SLOVENSKI STANDARD
oSIST ISO 20264:2021

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Emisije nepremičnih virov - Določevanje masne koncentracije posameznih hlapnih organskih spojin (VOC) v odpadnih plinih nezgorevalnih procesov

Stationary source emissions - Determination of the mass concentration of individual volatile organic compounds (VOCs) in waste gases from non-combustion processes

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Émissions de sources fixes - Détermination de la concentration en masse de composés organiques volatils (COV) individuels dans les gaz résiduaires issus de processus sans combustion

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Foreword

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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 of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see www.iso.org/iso/foreword.html.

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

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

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Introduction

There are various volatile organic compounds (VOCs) emitted from stationary sources where organic solvents are used for painting, printing, cleaning, degreasing and chemicals production. In order to understand how to reduce the environmental risk due to VOCs, it is necessary to measure not only the concentration of total VOCs but also the concentration of individual VOCs in waste gases. This is because individual VOCs have different potentials to form O_3 and suspended particulate matter (SPM). Also, there are VOCs of high toxicity (e.g. benzene, toluene, propyl acetate, propanol, formaldehyde, some chlorinated organic compounds) of concern.

Fourier Transform Infrared (FTIR) spectrometry is proposed to provide these measurements as it provides a measurement in the infrared (IR) region over a wide spectral band. Analysis of the recorded spectra enables the concentration of a wide number of compounds to be quantified simultaneously. Overlap of IR absorption features with each VOC can affect the quantification of each compound. However, by using appropriate chemometric procedures for the overlapping IR spectra of VOCs, the concentrations are quantified for the individual compounds of interest.

This document specifies the measurement method for determining concentrations of individual VOCs in waste gases from non-combustion processes by using FTIR spectroscopy.

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Stationary source emissions — Determination of the mass concentration of individual volatile organic compounds (VOCs) in waste gases from non-combustion processes

1 Scope

This document specifies the use of FTIR spectrometry for determining the concentrations of individual volatile organic compounds (VOCs) in waste gases from non-combustion processes. The method can be employed to continuously analyse sample gas which is extracted from ducts and other sources. A bag sampling method can also be applied, if the compounds do not adsorb on the bag material, and is appropriate in cases where it is difficult or impossible to obtain a direct extractive sample.

The principle, sampling procedure, IR spectral measurement and analysis, calibration, handling interference, QA/QC procedures and some essential performance criteria for measurement of individual VOCs are described in this document.

NOTE 1   The practical minimum detectable concentration of this method depends on the FTIR instrument (i.e. gas cell path length, resolution, instrumental noise and analytical algorithm) used, compounds, and interference specific (e.g. water and CO_2).

2 Normative references

There are no normative references in this document.

3 Terms and definitions

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

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

3.1 Terms related to FTIR

3.1.1
absorbance
negative logarithm of the transmission, $A = -\log(I/I_0)$, where I is the transmitted intensity of the light and I_0 is the incident intensity

3.1.2
resolution
minimum separation that two spectral features can have and still, in some manner, be distinguished from one another

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3.2 Terms related to performance characteristics

3.2.1
reference spectrum
plot of absorbance versus wavenumber for a known gas or known mixture of gases, which are obtained under controlled conditions of pressure and temperature, path length, and known concentration

Note 1 to entry: The reference spectra are used to prepare the chemometric model used to obtain the unknown concentrations of analytes in sample spectra.

Note 2 to entry: See 10.3.1.

3.2.2
validation spectrum
plot of absorbance versus wavenumber for *calibration verification gas* (3.2.6)

Note 1 to entry: See 10.3.4.

3.2.3
background spectrum
plot of absorbance versus wavenumber for *zero gas* (3.2.5)

3.2.4
response time
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

[SOURCE: ISO 9169:2006, 2.2.4]

3.2.5
zero gas
high purity nitrogen (99,999 %) or synthetic air (99,999 %) is used to measure a *background spectrum* (3.2.3) and to determine the limit of detection, as well as to purge sample lines and sampling system components, to dilute sample and *calibration verification gas* (3.2.6), and to conduct blank measurements

3.2.6
calibration verification gas
gas or gas mixture where the concentration(s) and uncertainty(ies) are known, used to check the high level concentration point of the measuring system

Note 1 to entry: The gas or gases used is included in the analytical algorithm used to quantify the concentration of target analyte, and have absorption lines distinguishable from baseline noise at wavenumbers that are within the upper and lower wavenumber limits where the target analyte displays absorption lines distinguishable from baseline noise. An absorption feature is considered distinguishable from baseline noise if it is greater than three times the standard deviation of the baseline noise.

Note 2 to entry: This concentration is often chosen around 70 % to 80 % of full scale.

3.2.7
lack of fit
systematic deviation within the range of application between the measurement results obtained by applying the calibration function to the observed response of the measuring system, measuring reference materials and the corresponding accepted value of such reference materials

Note 1 to entry: Lack of fit can be a function of the measurement result.

[SOURCE: ISO 9169:2006, 2.2.9]

3.2.8
analytical interference
situation that arises when two or more compounds have overlapping absorbance bands in their infrared spectra

3.2.9
limit of detection
LOD
minimum concentration of a compound that can be detected by an instrument with a given statistical probability

Note 1 to entry: Usually the detection limit is given as three times the standard deviation of noise in the system.

3.2.10
analytical algorithm
method used to quantify the concentration of the target analytes and interferences in each FTIR spectrum

Note 1 to entry: The analytical algorithm should be used to account for the *analytical interferences* (3.2.8) by conducting the analysis in a portion of the infrared spectrum that is the most unique for that particular compound.

3.2.11
chemometrics
chemical discipline that uses mathematical and statistical methods, (a) to design or select optimal measurement procedures and experiments, and (b) to provide maximum chemical information by analysing chemical data

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3.2.12
independent reading
reading that is not influenced by a previous individual reading as the two individual readings are separated by at least four response times

4 Symbols and abbreviated terms

I_0	intensity of incident radiation
I	intensity of transmitted radiation
A	absorbance
T	transmittance
α	absorptivity
l	optical path length
c	sample concentration
C_{CLS}	the known concentration from the reference spectra
C_{VAL}	the predicted concentrations from the validation spectra
FTIR	Fourier transform infrared
CLS	classical least squares

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PLS   partial least squares

ILS   inverse least squares

SEV   standard error of validation

5 Measurement principle

5.1 General

A sample gas is extracted from ducts and other sources via a sampling system and continually introduced into a gas cell of an FTIR system. The IR spectra of the sample gas is measured using an FTIR spectrometer. When a sampling bag is used, the gas sampled in the bag is transferred to the gas cell. IR spectra obtained are analysed by using analytical algorithm. Some VOCs might adsorb onto the sampling bag surface, reducing meaured VOC concentration. Losses by absorption shall be tested and documented before sampling.

5.2 FTIR Spectrometer components

Figure 1 illustrates the basic FTIR spectrometer configuration required for gas phase analyses. The IR radiation emitted by the IR source contains energy at all wavelengths between 2,5 and 14 µm, which is 700 to 4 000 cm^{-1} for most IR systems conducting these analyses. The IR radiation passes through an interferometer, where the motion of an optical element — usually a mirror — optically modulates the IR beam. The modulated IR beam then enters an absorption cell through a window and interacts with the gases of interest. In "multi-pass" (for example "White") absorption cells, mirrors within the cell direct the IR beam through the sample gas multiple times; in such cells, the absorption pathlength can be from 4 to 50 (or more) times the cell's physical length. (A larger absorption path length generally leads to greater sensitivity.) The IR beam then exits the sample cell via a second window and is re-focused onto an IR detector.
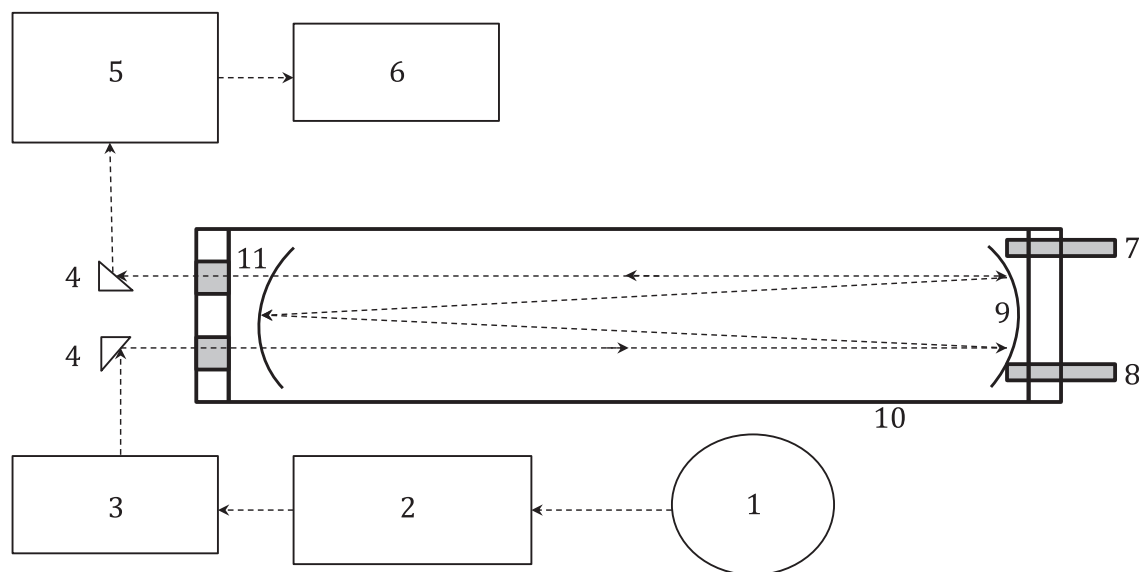
Key

1	IR source	7	absorption cell exhaust
2	aperture or filter 1 (optional)	8	absorption cell inlet
3	interferometer	9	mirror
4	focusing optics	10	absorption cell
5	aperture or filter 2 (optional)	11	infrared window
6	IR detector		

Figure 1 — FTIR spectrometer components and beam path

5.3 Interferogram

A beam of the broadband IR radiation is divided into two or more paths with different optical path lengths and is recombined to give a detector signal with repetitive interference maxima and minima with the aid of an interferometer. Figure 2 shows the Michelson interferometer as an example.

The interferogram is obtained by plotting the detector signal against the difference in optical path length. Given a difference in optical path lengths corresponding to an even multiple of the wavelength, the interference is constructive, and given an odd multiple, the interference is destructive. An additional laser with its own detector is contained in an FTIR system. The radiation emitted by the laser and the broad band IR source passes through the interferometer simultaneously, although the interferograms are recorded by separate detectors. From the positions of the peaks of the interferogram of the laser irradiation, it is possible to determine the difference in the optical path length, as the laser's input frequency is known and is constant (e.g. 632,8 nm for a HeNe laser).