



# SLOVENSKI STANDARD

## SIST EN 14791:2005

01-december-2005

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Stationary source emissions - Determination of mass concentration of sulphur dioxide -  
Reference method

Emissionen aus stationären Quellen - Bestimmung der Massenkonzentration von  
Schwefeldioxid - Referenzverfahren

Emissions de sources fixes - Détermination de la concentration massique du dioxyde de  
soufre - Méthode de référence

Ta slovenski standard je istoveten z: EN 14791:2005

### ICS:

13.040.40      Ö{ ã ã Á ^] ! ^{ ã } ã Ö ã [ ç      Stationary source emissions

SIST EN 14791:2005

en,fr,de

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EUROPEAN STANDARD  
NORME EUROPÉENNE  
EUROPÄISCHE NORM

**EN 14791**

November 2005

ICS 13.040.40

English Version

**Stationary source emissions - Determination of mass  
concentration of sulphur dioxide - Reference method**

Emissions de sources fixes - Détermination de la  
concentration massique du dioxyde de soufre - Méthode de  
référence

Emissionen aus stationären Quellen - Bestimmung der  
Massenkonzentration von Schwefeldioxid -  
Referenzverfahren

This European Standard was approved by CEN on 30 September 2005.

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 Central Secretariat 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 Central Secretariat has the same status as the official versions.

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## Foreword

This European Standard (EN 14791:2005) 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of an identical text or by endorsement, at the latest by May 2006, and conflicting national standards shall be withdrawn at the latest by May 2006.

This European Standard has been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association, and supports essential requirements of EU Directive(s).

For relationship with EU Directive(s), see informative Annex ZA, which is an integral part of this European Standard.

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

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## EN 14791:2005 (E)

## 1 Scope

This European Standard describes a manual method for sampling and determining SO<sub>2</sub> content in ducts and stacks emitting to the atmosphere by two analytical methods: Ion chromatography and Thorin method.

This European Standard is the Standard Reference Method (SRM) for periodic monitoring and for calibration or control of Automatic Measuring Systems (AMS) permanently installed on a stack, for regulatory purposes or other purposes. To be used as the SRM, the user shall demonstrate that the performance characteristics of the method are better than the performance criteria defined in this European Standard and that the overall uncertainty of the method is less than  $\pm 20,0$  % relative at the daily Emission Limit Value (ELV).

An Alternative Method to this SRM may be used provided that the user can demonstrate equivalence according to the Technical Specification CEN TS 14793, to the satisfaction of his national accreditation body or law.

This Standard Reference Method has been evaluated during field tests on waste incineration, co-incineration and large combustion installations. It has been validated for sampling periods of 30 min in the range of (0,5 to 2 000) mg/m<sup>3</sup> SO<sub>2</sub> for Ion Chromatography variant and 5 mg/m<sup>3</sup> to 2 000 mg/m<sup>3</sup> SO<sub>2</sub> for Thorin method according to emission limit values laid down in the following Council Directives:

- Council Directive 2001/80/EC on the limitation of emissions of certain pollutants into the air from large combustion plants;
- Council Directive 2000/76/EC on waste incineration plants.

The limit values of EU Directives are expressed in mg SO<sub>2</sub>/m<sup>3</sup>, on dry basis and at the reference conditions of 273 K and 101,3 kPa.

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## 2 Normative references

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

ENV 13005, *Guide to the expression of uncertainty in measurement*.

EN 13284-1, *Stationary source emissions — Determination of low range mass concentration of dust — Part 1: Manual gravimetric method*.

CEN/TS 14793, *Stationary source emission — Intralaboratory validation procedure for an alternative method compared to a reference method*.

## 3 Terms, definitions, symbols and abbreviations

### 3.1 Terms and definitions

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

#### 3.1.1

##### **absorber**

device in which sulphur oxide is absorbed into an absorption liquid



**3.1.2****absorption efficiency ( $\varepsilon$ )**

ratio  $\varepsilon$  of quantity of the analyte  $q_1$  collected in the first absorber divided by the quantity of the analyte collected in the first and the second absorber ( $q_1 + q_2$ )

$$\varepsilon = q_1 / (q_1 + q_2)$$

**3.1.3****analytical detection limit ( $L_D$ )**

concentration value of the measurand below which there is at least 95 % level of confidence that the measured value corresponds to a sample free of that measurand

**3.1.4****automatic measuring system (AMS)**

measuring system permanently installed on site for continuous monitoring of emissions

NOTE 1 An AM is a method which is traceable to a reference method.

NOTE 2 Apart from the analyser, an AMS includes facilities for taking samples (e.g. probe, sample gas lines, flow meters, regulators, delivery pumps) and for sample conditioning (e.g. dust filter, moisture removal devices, converters, diluters). This definition also includes testing and adjusting devices, that are required for regular functional checks.

**3.1.5****calibration of an AMS**

statistical relationship between values of the measurand indicated by the measuring system (AMS) and the corresponding values given by the standard reference method (SRM) used during the same period of time and giving a representative measurement on the same sampling plane

NOTE The result of calibration permits to establish the relationship between the values of the SRM and the AMS (calibration function).

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**3.1.6****calibration of the SRM**

set of operations that establish, under specified conditions, the relationship between values of quantities indicated by a measuring instrument or measuring system, and the corresponding values realized by standard reference methods implemented in the state of the art in order to provide representative results

[VIM]

**3.1.7****chemical blank value**

sulphate ion content of an unexposed sample of the absorption solution, plus reagents that are added to the solution before analysis if necessary

**3.1.8****emission limit value (ELV)**

emission limit value according to EU Directives on the basis of 30 min, 1 h or 1 day

**3.1.9****field blank**

value determined by a specific procedure used to ensure that no significant contamination has occurred during all steps of the measurement and to check that the operator can achieve a quantification level adapted to the task

**EN 14791:2005 (E)****3.1.10****influence quantity**

quantity that is not the measurand but that affects the result of the measurement

[adapted VIM 2.7]

Examples

- ambient temperature;
- atmospheric pressure;
- presence of interfering gases in the flue gas matrix.

**3.1.11****measurand**

particular quantity subject to measurement

[VIM 2.6]

**3.1.12****measurement series**

several successive measurements carried out on the same sampling plane and at the same process operating conditions

[EN 13284-1]

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**3.1.13****measuring system**

complete set of measuring instruments and other equipment assembled to carry out specified measurements

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[VIM 4.5]

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**3.1.14****performance characteristic**

one of the quantities (described by values, tolerances, range, ...) assigned to equipment in order to define its performance

**3.1.15****repeatability in the laboratory**

closeness of the agreement between the results of successive measurements of the same measurand carried out under the same conditions of measurement

NOTE 1 Repeatability conditions include:

- same measurement procedure;
- same laboratory;
- same sampling equipment, used under the same conditions;
- same location;
- repetition over a short period of time.

NOTE 2 Repeatability may be expressed quantitatively in terms of the dispersion characteristics of the results.

In this European Standard the repeatability is expressed as a value with a level of confidence of 95 %.

[VIM 3.6]

**3.1.16****repeatability in the field**

closeness of the agreement between the results of simultaneous measurements of the same measurand carried out with two equipments under the same conditions of measurement

NOTE 1 These conditions include:

- same measurement procedure; [SIST EN 14791:2005](https://standards.iteh.ai/catalog/standards/sist/46577db9-6254-476b-b0c0-4f501910e005/sist-en-14791-2005)
- two equipments, the performances of which are fulfilling the requirements of the reference method, used under the same conditions; <https://standards.iteh.ai/catalog/standards/sist/46577db9-6254-476b-b0c0-4f501910e005/sist-en-14791-2005>
- same location;
- implemented by the same laboratory;
- typically calculated on short periods of time in order to avoid the effect of changes of influence parameters (e.g. 30 min).

NOTE 2 Repeatability may be expressed quantitatively in terms of the dispersion characteristics of the results.

In this European Standard the repeatability under field conditions is expressed as a value with a level of confidence of 95 %.

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## 3.1.17

**reproducibility in the field**

closeness of the agreement between the results of simultaneous measurements of the same measurand carried out with several sets of equipment under the same conditions of measurement

NOTE 1 These conditions are called field reproducibility conditions and include:

- same measurement procedure;
- several sets of equipment, the performance of which fulfils the requirements of the reference method, used under the same conditions;
- same location;
- implemented by several laboratories.

NOTE 2 Reproducibility may be expressed quantitatively in terms of the dispersion characteristics of the results.

In this European Standard the reproducibility under field conditions is expressed as a value with a level of confidence of 95 %.

## 3.1.18

**sampling location**

specific area close to the sampling plane where the measurement devices are set up

## 3.1.19

**sampling plane**

plane normal to the centreline of the duct at the sampling position

[EN 13284-1]

## 3.1.20

**sampling point**

specific position on a sampling plane at which a sample is extracted

[EN 13284-1]

## 3.1.21

**standard reference method (SRM)**

measurement method recognised by experts and taken as a reference by convention, which gives, or is presumed to give, the accepted reference value of the concentration of the measurand (3.1.11) to be measured

## 3.1.22

**uncertainty**

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

## 3.1.22.1

**standard uncertainty  $u$** 

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

## 3.1.22.2

**combined uncertainty  $u_c$** 

standard uncertainty  $u_c$  attached to the measurement result calculated by combination of several standard uncertainties according to GUM

**3.1.22.3****expanded uncertainty  $U$** 

quantity defining a level of confidence about the result of a measurement that may be expected to encompass a specific fraction of the distribution of values that could reasonably be attributed to a measurand

$$U = k \times u$$

NOTE In this European Standard, the expanded uncertainty is calculated with a coverage factor of  $k = 2$ , and with a level of confidence of 95 %.

**3.1.22.4****overall uncertainty  $U_c$** 

expanded combined standard uncertainty attached to the measurement result calculated according to GUM

$$U_c = k \times u_c$$

**3.1.23****uncertainty budget**

calculation table combining all the sources of uncertainty according to EN ISO 14956 or ENV 13005 in order to calculate the overall uncertainty of the method at a specified value

**3.2 Symbols**

$C_m$	mass concentration of sulphur dioxide in the sample gas, in milligrams per cubic metre (of gas)
$Cl_r$	repeatability confidence interval, in milligrams per cubic metre
$Cl_R$	reproducibility confidence interval, in milligrams per cubic metre
$f_a$	equivalent mass of sulphur dioxide of 1 mm of titration solution (barium perchlorate standard volumetric solution) used for titration in Thorin method, in milligrams per millilitre
$f_v$	ratio of the volume of the pre-treated sample solution (sample absorption solution pre-treated before analyse) to the volume of the aliquot taken for the titration in Thorin method
$L_D$	limit of detection, in milligrams per litre of $\text{SO}_4^{2-}$
$L_Q$	limit of quantification, in milligrams per litre of $\text{SO}_4^{2-}$
$m_s$	weight of the sample solution (absorption solution used for sampling + rinsing solution), in grams
$P_m$	absolute pressure at the gas volume meter, in kilopascals
$P_{std}$	standard pressure: 101,3 kPa
$P_{sat}(T_m)$	saturation vapour pressure of water at volume gas meter temperature, in kilopascals
$q_s$	mass concentration of sulphate in sample absorption solution, in milligrams per litre (of solution)
$q_{cb}$	mass concentration of sulphate in chemical blank solution, in milligrams per litre (of solution)
$r$	repeatability, in milligrams per cubic metre or percentage
$R$	reproducibility, in milligrams per cubic metre or percentage

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$R_s$	peak resolution $S_s$ : volume of titration solution used for titration of sample absorption solution, in millilitre
$S_{cb}$	volume of titration solution used for titration of chemical blank solution, in millilitre
$S_r$	repeatability standard deviation, in milligrams per cubic metre or percentage
$S_R$	reproducibility standard deviation, in milligrams per cubic metre or percentage
$S_{r\ limit}$	maximum allowable repeatability standard deviation, in milligrams per cubic metre
$S_s$	volume of titration solution used for the titration of the aliquot of the pre-treated sample solution, in millilitres
$t_1$	retention time of the first peak, in seconds
$t_2$	retention time of the second peak, in seconds
$T_j$	temperature at the gas meter, in Kelvin
$T_m$	mean temperature at the gas volume meter in Kelvin
$T_{std}$	standard temperature, 273 K
$V_1$	reading at the gas volume meter at the beginning of the sampling period in cubic metres
$V_2$	reading at the gas volume meter at the end of the sampling period in cubic metres
$V_{m\ (std)}$	dry gas volume measured, corrected to standard conditions, in cubic metres
$V_s$	volume of the sample solution (absorption solution used for sampling + rinsing solution), in litres
$w_1$	peak width, on the time axis, of the first peak, in seconds
$w_2$	peak width on the time axis, of the second peak, in seconds
$\varepsilon$	absorption efficiency in percentage
$\sigma$	conductivity in micro-siemens per metre
$\rho_{20}^4$	density of a liquid at 20 °C compared to water's at 4 °C in kilogrammes per litre
$\chi$	volume content in percentage

**3.3 Abbreviations**

PE	Polyethylene
PTFE	Polytetrafluoroethene

## 4 Principle

### 4.1 General

This European Standard describes the Standard Reference Method (SRM) with two alternative analytical techniques for determining sulphur dioxide (SO<sub>2</sub>) content emitting to atmosphere from ducts and stacks. The specific components and the requirements for the measuring system are described. A number of performance characteristics with associated minimum performance criteria are specified for the measuring system (see Tables 2 and 3 in 10.2). These performance characteristics and the overall uncertainty of the method shall meet the specifications given in this European Standard.

### 4.2 Measuring principle

A representative sample of gas is extracted via a heated temperature-controlled probe. The sample is filtered and drawn through hydrogen peroxide absorber solutions for a specified time and at a controlled flow rate. The sulphur dioxide in the sampled gas is absorbed and oxidised to sulphate ion. The mass concentration of sulphate in the absorption solutions is subsequently determined using ion chromatography or by titration with a barium perchlorate solution using Thorin as indicator. SO<sub>3</sub> is also absorbed and transformed in sulphate ion and is therefore an interferent.

This European Standard has been validated with a 0,3 % H<sub>2</sub>O<sub>2</sub> absorption solution up to 1 000 mg/m<sup>3</sup> and with a 3,0 % H<sub>2</sub>O<sub>2</sub> absorption solution up to 2 000 mg/m<sup>3</sup>. Typical concentration of the absorption solution is 0,3 % H<sub>2</sub>O<sub>2</sub>. However, for concentrations higher than 1 000 mg/m<sup>3</sup> it is recommended, in case of a bad efficiency either to decrease the flow or to increase the concentration.

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## 5 Reagents

### 5.1 General

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During the analysis, use only reagents of recognised analytical grade.

Normal, accepted laboratory safety practices and cleaning procedures for glassware should be followed during reagent preparation.

**WARNING — Use the reagents in accordance with the appropriate health and safety regulations.**

### 5.2 Hydrogen peroxide

Commercially available solution of H<sub>2</sub>O<sub>2</sub>; mass content 30 %,  $\rho_{20}^4 = 1,11 \text{ kg/l}$ .

### 5.3 Water

H<sub>2</sub>O; ultra pure water with conductivity  $\sigma < 10 \mu\text{S m}^{-1}$ .

### 5.4 Absorption solution, H<sub>2</sub>O<sub>2</sub>

The absorption solution is a hydrogen peroxide solution (5.2) diluted to a mass concentration of 0,3 % H<sub>2</sub>O<sub>2</sub> in water (5.3). For concentrations higher than 1 000 mg/m<sup>3</sup> it is suggested in case of a bad efficiency, either to decrease the flow, or increase the concentration of the absorption solution.