



SLOVENSKI STANDARD

SIST EN 13649:2002

01-maj-2002

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Stationary source emissions - Determination of the mass concentration of individual gaseous organic compounds - Activated carbon and solvent desorption method

Emissionen aus stationären Quellen - Bestimmung der Massenkonzentration von einzelnen gasförmigen organischen Verbindungen - Aktivkohleadsorptions- und Lösemitteldesorptionsverfahren

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Emissions de sources fixes - Détermination de la concentration massique en composés organiques gazeux individuels - Méthode par charbon actif et description des solvants

Ta slovenski standard je istoveten z: EN 13649:2001

ICS:

13.040.40 0{ ã ã Á ^] | ^ { ã } ã ã [ç Stationary source emissions

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EUROPEAN STANDARD

EN 13649

NORME EUROPÉENNE

EUROPÄISCHE NORM

November 2001

ICS 13.040.40

English version

Stationary source emissions - Determination of the mass concentration of individual gaseous organic compounds - Activated carbon and solvent desorption method

Emissions de sources fixes - Détermination de la concentration massique en composés organiques gazeux individuels - Méthode par charbon actif et désorption des solvants

Emissionen aus stationären Quellen - Bestimmung der Massenkonzentration von einzelnen gasförmigen organischen Verbindungen - Aktivkohleadsorptions- und Lösemitteldesorptionsverfahren

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

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Management Centre or to any CEN member.

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.



EUROPEAN COMMITTEE FOR STANDARDIZATION
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EN 13649:2001 (E)

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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 annexes A, B, C, D and E 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.

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1 Scope

This European Standard specifies procedures for the sampling onto activated carbon, the preparation and the analysis of samples of volatile organic components such as those arising from solvent using processes. It can be used as a reference method.

NOTE See Council Directive 1999/13/EEC.

The results obtained using this Standard are expressed as the mass concentration (mg/m^3) of the individual gaseous organic components. This Standard is suitable for use in the range of about $0,5 \text{ mg}/\text{m}^3$ to $2000 \text{ mg}/\text{m}^3$.

For the measurement of the mass concentration of total organic carbon arising from solvent using processes then EN 13526 should be used.

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).

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EN 13526:2001, *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.*

ISO 5725-1, *Accuracy (trueness and precision) of measurement methods and results Part 1: General principles and definitions.*

ISO 9169, *Air Quality – Determination of performance characteristics of measurement methods.*

3 Terms and definitions

For the purposes of this European Standard the following terms and definitions apply.

3.1**desorption efficiency**

ratio of the mass of the recovered organic material to the mass of organic material added to the carbon adsorbent expressed as a percentage

3.2**detection limit**

minimum concentration of a substance which produces an observable response, as referred to in ISO 9169

3.3**dilution gas**

gas used to dilute sampled flue gas to prevent water condensation

3.4**flue gas**

gaseous waste product from a solvent using process

4 Principle**4.1 General**

There are three steps in the measurement of individual gaseous organic components. They are flue gas sampling, the treatment of sampled material, and the chemical analysis by gas chromatography.

4.2 Flue gas sampling

The principles of sampling are as follows:

- Organic components from a measured volume of gas shall be adsorbable onto the activated carbon.
- Particulate material which might interfere with the measurement should be removed.

- Water condensation should be prevented.

4.3 Treatment of sampled material

The collected organic material shall be desorbed using an extraction solvent.

4.4 Chemical analysis

An aliquot of the sample is injected into a gas chromatography system equipped with a capillary column and an appropriate detector (e.g. flame ionisation detector, mass selective detector etc.). The components are separated by gas chromatography. The detector signals are evaluated using the calibration function.

4.5 Desorption efficiency determination

At the sample preparation stage a known amount of a reference material is introduced on to a blank carbon tube to determine component desorption efficiency.

The desorption efficiency can be determined as shown in annex B.

NOTE The desorption efficiency should be better than 80 %.

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5 Apparatus and materials SIST EN 13649:2002

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5.1 Sampling system – general aspects

The sampling system 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;

NOTE 1 Stainless steel, glass, polytetrafluoroethylene and polypropylenefluoride are well proven construction materials.

- the sampling line shall be as short as possible;
- the sampling line shall include a filtering device directly after the probe to trap particles;
- it shall be designed to prevent condensation.

NOTE 2 Many of the solvent using processes covered by the Council Directive 1999/13/EEC produce flue gases which do not have a high water content. Where high solvent concentrations or the condensation of water vapour is expected this CEN Standard recommends the use of dilution sampling systems.

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Examples of systems suitable for sampling both dry and wet flue gases are described in this standard.

5.1.1 Sampling without dilution

An example of a suitable sampling system is shown in Figure 1.

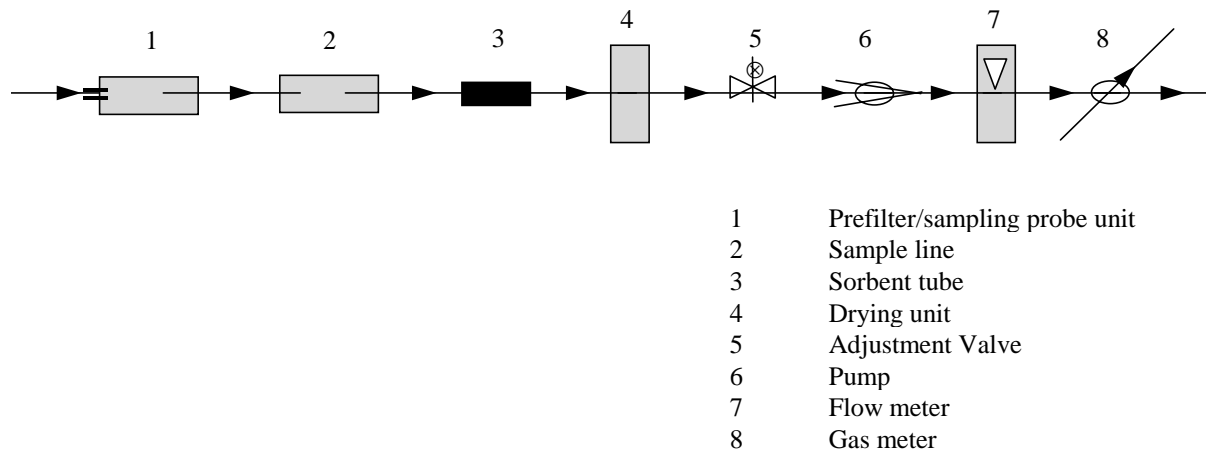


Figure 1 - Example of a flue gas sampling system

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5.1.2 Sampling using dynamic dilution

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An example of a dynamic dilution system is shown in Figure 2.

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The dilution should be adjustable and shall be constant to within 5 % during operation.

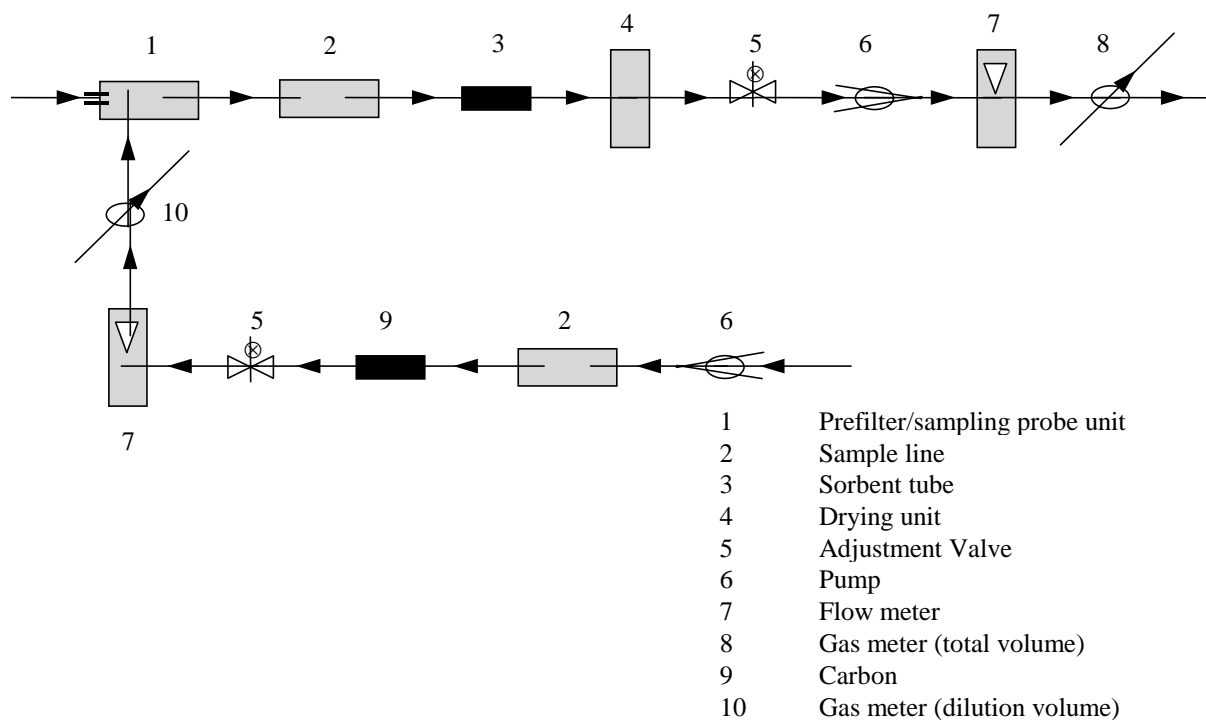


Figure 2 - Example of a dynamic dilution sampling system

5.1.3 Sampling using static dilution

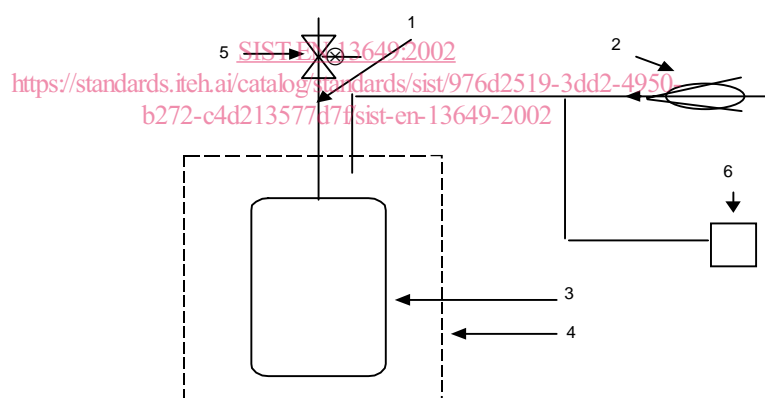
Static dilution sampling can be carried out by part filling a sample bag, fabricated from an inert material, with a known volume of dry dilution gas, a known volume of flue gas is then added. A heated sampling probe can be used to ensure that no condensation occurs before the sample enters the bag. Following sampling the bag is emptied through the sorbent tube.

NOTE 1 A suitable procedure is the 'lung principle' in which a sample bag is placed in a rigid, leak proof container, the air is removed from the container using a vacuum pump, the reduced pressure in the container causes the bag to fill with a volume of sample equal to that which has been removed from the container. A pressure control device operating at not greater than 250 Pa should be used to prevent the bag bursting.

NOTE 2 A suitable bag material include polyvinyl fluoride.

NOTE 3 A suitable procedure for collecting diluted gas samples is given in annex D.

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Key

- 1 PTFE tube
- 2 Pump
- 3 Bag (partially filled with dilution gas)
- 4 Rigid container
- 5 PTFE valve
- 6 Pressure control device

Figure 3 - Example of a static dilution sampling system

EN 13649:2001 (E)**5.1.4 Sorbent tube**

The sorbent tube, filled with activated carbon as the adsorbent, shall have the following characteristics:

- a main adsorbent layer containing not less than 100 mg of activated carbon;

NOTE 1 A suitable material is activated carbon prepared from coconut shell.

- a security adsorbent layer to detect breakthrough;
- the sorbent tube construction material shall be inert;

NOTE 2 A suitable material is glass.

- the sorbent tubes shall be free from contamination.

Open or used sorbent tubes shall not be reused. Sorbent tubes shall be used in accordance with the manufacturers instructions to avoid leakage and sample loss.

NOTE 3 Typical desorption efficiencies of common solvents from tubes of this type are given in annex B.

5.1.5 Dilution gas

Dilution gas should be nitrogen or dry air containing less than 0.1% of the emission limit value of any of the principal components under investigation.

NOTE Nitrogen of purity 99,999 % or ambient air, purified by passage through activated carbon, normally meets these requirements.

5.2 Analytical reagents**5.2.1 General**

Only reagents of recognised analytical grade or better quality shall be used.

5.2.2 Extraction Solvent

Extraction solvents shall be of chromatographic quality and free from components co-eluting with the components of interest.

NOTE 1 Carbon disulphide (CS₂) is a suitable extraction solvent for most of the components likely to be encountered in solvent using processes.

NOTE 2 For polar substances other extraction solvents can be used (see annex B).

5.2.3 Reference materials for calibration of the analytical procedure

The chromatograph shall be calibrated with those reference materials which correspond to the components likely to arise in the process under investigation.

For the purpose of calibration the reference materials shall be prepared in a solution of the extraction solvent to be used. The extraction solvents are highly volatile and fresh reference standards should be prepared regularly.

5.3 Analytical apparatus

Laboratory apparatus suitable for capillary column chromatography shall be used.

6 Sampling procedure

6.1 General Aspects

The sample shall be representative of both the process emission and the local distribution of the measured constituents in the sampling plane.

The following points should be considered when sampling:

- the nature of the plant process e.g. steady state or cyclic. If possible the sampling programme should be carried out under steady operating conditions of the plant;
- the expected concentration to be measured and any required averaging period, both of which can influence the sampling time.

6.2 Choice of sampling procedure

The choice of sampling procedure shall be governed by the characteristics of the gas.

Sampling directly onto the sorbent material can be used when the flue gas does not have a high water content and the mass concentration of (organic) material is such that break through is unlikely to occur.

The temperature of the flue gas should not exceed 40 °C during adsorption. Provided that no condensation occurs hotter flue gas can be cooled down using a cooling section (inert sampling line). Sampling using dilution or equivalent methods shall be used if the exhaust gas is so moist that condensation occurs or the mass concentration of organic components is such that there is a risk of exceeding the capacity of the sorbent tubes. Dilution can be either static or dynamic.

NOTE A suitable procedure for dilution sampling is given in annex D.

6.3 Minimum sampling time and flow rate

The sampling time shall be chosen taking into consideration any required averaging period, process conditions, sampling and analytical equipment, and the lower detection limit of the