by CEN on 6 May 1999.

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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 Central Secretariat has the same status as the official versions.

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

Central Secretariat: rue de Stassart, 36 B-1050 Brussels

Ref. No. EN 12619:1999 E

Contents

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of an identical text or by endorsement, at the latest by December 1999, and conflicting national standards shall be withdrawn at the latest by December 1999.

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

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 stationary source combustion emissions.

This European Standard is suitable for the measurement of low level gaseous or vapour phase TOC emissions such as those from municipal waste incinerators and hazardous waste incinerators.

NOTE: See Council Directive 89/369/EEC which is under revision and Council Directive 94/67/EC.

This standard is not recommended for performing measurements on solvent using processes. Minimum operational requirements for long term emissions monitoring are suggested in annex A; it is likely that these will be modified by subsequent European Standards. 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 0 mg/m^3 to 20 mg/m^3.

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 B.

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.

ISO 6879: 1995 Air quality - Performance characteristics and related concepts for air quality measuring methods.
ISO 7504: 1984 Gas analysis - Vocabulary

3 Terms and definitions

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

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

3.2 Complementary gas: The most abundant component, usually nitrogen, in a gas mixture.

3.3 Control gas mixture: A gas mixture used to check the minimum performance requirements of the FID.

3.4 Detection threshold: The minimum concentration of a substance which produces an observable response, as detailed in annex C and referred to in ISO 7504:1984.

3.5 Flame ionisation detector (FID): An instrument using flame ionisation detection.

3.6 Flue gas: The product from a combustion or incineration process containing gaseous and particulate components.

3.7 Fuel gas: A gas of known composition used to maintain the flame of the FID.

3.8 Mass concentration of gaseous total organic carbon: The 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.

3.9 Range: The 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: The dimensionless quotient of the response of the FID with any carbon based compound or compounds to its response to propane.

3.11 Response time: The 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: A gas used to adjust and check a specific point on a calibration curve.

3.13 Total organic carbon (TOC): A measure of the amount of gaseous or vapour phase, organic carbon which, within the context of this Standard is measured by the FID and expressed as total organic carbon in milligrams per cubic metre.

3.14 Zero gas: A gas or a gas mixture used to check and adjust the zero point on a calibration curve.

4 The principle of the technique

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.

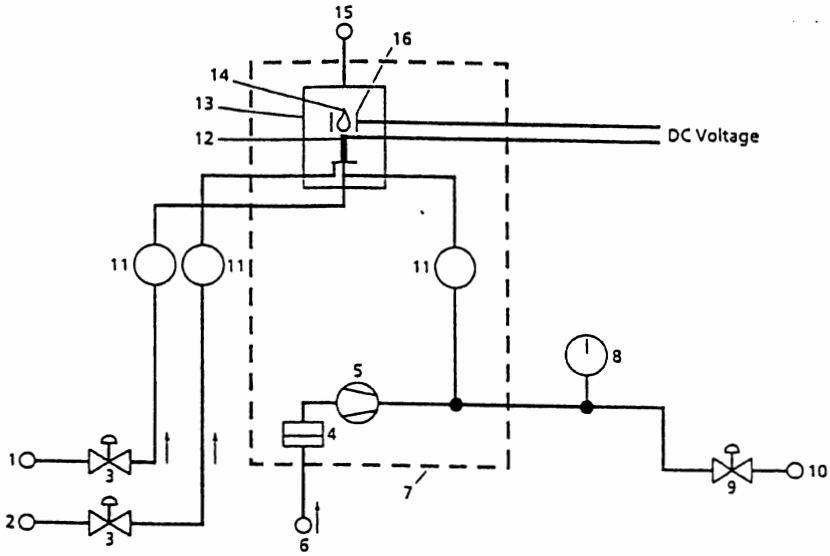
4.2 Flame ionisation detector

The measurement effect utilised by the 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 main advantage of the FID is that it responds strongly to organic carbon containing components and less to other 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. Pressure regulator
4. Fine dust filter
5. Sampling gas pump
6. Sample gas
7. Heated housing
8. Pressure gauge
9. Back-pressure regulator
10. Bypass
11. Flow meters
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 (C_3H_8) for which the response factor, defined in this Standard, has been set at 1. The final value will be expressed as TOC in milligrams per cubic metre.

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, including the filter needed to remove fine particles which could clog the burner, is heated to avoid sample condensation.

5 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 characteristics defined in table 1.

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

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

Performance characteristics (see Note 1)	Minimum performance requirements
Measuring range (see Note 2)	0 mg/m³ to 20 mg/m³
Detection threshold	0,4 mg/m³
Response time (0 % to 90 %)	less than 1 min
Linearity deviation	permissible deviation 0,4 mg/m³
Range of response factors:-	Permissible range
Aliphatic hydrocarbons (see Note 3)	0,90 to 1,10
Aromatic hydrocarbons (see Note 4)	0,85 to 1,10
Methylene chloride	0,75 to 1,15
Control gas mixture	permissible deviation 15 % of the given concentration
Oxygen interference (see Note 5)	permissible interference 0,8 mg/m³
Effects of interference gases	permissible interference 1 mg/m³

Note 1: The methods for determining instrument characteristics are given in Annex C.
Note 2: The instrument shall be capable of measuring concentrations up to 50 mg/m³.
Note 3: For the purposes of this Standard the aliphatic hydrocarbons are represented by methane and ethane, see Annex C.
Note 4: For the purposes of this Standard the aromatic hydrocarbons are represented by benzene and toluene, see Annex C.
Note 5: Oxygen interference shall be determined as shown in Annex C.

5.1.2 Sampling device

Several acceptable configurations exist, a typical example is shown in figure 2.

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

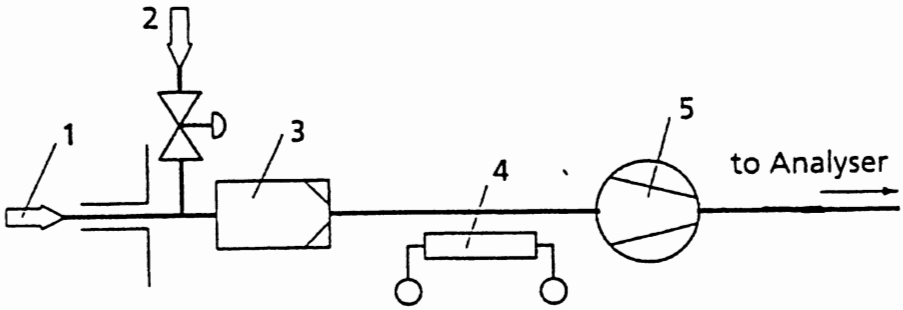
- it shall be made of a material that is chemically and physically inert to the constituents of the flue gas under analysis;

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

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

- it 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;

- 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 bandage
5. Heated sampling pump

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

5.2 Operational gases

A number of operational gases are required when using the Standard. If these are supplied in compressed gas cylinders they shall be fitted with regulators.

5.2.1 Combustion air

The TOC concentration of the combustion air shall not exceed 0,2 mg/m^3.

5.2.2 Fuel gases

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

- hydrogen;
- hydrogen/helium mixture;
- hydrogen/nitrogen mixture.

The concentration of TOC shall not exceed 0,2 mg/m^3. Gases with a purity of 99,999 % (percent by volume) usually meet this requirement.

NOTE. The fuel gas pipe should be made from stainless steel or copper.

5.2.3 Zero gas

Nitrogen (containing less than 0,2 mg/m^3 TOC) should be used but air can be used provided that the organic carbon concentration does not exceed 0,2 mg/m^3.

5.2.4 Span gas

The span gas shall be propane. It shall be prepared in a complimentary gas containing less than 0,2 mg/m^3 TOC. It shall have a known concentration and the analysis shall have a maximum permissible uncertainty of 2 %. The span gas should have a TOC concentration of approximately 16 mg/m^3.

5.2.5 Control gas mixture

For the purposes of this Standard the control gas mixture shall consist of methane (approximately 2,0 mg/m^3), ethane (approximately 1,5 mg/m^3), toluene (approximately 0,5 mg/m^3), benzene (approximately 0,5 mg/m^3) and methylene chloride (approximately 0,5 mg/m^3) with oxygen (approximately 11 %), carbon dioxide (approximately 10 %), carbon monoxide (approximately 50 mg/m^3) and nitrogen as the complimentary gas; the total organic carbon concentration shall be expressed in milligrams per cubic metre. The mixture shall be of a known composition and the TOC analysis shall have a maximum permissible uncertainty of 6 %. The control gas mixture shall be prepared in a pressurised gas cylinder and the manufacturer's advice shall be followed for storage and transport.

5.2.6 Gases for determining interferences

The procedure for testing interference is given in C.1.3.

6 Measurement procedure

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 C.

6.1 Adjustments and checks

6.1.1 Instrument adjustment

The FID shall be set up according to the manufacturer's instructions in order to ensure that the instrument is correctly adjusted to meet the requirements of table 1. The safety procedures detailed in annex D shall be followed.

The zero and span gas shall be introduced under the same flow and pressure conditions using the sample port or according to the manufacturers instructions when using individual zero and span ports. The adjustment procedure shall be as follows:

a) feed the zero gas into the FID and set the zero;