SLOVENSKI STANDARD
SIST ISO 10849:1996

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Stationary source emissions -- Determination of the mass concentration of nitrogen oxides -- Performance characteristics of automated measuring systems

iTeh STANDARD PREVIEW

Émissions de sources fixes -- Détermination de la concentration en masse des oxydes d'azote -- Caractéristiques de performance des systèmes de mesurage automatiques

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Ta slovenski standard je istoveten z: **ISO 10849:1996**

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INTERNATIONAL STANDARD

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Stationary source emissions — Determination of the mass concentration of nitrogen oxides — Performance characteristics of automated measuring systems

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Reference number
ISO 10849:1996(E)

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 10849 was prepared by Technical Committee ISO/TC 146, *Air quality*, Subcommittee SC 1, *Stationary source emissions*.

Annex A forms an integral part of this International Standard. Annexes B and C are for information only.

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ISO 10849:1996(E)

Introduction

Nitrogen oxides are produced during most combustion processes. In fossil fuel combustion, nitrogen oxides are produced from nitrogen combined in the fuel and from the oxidation of nitrogen in the air used for combustion. The quantity of nitrogen oxides produced depends upon the nitrogen content of the fuel, the boiler design, the burner design and the boiler operating conditions.

In flue gases from conventional combustion systems, the nitrogen oxides consist of approximately 95 % nitrogen monoxide (NO). The remaining oxide is predominantly nitrogen dioxide (NO_2) formed from the oxidation of NO when the flue gas temperature decreases. These two oxides (NO + NO_2) are generally designated as NO_x. It should be noted that in other processes the ratio of NO to NO_2 may be different and other nitrogen oxides may be present.

There are numerous ways of determining nitrogen oxides in the gases of combustion plants, both wet chemical/analytical methods and instrumental techniques.

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ISO 10849:1996(E)

Stationary source emissions — Determination of the mass concentration of nitrogen oxides — Performance characteristics of automated measuring systems

1 Scope

This International Standard specifies the fundamental structure and the most important performance characteristics of automated measuring systems for oxides of nitrogen to be used on stationary source emissions, for example combustion plants. The procedures to determine the performance characteristics are also specified. Furthermore, it describes methods and equipment to determine NO or NO_x ($NO + NO_2$) in flue gases including the sampling system and sample gas conditioning system. Dinitrogen monoxide (N_2O) is not determined by the methods described in this International Standard. The given performance characteristics refer to the complete measuring system, from sampling to analyser.

This International Standard describes extractive and non-extractive systems in connection with a range of analysers that operate using, for example, the following principles:

— chemiluminescence;

— non-dispersive infrared spectroscopy;

— non-dispersive ultraviolet spectroscopy;

— differential optical absorption spectrometry.

NOTE 1 Commercial devices using the described techniques, that meet the requirements of this International Standard, are available.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publi-

cation, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 6879:1995, *Air quality — Performance characteristics and related concepts for air quality measuring methods.*

ISO 7996:1985, *Ambient air — Determination of the mass concentration of nitrogen oxides — Chemiluminescence method.*

ISO 9096:1992, *Stationary source emissions — Determination of concentration and mass flow rate of particulate material in gas-carrying ducts — Manual gravimetric method.*

ISO 9169:1994, *Air quality — Determination of performance characteristics of measurement methods.*

ISO 10396:1993, *Stationary source emissions — Sampling for the automated determination of gas concentrations.*

3 Definitions

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

3.1 automated measuring system (AMS): System that may be attached to a chimney to continuously measure and record the mass concentration of nitrogen oxides passing through the chimney.

3.2 analyser: Analytical part in an extractive AMS.

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3.3 verified (AMS): AMS previously verified against this International Standard.

3.4 calibration gas: Gas of known, reliable and stable composition that may be used to check the response of the AMS and should be used for the calibration of the AMS.

3.5 comparative measurements: Measurements that are taken on the same chimney in the same sampling plane for the same period of time, with the AMS under test and with the comparative method providing pairs of measured values.

3.6 comparative method: Defined test method for the comparative measurements of stationary source emissions containing nitrogen oxides. This can be a manual method or an AMS verified according to this International Standard, with a different measuring principle.

NOTE 2   The naphthylethylenediamine (NEDA) method according to ISO 11564, has been proven to be a suitable manual method. Also, validated national standards with known performance characteristics (standard deviation, lower detection limit, effect of interfering substances) may be applied.

3.7 standard deviation, s_A: Measure of the working precision of the AMS.

The standard deviation, s_A, is derived from the difference in the pairs of measured values of nitrogen oxides by the AMS under test and the comparative method, on the basis of a sufficient number of comparative measurements spread over the period of unattended operation (see annex A).

  s_c is the standard deviation of the comparative method.

  s_D is the standard deviation of the paired values.

  s_x is the standard deviation of the blank readings.

NOTES

3   It is not possible to determine directly the standard deviation, s_A, of an AMS under repeatable working conditions or in a laboratory, because:

— commercially available calibration gas mixtures containing nitrogen monoxide do not have all the properties of actual waste gas and do not cover all possible influences;

— the mass concentration of nitrogen oxides in waste gas usually varies with time;

— it is not possible to maintain the properties of a waste gas present in the waste gas flue in a waste gas sample transferred into a vessel.

Therefore, the evaluation of the standard deviation, s_A, is performed by comparison with an independent manual method or an analyser with a different principle of detection.

Applying the comparative method in combination with the test for systematic errors ensures a satisfying accuracy of the automated measuring system.

4   The standard deviation, s_A, is a measure of the working precision under site conditions. Therefore, it contains, in addition to random errors, the effects of interfering substances, the effects of temperature changes and of any zero and span drifts, because they cannot be eliminated in practice.

The standard deviation, s_A, is an upper limiting value for the AMS. Known systematic errors of the measured values of the independent comparative method are to be taken into account.

5   This is suitable for finding the precision of the measuring result of the automated measuring method, as long as the standard deviation, s_c, of the measured values of the comparative method is significantly smaller than the standard deviation, s_D, of the difference in pairs of measured values.

If the AMS under investigation has a substantially smaller standard deviation, s_A, than the comparative method, s_c, the method above can still be used, although the value of s_A will have a large uncertainty. If the uncertainty in s_c is unknown and hence the limits of s_A cannot be established, the value of s_A can be used as a qualitative rather than a quantitative assessment of the AMS performance.

3.8 chimney: Stack or final exit duct on a stationary process used for the dispersion of residual process gases.

3.9 mass concentration: Concentration of a substance in an emitted waste gas, expressed in milligrams per cubic metre.

NOTE 6   The concentration of nitrogen oxides can be expressed as ppm, as milligrams of NO per cubic metre or as milligrams of NO_2 per cubic metre.

NO: 1 ppm $(V/V) = 1{,}34$ mg/m^3

NO_2: 1 ppm $(V/V) = 2{,}05$ mg/m^3

Concentrations should be related to standard atmospheric conditions (273 K, 101,3 kPa) and dry gas.

Depending on national regulations, the concentrations should be referred to defined oxygen or carbon dioxide concentrations.

In this International Standard, all concentrations of nitrogen oxides are expressed as milligrams of NO_2 per cubic metre.

3.10 stationary source emissions: Those gases that have been emitted by a stationry plant or process and are transported to a chimney for dispersion into the atmosphere.

3.11 calibration curve: Curve describing the dependence of the measured signal on a given calibration gas.

3.12 period of unattended operation: Maximum admissible interval of time for which the performance characteristics will remain within a predefined range without external servicing, e.g. refill, calibration, adjustment. [ISO 6879]

NOTE 7   For long-term monitoring installations a minimum of 7 d of unattended operation is required.

3.13 calibration: Setting and checking of the AMS before determining the performance characteristics or before beginning any measurement of NO_x. Further steps of the calibration of an AMS, like comparative measurements, may be part of national regulations.

4   Principle

With extractive systems, a representative sample of gas is taken from the stack with a sampling probe and conveyed to the analyser through the sample line and sample gas conditioning system.

Non-extractive systems do not require any sampling transfers out of the stack. For the installation of these systems, a representative place in the stack is to be chosen. *In-situ* systems may sample a larger part of the flue gas.

The values from the analyser are recorded and/or stored by means of electronic data processing.

The systems described here basically only measure nitrogen monoxide. If, with the systems, the nitrogen dioxide content or the total quantity of the nitrogen oxides ($NO + NO_2$) is to be determined, a converter to reduce nitrogen dioxide to nitrogen monoxide is to be used. The converter may be a separate piece of equipment or incorporated into the NO analyser.

Systems also exist, mainly using ultraviolet techniques, that can monitor nitrogen dioxide directly. These systems are mostly combined with NO analysers.

In most of the cases, it is considered that only nitrogen monoxide has to be measured, because the NO_2 content is negligible. However, in some cases nitrogen dioxide may occur in large quantities and has to be taken into account, either by direct measurement or by using a converter. The sampling will, however, be difficult, due to the high reactivity of nitrogen dioxide.

5   Description of the measuring equipment

5.1   Sampling and sample gas conditioning systems for extractive systems
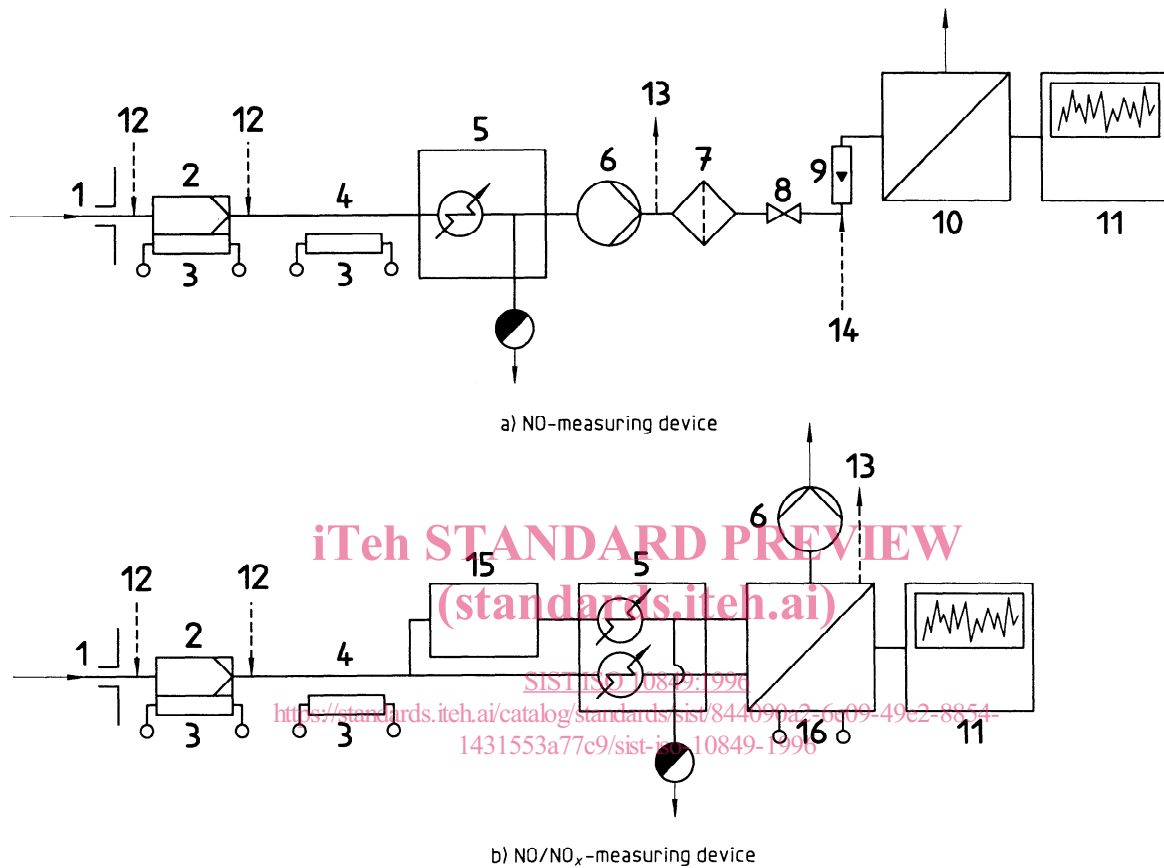
5.1.1   General

A more detailed description of sampling and sample gas conditioning systems for extractive methods is given in ISO 10396.

Figure 1 a) shows a typical arrangement of a complete measuring system for NO. This system is suitable for use with all the analysers that are described in 5.2.

In addition to this arrangement, there are also automated measuring systems for the NO_x measurement that heat the sample gas to above water and acid dew-points (or the dew-point of other condensable substances) to avoid losses of NO_2. In this case, the system can be simplified. It is important that all the components carrying the sample gas to the analyser are also heated above water and acid dew-points.

In the case that higher amounts of NO_2 are in the sample gas, the use of a gas cooler can produce errors on the NO_2 measurement due to the solubility of NO_2 in the condensed water and depending on the content of water vapour in the flue gas. A possible arrangement to avoid losses of NO_2 is shown in figure 1 b).

The sampling of gas shall be representative, that is, the sampling location shall be typical of the entire duct. The representativeness of the sampling location requires confirmation by means of a network measurement in accordance with the guidelines given in ISO 10396. The sampling points for the network measurement shall be located in accordance with ISO 9096. Checking of representativeness shall be done before the first installation of a measuring system and shall be repeated in the case of uncertainty.

a) NO-measuring device

b) NO/NO$_x$-measuring device

Key:

1	Gas sampling probe	9	Flowmeter
2	Particulate filter	10	NO-analyser
3	Heating	11	Recorder
4	Sampling line (heated if necessary)	12	Inlet for zero and calibration gas (preferably in front of the filter) to check the complete system
5	Sample cooler with condensate separator	13	Bypass for excess gas
6	Sample pump	14	Inlet for zero and span gas to check the analyser
7	Filter	15	Converter
8	Needle valve	16	NO/NO$_x$-analyser

Figure 1 — Examples of the installation of measuring devices

The components described in 5.1.2.1 to 5.1.2.8 have, for example, proven to be successful for measurements at gas-, oil- and coal-fired plants (precautions need be observed because of the high corrosiveness of condensable acid gases, e.g. HCl, SO_3 or NO_2).

5.1.2 Components

5.1.2.1 Sampling probe, made of suitable, corrosion-resistant material. For gas temperatures up to 220 °C, polytetrafluoroethylene (PTFE) is an acceptable material. At temperatures > 250 °C, stainless steel and certain other materials can alter the ratio of $NO:NO_2$. In this case, ceramic or glass material is required, if it is necessary to determine the ratio. Cooling may be considered necessary in order to maintain the gas concentrations in the flue gas.

5.1.2.2 Filter, made of ceramic or sinter metal with 10 μm pore size. The filter shall be heated above the water or acid dew-point.

5.1.2.3 Sample line, made of PTFE or stainless steel. The lines shall be operated 15 K above the dew-point of condensable substances (generally the water or acid dew-point). The inner diameter of the line depends on the quantity of sample gas required, with 4 mm as a minimum (and preferably 4 mm to 8 mm).

5.1.2.4 Sample cooler or permeation drier, to separate water vapour from the flue gas. The dew-point shall be sufficiently below the ambient temperature. A cooling temperature of 2 °C to 5 °C is convenient. Sufficient cooling is required for the volume of gas being sampled and the amount of water vapour that it contains.

The design of the sample gas cooler shall be such that absorption of NO_2 in the condensate is restricted to a minimum. This ensures that loss of NO_2 dissolved in the condensate, which is drained from the sample cooler, is at a minimum.

The use of a permeation drier also ensures that NO_2 losses are negligible.

5.1.2.5 Sampling pump (corrosion-resistant), of which the performance shall be such that it can supply the connected analyser with its required gas flow. The quantity of sample gas required can vary between

30 l/h and 500 l/h, dependent upon the analyser and the expected response time.

5.1.2.6 Secondary filter, to separate fine dust, with a pore size of 1 μm to 2 μm, made for example from glass fibre, sintered ceramics, stainless steel or PTFE fibre.

5.1.2.7 Flow controller and flowmeter, to set the required flow and constructed of corrosion-resistant material.

5.1.2.8 NO_2/NO converter, necessary if NO_2 has to be measured with a NO analyser (only possible in combination with extractive systems).

Different types of converters exist, for example:

— carbon converters;

— carbon-molybdenum converters;

— stainless steel converters;

— thermal converters.

In some situations (e.g. when ammonia is present in the sample gas) interferences can occur depending on the operating temperature of the converter. In these cases, it is necessary to take into account such possibilities when selecting the converter type.

The converter can be bypassed with a three-way valve. If the sample gas flows through the converter, the total quantity (NO + NO_2) is obtained; when the converter is bypassed, the NO content is obtained. The amount of NO_2 can be calculated as the difference between NO_x and NO.

The converter shall have an efficiency > 95 %, which can be tested using calibration gases containing NO_2 in synthetic air, or with a converter efficiency tester. This method is described in detail in ISO 7996 and it is not suitable if the NO analyser is prone to interference by ozone.

The principle of an efficiency tester is shown in figure 2. A constant flow of a NO-calibration gas is mixed with a constant flow of air or oxygen, that contains different amounts of ozone, produced by an adjustable ozone generator. Ozone reacts with NO to produce NO_2. Thus the total amount of nitrogen oxides (NO + NO_2) remains constant, while the ratio ($NO:NO_2$) changes.

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