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**Stationary source emissions —  
Automatic method for the determination  
of the methane concentration using flame  
ionisation detection (FID)**

*Émissions de sources fixes — Méthode automatique pour la  
détermination de la concentration en méthane par détection à ionisation  
de flamme (FID)*

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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.

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Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 25140 was prepared by Technical Committee ISO/TC 146, *Air quality*, Subcommittee SC 1, *Stationary source emissions*.

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## Introduction

Methane (CH<sub>4</sub>) is a gas of relevance to the climate (greenhouse gas) and contributes directly to the atmospheric greenhouse effect. The emissions of methane originate from natural and anthropogenic sources. Significant sources are, for example, cattle breeding, cultivation of rice, extraction and transport of natural gas, and landfills. Other important sources contributing to emissions of methane are, for example, composting plants, the use of biogas and natural gas, and biomass firings. This International Standard specifies a method of measurement for the determination of methane emissions from stationary sources.

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# Stationary source emissions — Automatic method for the determination of the methane concentration using flame ionisation detection (FID)

## 1 Scope

This International Standard specifies the principle, the essential performance criteria, and quality assurance and quality control procedures for an automatic method for measuring methane in the waste gas of stationary sources using flame ionisation detection. It is applicable to measurements of methane in dry or wet waste gases. The method allows continuous monitoring with permanently installed measuring systems as well as intermittent measurements of methane emissions.

NOTE 1 This International Standard is specific to automatic methods for measuring methane in the waste gas of stationary sources using flame ionisation detection. It supplements the general requirements of other international or national standards on performance testing, QA/QC procedures, and the test report as specified, for example, in EN 15267-3<sup>[7]</sup>, EN 14181<sup>[5]</sup>, and EN 15259<sup>[6]</sup>.

This International Standard does not specify an independent method of measurement.

NOTE 2 An independent method of measurement, e.g. to calibrate or validate permanently installed measuring systems, is specified in ISO 25139<sup>[3]</sup>.

NOTE 3 In EN 14181<sup>[5]</sup>, “independent method of measurement” is called “standard reference method (SRM)”.

## 2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 9169:2006, *Air quality — Definition and determination of performance characteristics of an automatic measuring system*

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

ISO 20988, *Air quality — Guidelines to estimating measurement uncertainty*

## 3 Terms and definitions

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

### 3.1

#### automatic measuring system

#### AMS

⟨air quality⟩ measuring system interacting with the waste gas under investigation, returning an output signal proportional to the physical unit of the measurand in unattended operation

NOTE 1 Adapted from ISO 9169:2006, 2.1.2.

NOTE 2 In the sense of this document, an AMS is a system that can be attached to a duct to continuously or intermittently measure and record the mass concentrations of methane passing through the duct.

**3.2  
analyser**

⟨stationary source emissions⟩ analytical part in an extractive or *in situ* automatic measuring system

NOTE Adapted from ISO 12039:2001<sup>[2]</sup>, 3.3.

**3.3  
measurand**

particular quantity subject to measurement

[ISO/IEC Guide 98-3:2008<sup>[4]</sup>, B.2.9]

EXAMPLE The mass concentration of methane in air.

**3.4  
mass concentration**

⟨stationary source emissions⟩ concentration of a substance in an emitted waste gas expressed as mass per volume

[ISO 12039:2001<sup>[2]</sup>, 3.10]

NOTE Mass concentration is often expressed in milligrams per cubic metre.

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**3.5  
independent reading**

⟨stationary source emissions⟩ reading that is not influenced by a previous individual reading by separating two individual readings by at least four response times

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**3.6  
individual reading**

⟨stationary source emissions⟩ reading averaged over a time period equal to the response time of the automatic measuring system

**3.7  
interferent**

interfering substance

⟨air quality⟩ substance present in the air mass under investigation, other than the measurand, that affects the response

[ISO 9169:2006, 2.1.12]

**3.8  
adjustment**

⟨automatic measuring system⟩ operation of bringing an automatic measuring system into a state of performance suitable for its use

NOTE Adjustment can be automatic, semi-automatic or manual.

[ISO 9169:2006, 2.1.5]

**3.9  
calibration**

⟨stationary source emissions⟩ procedure for establishing the statistical relationship between values of the measurand indicated by the automatic measuring system and the corresponding values given by an independent method of measurement implemented simultaneously at the same measuring point



NOTE 1 An independent method of measurement for the purpose of calibration of permanently installed methane measuring systems is specified in ISO 25139<sup>[3]</sup>.

NOTE 2 In EN 14181<sup>[5]</sup>, “independent method of measurement” is called “standard reference method (SRM)”.

### 3.10 interference

⟨air quality⟩ negative or positive effect upon the response of the measuring system, due to a component of the sample that is not the measurand

### 3.11 zero gas

⟨stationary source emissions⟩ gas or gas mixture used to establish the zero point on a calibration curve within a given concentration range

[ISO 12039:2001<sup>[2]</sup>, 3.4.2]

### 3.12 span gas

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

NOTE Adapted from ISO 12039:2001<sup>[2]</sup>, 3.4.1.

### 3.13 reference gas

⟨stationary source emissions⟩ gas of known, reliable and stable composition that may be used to check the response of an automatic measuring system and to calibrate the automatic measuring system

### 3.14 zero point

⟨stationary source emissions⟩ specified value of the output quantity (measured signal) of the automatic measuring system and which, in the absence of the measured component, represents the zero crossing of the calibration line

### 3.15 span point

value of the output quantity (measured signal) of the automatic measuring system for the purpose of calibration or adjustment that represents a correct measured value generated by a reference material

NOTE This concentration is often chosen to be around 80 % of the upper limit of the measuring range or around the emission limit value.

### 3.16 performance characteristic

⟨air quality⟩ one of the quantities assigned to equipment in order to define its performance

NOTE Performance characteristics can be described by values, tolerances or ranges.

### 3.17 response time

⟨air quality⟩ time interval between the instant when a stimulus is subjected to a specified abrupt change and the instant when the response reaches and remains within specified limits around its final stable value, determined as the sum of the lag time and the rise time in the rising mode, and the sum of the lag time and the fall time in the falling mode

[ISO 9169:2006, 2.2.4]

**3.18**

**lag time**

⟨air quality⟩ by convention, time taken for the output signal to reach 10 % of the final change in the output signal when a step function is applied by applying a reference material to the automatic measuring system initially in the basic state

[ISO 9169:2006, 2.2.2]

**3.19**

**rise time**

⟨air quality⟩ by convention, time taken for the output signal to pass from 10 % to 90 % of the final change in the output signal when a reference material is abruptly applied to the automatic measuring system initially in the basic state

NOTE For instruments where transient oscillations occur in the approach to the final output signal, 90 % of the final change is considered as reached when the oscillations fall to less than 10 % of the final change in the output signal.

[ISO 9169:2006, 2.2.3]

**3.20**

**fall time**

⟨air quality⟩ by convention, time taken for the output signal to pass from 90 % to 10 % of the initial output signal produced by a reference material applied to the automatic measuring system, when the application of this reference material is abruptly terminated to put the automatic measuring system in the basic state

NOTE For instruments where transient oscillations occur in the approach to the final output signal, the 10 % of the initial output signal is considered as reached when the oscillations in the vicinity of the final output signal fall to less than 10 % of the initial output signal.

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[ISO 9169:2006, 2.2.1]

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**3.21**

**linearity**

⟨air quality⟩ maximum deviation between a linear calibration curve and the true value of the measurand, evaluated in practice as the maximum lack of fit within the measuring range

[ISO 9169:2006, 2.1.20]

**3.22**

**lack of fit**

systematic deviation, within the range of application, between the accepted value of a reference material applied to the measuring system and the corresponding result of measurement produced by the measuring system

[ISO 9169:2006, 2.2.9]

**3.23**

**residence time**

⟨stationary source emissions⟩ time period for the sampled gas to be transported from the inlet of the probe to the inlet of the measurement cell

**3.24**

**period of unattended operation**

maximum interval of time for which the performance characteristics remain within a predefined range without external servicing, e.g. refill, adjustment

[ISO 9169:2006, 2.2.11]

NOTE The period of unattended operation is often called maintenance interval.

**3.25****uncertainty (of measurement)**

measurement uncertainty

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

[ISO/IEC Guide 98-3:2008<sup>[4]</sup>, 2.2.3]**3.26****standard uncertainty**

uncertainty of the result of a measurement expressed as a standard deviation

[ISO/IEC Guide 98-3:2008<sup>[4]</sup>, 2.3.1]**3.27****expanded uncertainty**

quantity defining an interval about the result of a measurement that may be expected to encompass a large fraction of the distribution of values that could reasonably be attributed to the measurand

NOTE 1 The fraction may be viewed as the coverage probability or level of confidence of the interval.

NOTE 2 To associate a specific level of confidence with the interval defined by the expanded uncertainty requires explicit or implicit assumptions regarding the probability distribution characterized by the measurement result and its combined standard uncertainty. The level of confidence that may be attributed to this interval can be known only to the extent to which such assumptions may be justified.

NOTE 3 Expanded uncertainty is termed overall uncertainty in paragraph 5 of Recommendation INC-1 (1980).

[ISO/IEC Guide 98-3:2008<sup>[4]</sup>, 2.3.5 and 0.7 for a translation of Recommendation INC-1 (1980)]

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AMS	automatic measuring system
$e_i$	residual (lack of fit) at level $i$
FID	flame ionisation detection; flame ionisation detector
$i$	series element number
$M_{\text{CH}_4}$	molecular mass of methane (16 g/mol)
$M_{\text{H}_2\text{O}}$	molecular mass of water (18 g/mol)
$m_{\text{H}_2\text{O}, \text{v}}$	mass of water vapour
$n$	number of measurements
$n_{\text{neg}}$	number of interfering substances with a negative effect on the measured signal
$n_{\text{pos}}$	number of interfering substances with a positive effect on the measured signal
QA/QC	quality assurance and quality control
$s_r$	repeatability standard deviation
$S_{\text{pos}}$	sum of the positive interfering effects
$S_{\text{neg}}$	sum of the negative interfering effects

$V_0$	volume of the dry gas sampled
$V_m$	standard molar volume (22,4 l/mol)
$\bar{x}$	average of the measured values $x_i$
$x_i$	$i$ th measured value
$\bar{x}_i$	average of the measured values at level $i$
$\hat{x}_i$	value estimated by the regression line at level $i$
$x_{i, \text{neg}}$	$i$ th negative deviation in units of the measurand (e.g. mass concentration) caused by an interfering substance with a negative effect on the measured signal
$x_{i, \text{pos}}$	$i$ th positive deviation in units of the measurand (e.g. mass concentration) caused by an interfering substance with a positive effect on the measured signal
$\gamma_{\text{CH}_4, \text{s}}$	methane mass concentration at standard conditions of temperature and pressure (wet gas)
$\gamma_{\text{CH}_4, (\text{H}_2\text{O})_0}$	methane mass concentration at reference conditions of water vapour (dry gas)
$\gamma_{\text{CH}_4, \text{O}_2}$	methane mass concentration at reference conditions of oxygen
$\gamma_i$	test gas concentration at level $i$
$\rho_{\text{H}_2\text{O}, \text{v}}$	density of water vapour
$\varphi_{\text{CH}_4, \text{o}}$	methane content, as a volume fraction, at operating conditions
$\varphi_{\text{H}_2\text{O}, \text{m}}$	measured water vapour content, as a volume fraction, in the waste gas
$\varphi_{\text{O}_2, \text{m}}$	measured oxygen content, as a volume fraction, in the waste gas
$\varphi_{\text{O}_2, \text{ref}}$	reference oxygen content, as a volume fraction

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## 5 Apparatus and principles of operation

### 5.1 Measurement method

**5.1.1 Analyser.** The extractive analytical system consists of two elements: the flame ionisation detector (FID) and the associated sampling system.

Measurement by FID is based on the ionisation of organically bound carbon atoms in a hydrogen flame. The ionisation current measured by the FID depends on the number of carbon-hydrogen bonds in the organic compounds broken during combustion in the fuel gas flame, the nature of bonding (straight chain or branched chain) and whether other bound elements are present.

The main advantage of the FID is that it responds strongly to organic compounds and less to inorganic waste gas components, such as CO, CO<sub>2</sub>, NO, and H<sub>2</sub>O.

To determine methane alone, the FID is equipped with a catalytic converter, which oxidises all organic compounds in the sample gas except methane. Care shall be taken to avoid poisoning or contamination of the converter by sulfur-, nitrogen-, and chlorine-containing compounds. To avoid memory effects (signal offsets caused by contamination of the tube system within the instrument) and response delays in the system, the catalytic converter should be located close to the FID and heated.