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Emisije nepremičnih virov - Vzorčenje in določevanje selenovih spojin v odpadnih plinih

Stationary source emissions - Sampling and determination of selenium compounds in flue gas

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Émission des sources fixes - Échantillonnage et détermination des composés de sélénium dans les effluents gazeux

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**Stationary source emissions —
Sampling and determination of
selenium compounds in flue gas**

*Émission des sources fixes — Échantillonnage et détermination des
composés de sélénium dans les effluents gazeux*

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ISO copyright office
Ch. de Blandonnet 8 • CP 401
CH-1214 Vernier, Geneva, Switzerland
Tel. +41 22 749 01 11
Fax +41 22 749 09 47
copyright@iso.org
www.iso.org

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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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: [Foreword - Supplementary information](#)

The committee responsible for this document is ISO/TC 146, *Air quality*, Subcommittee SC 1, *Stationary source emissions*.

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ISO 17211:2015(E)**Introduction**

Since it is estimated that selenium is exhausted from stationary sources like coal combustion plants, the investigation of the emission amounts of selenium from the stationary source is increasingly important for preventing a potential risk.

This International Standard describes a method for the sampling and determination of selenium compounds in a flue gas passing through ducts or chimneys. Selenium compounds generally exist both in vapour phase and in solid phase in flue gases, this method allows the determination of both gaseous and particulate selenium concentrations in flue gases.

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Stationary source emissions — Sampling and determination of selenium compounds in flue gas

1 Scope

This International Standard describes the method for the sampling and determination of selenium compounds in both vapour phase and solid phase that are entrained in flue gases carried in stacks or ducts. The selenium content in flue gas is expressed as a mass concentration of elemental selenium in the stack gas.

Particulate and gaseous selenium compounds are captured by a filter and an absorber solution, respectively. The total concentration of selenium compounds in flue gas is expressed as the sum of both concentrations.

The concentrations of selenium in both samples are determined using inductively coupled plasma optical emission spectrometry (ICP-OES), inductively coupled plasma mass spectrometry (ICP-MS) or graphite furnace atomic absorption spectrometry (GFAAS). Hydride generation (HG) techniques coupled to atomic spectrometry can also be used such as HG-AAS, HG-AFS (atomic fluorescence spectrometry), HG-ICP-OES and HG-ICP-MS.

The detection limit for gaseous selenium compounds is $0,3 \mu\text{g}/\text{m}^3$ using HG-ICP-MS at a sampling volume of $0,12 \text{ m}^3$. The detection limit for particulate selenium compounds is $0,001 2 \mu\text{g}/\text{m}^3$ using this technique at a sampling volume of $2,0 \text{ m}^3$.

2 Normative references

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

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

ISO 9096, *Stationary source emissions — Manual determination of mass concentration of particulate matter*

ISO 10396, *Stationary source emissions — Sampling for the automated determination of gas emission concentrations for permanently-installed monitoring systems*

ISO 11885, *Water quality — Determination of selected elements by inductively coupled plasma optical emission spectrometry (ICP-OES)*

ISO 12141, *Stationary source emissions — Determination of mass concentration of particulate matter (dust) at low concentrations — Manual gravimetric method*

ISO 15586, *Water quality — Determination of trace elements using atomic absorption spectrometry with graphite furnace*

ISO 16911-1, *Stationary source emissions — Manual and automatic determination of velocity and volume flow rate in ducts — Part 1: Manual reference method*

ISO 17294-1, *Water quality — Application of inductively coupled plasma mass spectrometry (ICP-MS) — Part 1: General guidelines*

ISO 17294-2, *Water quality — Application of inductively coupled plasma mass spectrometry (ICP-MS) — Part 2: Determination of 62 elements*

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ISO/TS 17379-1, *Water quality — Determination of selenium — Part 1: Method using hydride generation atomic fluorescence spectrometry (HG-AFS)*

ISO/TS 17379-2, *Water quality — Determination of selenium — Part 2: Method using hydride generation atomic absorption spectrometry (HG-AAS)*

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

ISO 23210:2009, *Stationary source emissions — Determination of PM₁₀/PM_{2,5} mass concentration in flue gas — Measurement at low concentrations by use of impactors*

ISO/IEC Guide 98-3, *Uncertainty of measurement — Part 3: Guide to the expression of uncertainty in measurement (GUM:1995)*

3 Terms and definitions

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

3.1 gaseous selenium compounds
selenium compounds passing through a filter having at least 99,5 % collection efficiency for 0,3 µm diameter particles

3.2 isokinetic sampling
sampling at a flow rate such that the velocity and direction of the gas entering the sampling nozzle are the same as those of the gas in the duct at the sampling point

3.3 particulate selenium compounds
selenium compounds contained in a solid phase particle collected by a filter having at least 99,5 % collection efficiency for 0,3 µm diameter particles

3.4 sampling point
specific position on the sampling section at which a sample is extracted

3.5 STP
standard conditions for temperature, 273,15 K, and pressure, 101,325 kPa

4 Symbols and abbreviated terms

4.1 Symbols

$C_{A1,Se}$	concentration of selenium in prepared sample of the first and second absorber solutions (µg/ml)
$C_{A2,Se}$	concentration of selenium in prepared sample of the third absorber solution (µg/ml)
$C_{R,Se}$	concentration of selenium in prepared sample of rinse solution that washed transfer line from the filter housing to the first impinger nozzle in main-stream sampling (µg/ml)
$C_{R1,Se}$	concentration of selenium in prepared sample of rinse solution that washed transfer line from the filter housing to the T-piece in side-stream sampling (µg/ml)
$C_{R2,Se}$	concentration of selenium in prepared sample of rinse solution that washed transfer line after the T-piece to the first impinger nozzle in side-stream sampling (µg/ml)

$C_{S,Se}$	concentration of selenium in prepared sample solution for particulate selenium analysis ($\mu\text{g/ml}$)
d	density of reagent solution (g/ml)
p_{atm}	atmospheric pressure (kPa)
p_{av}	average pressure difference between the sample gas before the gas meter and the atmosphere (kPa)
$q_{m,Se}$	rate of mass discharge of selenium expressed as elemental selenium (mg/s)
$q_{V,fg,i}$	volume flow rate of flue gas through the sampling plane at conditions i of temperature, pressure, moisture and oxygen content (m^3/s)
T_{av}	average temperature of the sample gas before the gas meter (K)
$u(y)$	standard uncertainty ($\mu\text{g}/\text{m}^3$)
V_{d}	volume of dry flue gas sample normalized to STP (m^3)
V_{f}	final gas meter reading at the end of sampling (m^3)
$V_{G,d}$	volume of dry flue gas sample for gaseous selenium analysis normalized to STP (m^3)
V_{i}	initial gas meter reading at the beginning of sampling (m^3)
V_{l}	volume of air drawn through the gas meter during any intermediate leak tests (m^3)
V_{m}	volume of dry flue gas sample (m^3)
$V_{\text{main,d}}$	volume of dry flue gas sample in main stream, normalized to STP, in side-stream sampling (m^3)
$V_{S,d}$	volume of dry flue gas sample for particulate selenium analysis normalized to STP (m^3)
$V_{\text{side,d}}$	volume of dry flue gas sampled in side stream, normalized to STP, in side-stream sampling (m^3)
V_{A1}	volume of recovered sample of the first and second absorber solutions (ml)
V_{A2}	volume of recovered sample of the third absorber solution (ml)
V_{R}	volume of recovered sample of rinse solution that washed transfer line from the filter housing to the first impinger nozzle in main-stream sampling (ml)
V_{R1}	volume of recovered sample of rinse solution that washed transfer line from the filter housing to the T-piece in side-stream sampling (ml)
V_{R2}	volume of recovered sample of rinse solution that washed transfer line after the T-piece to the first impinger nozzle in side-stream sampling (ml)
V_{S}	volume of prepared sample solution for particulate selenium analysis (ml)
w_{W}	average moisture content of the flue gas at the sampling plane during the sampling period (%)
$y_{1,j}$	j th concentration value of the first measuring system ($\mu\text{g}/\text{m}^3$)
$y_{2,j}$	j th concentration value of the second measuring system ($\mu\text{g}/\text{m}^3$)

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$\rho_{G,Se,dry}$	mass concentration of gaseous selenium expressed as elemental selenium in the flue gas on a dry basis at STP ($\mu\text{g}/\text{m}^3$)
$\rho_{S,Se,dry}$	mass concentration of particulate selenium expressed as elemental selenium in the flue gas on a dry basis at STP ($\mu\text{g}/\text{m}^3$)
$\rho_{Se,dry}$	mass concentration of total selenium expressed as elemental selenium in the flue gas on a dry basis at STP ($\mu\text{g}/\text{m}^3$)
$\rho_{Se,dry,0}$	mass concentration of selenium expressed as elemental selenium in the flue gas on a dry basis at STP and reference oxygen concentration ($\mu\text{g}/\text{m}^3$)
$\rho_{Se,i}$	mass concentration of selenium expressed as elemental selenium at conditions <i>i</i> of temperature, pressure, oxygen and moisture conditions ($\mu\text{g}/\text{m}^3$)
$\rho_{Se,wet}$	mass concentration of selenium expressed as elemental selenium in the flue gas on a wet basis at STP ($\mu\text{g}/\text{m}^3$)
$\rho_{Se,wet,0}$	mass concentration of selenium expressed as elemental selenium in the flue gas on a wet basis at STP and reference oxygen concentration ($\mu\text{g}/\text{m}^3$)
$\varphi_{O,d}$	volume fraction of the oxygen on a dry basis measured during the sampling (%)
$\varphi_{O,ref}$	volume fraction of the reference oxygen for the process (%)

4.2 Abbreviated terms

AAS	atomic absorption spectrometry
AFS	atomic fluorescence spectrometry
GFAAS	graphite furnace atomic absorption spectrometry
HG	hydride generation
ICP-MS	inductively coupled plasma mass spectrometry
ICP-OES	inductively coupled plasma optical emission spectrometry
FEP	hexafluoroethene propene, perfluoro(ethane-propene)
PFA	perfluoroalkoxy
PTFE	polytetrafluoroethylene

5 Principle

Selenium compounds generally exist both in vapour phase and in solid phase in a flue gas. Particulate and gaseous selenium compounds are captured by a filter and an absorber solution, respectively. The concentration of selenium in a flue gas is expressed as the sum of both concentrations.

To determine particulate selenium contents in a flue gas, a stack sample gas is taken isokinetically and particles are collected on a filter in accordance with ISO 9096 or ISO 12141.

To determine gaseous selenium content in a flue gas, a stack sample gas is taken through a filter. Gaseous selenium compounds that pass through the filter are collected in an absorber solution. Since some of gaseous selenium compounds, mostly SeO_2 , are adsorbed and in some cases reduced to elemental selenium on the inner surface of a sampling system in the presence of steam and SO_2 , the sampling