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Road vehicles — Test contaminants for filter evaluation —

Part 3:
Soot contaminant

Véhicules routiers — Poussière pour l'essai des filtres —

Partie 3: Poussière de suie

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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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This document was prepared by Technical Committee ISO/TC 22, *Road Vehicles*, Subcommittee SC 34, *Propulsion, powertrain and powertrain fluids*.

A list of all parts in the ISO 12103 series can be found on the ISO website.

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.

Introduction

This document specifies a grade of test aerosol, which is composed of soot from a combustion source similar to soot occurring in the environment that motor vehicles are commonly subjected to. This test contaminant is developed for air filter media and element testing.

Ambient aerosols include at least two distinct modes of aerosol: a sub-micron mode and a super-micron mode. Generally the sub-micron mode comes from combustion sources or condensation of gases. The super-micron mode comes from physical abrasion processes and wind-blown dust. The test dusts described in ISO 12103-1 can be used to simulate the super-micron mode of ambient aerosol for testing air filters. The soot aerosol described in this document is intended to simulate the sub-micron mode of ambient aerosol.

There are several possible methods of generating soot aerosol, to simulate the sub-micron mode for air filter testing purposes such as dispersing soot from a powder or using generated soot from a combustion process.

Particle size of soot dispersed from bulk powder exceeds the environmental soot considerably.

For generated soot from a combustion process, a new procedure is described in this document. Using aliphatic hydrocarbons, the soot consists of a combination of carbon, organic hydrocarbons and other substances.

Road vehicles — Test contaminants for filter evaluation —

Part 3:
Soot contaminant

1 Scope

This document defines particle size distribution by number and chemical content limits involving one grade of test aerosol made from combustion soot.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 15900, *Determination of particle size distribution — Differential electrical mobility analysis for aerosol particles*

ISO 29904:2013, *Fire chemistry — Generation and measurement of aerosols*

NIOSH, *Elemental carbon (diesel particulate): Method 5040, Issue 3*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 15900, ISO 29904 and the following 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
diffusion flame
flame from a burner which gets its oxygen from the ambient surrounding air by diffusion and convection mechanisms instead of having the oxygen forcibly premixed into the fuel

3.2
generation
process in which airborne particles are produced and injected into a defined airstream

3.3
mobility particle size
particle size provided by an electro-static classifier

Note 1 to entry: The method is based on a principle that uses the forces exerted on charged particle in an electro-static field. The method is used for particles in the nm range and the classifier is typically combined with a condensation particle counter to actually determine the concentration.

3.4
optical particle size
particle size as recorded by an optical particle sizing instrument or spectrometer

Note 1 to entry: The optical size differs in general from the physical size of a particle as it depends on particle properties like the light diffraction index.

3.5
particle size distribution
number, mass or volume of particles as function of the particle size

Note 1 to entry: In this document the term is used for number distributions only. Particle size distributions may have a wide variety of shapes but for the purpose of this document and application the distributions of *soot* (3.7) particles can be assumed to be of a lognormal type.

3.6
particle number sizer
system consisting of a method to classify particles by electrical mobility and measures the number concentration of particulate at each size through means of one or more condensation particle counters and or electrometers

3.7
soot
particles from a combustion process consisting of carbon and being created during incomplete incineration of organic fuels

Note 1 to entry: The particles start with clusters of several hundred carbon atoms and can form large grains up to several hundred µm. Small soot particles have the tendency to agglomerate. Soot particle from combustions consists of elemental carbon (EC) and organic carbon (OC). A lot of organic compounds are known and most of them are bound on the EC-agglomerates. Some of the organic compounds are carcinogenic like poly aromatic hydrocarbon (PAH).

3.8
thermal-optical transmission
method to measure elemental carbon (EC) with its relationship to atmospheric *soot* (3.7)

Note 1 to entry: For the thermal-optical transmission method (TOT), an emphasis on optical behaviour presents it as a method for the accurate measurement of light-absorbing particulate carbon and thus allows EC to be defined as black carbon (BC) as in the aethelometer.

4 Symbols and abbreviated terms

Symbol	Explanation
μ	mean value of a Gaussian standard distribution
σ	standard deviation of a Gaussian standard distribution
σ_{geo}	geometric standard deviation (the logarithm of the GSD is the standard deviation)

Abbreviations	Explanation
SMPS	scanning mobility particle sizer
Sub-micron	particles <1 µm
Super-micron	particles >1 µm
EC	elemental carbon

OC	organic carbon
TOT	thermal-optical transmission
PAH	poly aromatic hydrocarbons
EEPS	engine exhaust particle sizer spectrometer

5 Test contaminant definition

Test aerosols according to this document are generated by combustion of an organic fuel in the test lab. They consist primarily of agglomerates of carbon particles where the primary carbon particles are on the order of 20 nm diameter. Some organic compounds may be condensed on the carbon agglomerates during the combustion and dilution processes. The amount of condensed organic material determines the category of the test aerosol per 5.1.

5.1 Test contaminant designation

Soot contaminants are produced by burning a fuel (gas or liquid) and are listed in the grade as follows:

— ISO 12103-3, S1 for ≥67 % carbon content.

This flame shall lead to an aerosol consisting of 70 % ± 3 % elemental carbon, 29 % ± 3 % organic carbon, and <1 % other substances. The air-to-fuel ratio of the flame shall be set as ±10 % stoichiometric balanced with adjustment of an inert mixing gas addition to the fuel flow to adjust the particle distribution. The stoichiometric balance is described in Annex F.

Soot contaminant shall be tested per the methods in Annex E and the above requirements.

5.2 Generation of soot aerosol

Test aerosols according to this document are generated by combustion of an organic fuel in the test lab. They consist primarily of agglomerates of carbon particles where the primary carbon particles are on the order of 20 nm diameter. Organic compounds may be condensed on the carbon agglomerates during the combustion and dilution processes.

5.3 Particle size distribution

The unimodal particle size distribution shall be determined using electrical mobility sizing method and shall meet the distribution and tolerances given in Annex A. The size distributions shall be presented as normalized number distribution.

5.4 Stability of aerosol concentration and particle size distribution

The soot aerosol mass concentration limits and tolerances are given in Annex A.

Care should be taken to ensure that the concentration is low enough to prevent size changes due to coagulation from occurring in the test system. Recommendation for maximum number concentration is 10^7/cm^3. More detailed information is provided in Annex C.

5.5 Chemical composition

For generating the soot a diffusion flame with gas (e.g. propane) can be used. But any fuel yielding the defined chemical composition and size distribution is accepted. More detailed information is provided in Annex D.

This flame shall lead to an aerosol consisting of the content breakdowns presented in 5.1.

To achieve accurate reproducible filter test results, a soot aerosol ideally consisting of solely elemental carbon (EC) would be preferable. Because soot from combustion always contains more or less organic carbon (OC), the content of OC in the test aerosol shall be demonstrated.

Samples of the aerosol shall be taken and analysed per NIOSH test method 5040 issue 3 for EC/OC.

The ratio of OC shall be demonstrated by thermal-optical transmission analysis by the thermal protocol NIOSH 700+. It shall be demonstrated only once for each soot generator or after any significant modification of the unit, e.g. after changing geometric dimensions of the burner, using another fuel gas or other flow settings, etc.

It shall be demonstrated for all fixed operating points which fit the described standardized soot aerosol in this document. For soot generators with continuously adjustable settings it shall be demonstrated by a suitable method that the OC content does not exceed 29 % within the adjustable range. This can be realized by analysis of probes at some single set-points at the minimum and at the maximum of the adjustable range at least and one or more between.

For more information about the thermal-optical transmission and about the sampling method refer to Annex E.

6 Analysis equipment and operating procedure

This clause will identify the analysis equipment and procedure to sample, classify, and identify the soot aerosol, which is being produced in this document. General handling information is provided in Annex B.

6.1 Analysis equipment

The analysis equipment specified in ISO 29904:2013, 5.6.5 and 5.6.6, specifically the SMPS and EEPS are examples of particle number sizer equipment which shall be used to characterize the soot contaminant. ISO 29904 has four clauses per piece of equipment; principle, method description, parameters produced, and the advantages and disadvantages of each piece of equipment.

6.2 Particle size analysis procedure

Analysis of ISO-specified soot test aerosol shall be performed using a differential mobility classifier with condensation particle counter detector or equivalent equipment at the outlet of the soot generator.

Mobility particle size shall cover the range from 10 nm up to 400 nm by optical particle size, with at least 8 channels per decade. Particle measurement devices which do not fulfil this requirement are not suitable, since small particles will not be detected.

Characterizing of the distribution, the location of the characterization shall occur at the introduction point of the test. The sampling method specified in ISO 29904:2013, 5.6, shall be used to identify the method to sample the soot aerosol.

6.3 Instrument calibration

Calibration of the differential electrical mobility analyser shall be calibrated as per ISO 15900.

Annex A
(normative)

Particle size distributions by number

A.1 General

For typical soot sources used within the scope of this document the particles usually are lognormal distributed with regards to their size. These distributions can easily be converted into a standard normal distribution. A normal distribution is distinctly defined by two parameters. Therefore, this document describes the range of acceptable particle size distributions by defining those two parameters together with a set of tolerances. This annex describes how this is done and how real particle size distributions can be checked for conformance with those targets.

A.2 Lognormal distribution and Gaussian normal distribution

A typical particle size distribution for soot is shown in Figure A.1 on a linear size axis together with a mathematically fitted lognormal curve. The general formula of that function is:

$$f(x)=\frac{a}{\sqrt{2\cdot\pi}\cdot\sigma\cdot x}\cdot e^{\frac{-(\ln x-\mu)^2}{2\cdot\sigma^2}} \qquad \text{(A.1)}$$

for x > 0

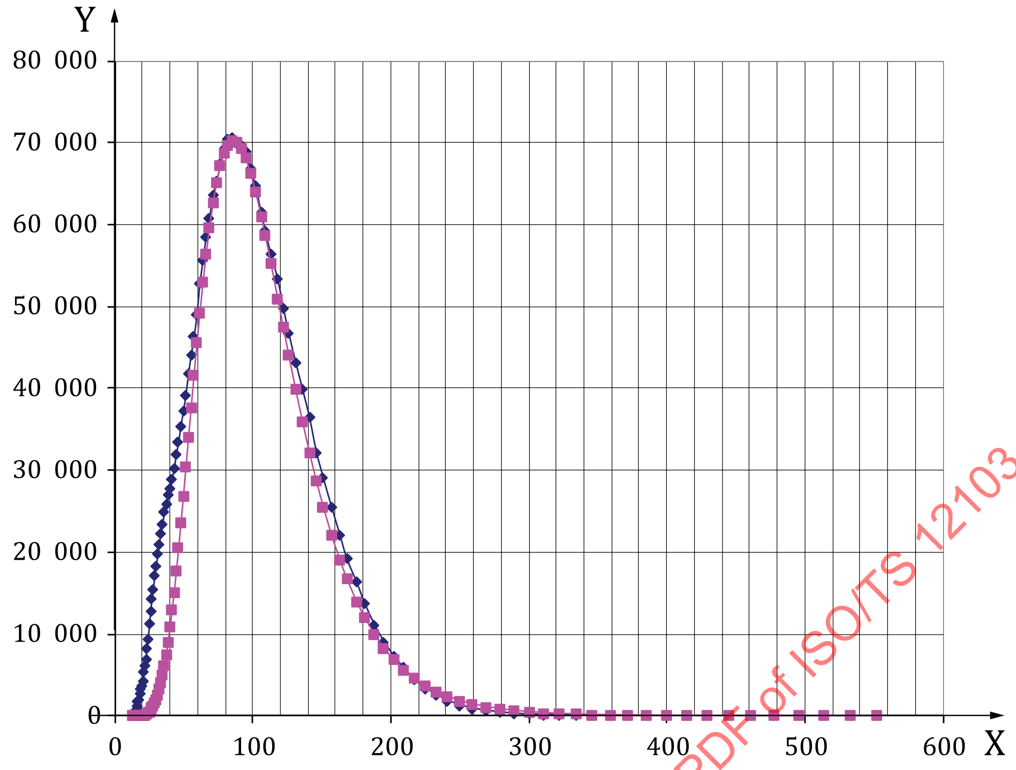
a is a scaling factor;

x is the particle diameter.

μ is the mean of distribution;

σ is the standard deviation.

$$\mu=\ln E-\frac{1}{2}\cdot\ln\left[1+\left(\frac{\sigma}{E}\right)^2\right] \qquad \text{(A.2)}$$



Key

X	mobility particle diameter (nm)
Y	particle counts
—◆—	example of a particle size distribution of real soot
—■—	computed lognormal-fit

Figure A.1 — A typical particle size distribution curve of a real soot aerosol from a diffusion flame and an engine

The same distribution becomes a Gaussian normal distribution if the particle counts are drawn versus the logarithm of their size channels [x becomes ln(x)]. In this form μ and σ represent the mean and the standard deviation of the distribution and therefore have a more obvious and vivid meaning than in Formula (A.1). This is shown in Figure A.1.

$$f(x)=\frac{a}{\sqrt{2\cdot\pi}\cdot\sigma}\cdot e^{\frac{-(x-\mu)^2}{2\cdot\sigma^2}} \qquad \text{(A.3)}$$

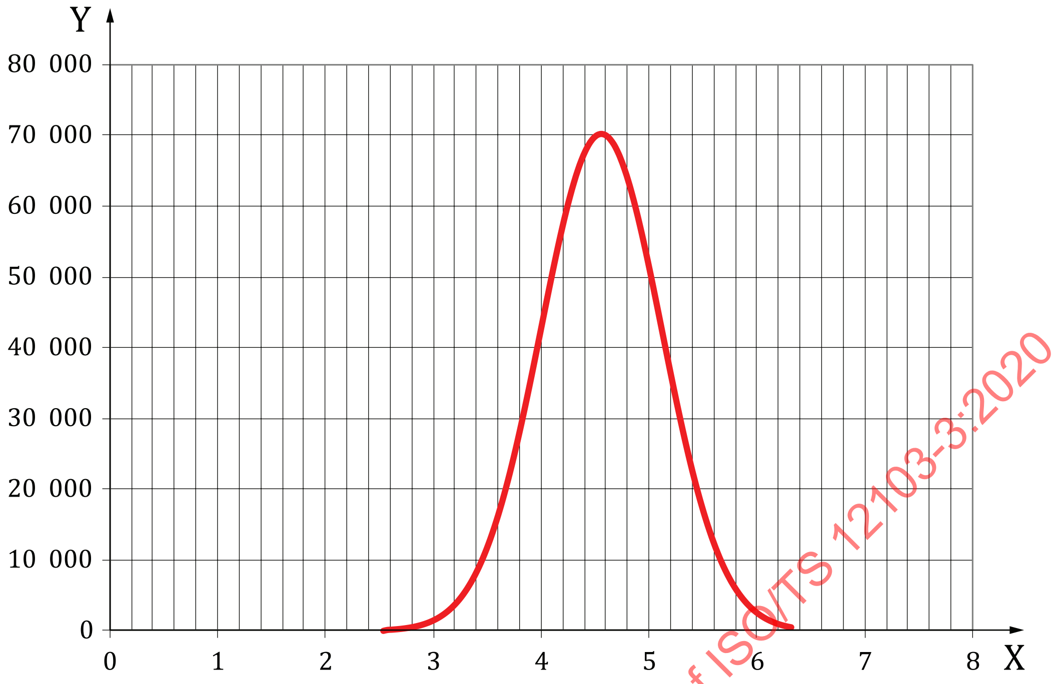
where

a is a scaling factor;

$x =$ ln(d) (particle diameter);

μ is the mean of distribution;

σ is the standard deviation.



Key

X logarithm of particle diameter [ln(d)]

Y particle counts (arbitrary)

Figure A.2 — Normal distribution with μ = 95 nm and σ_{geo} = 1,75

In this document the geometric standard deviation σ_{geo} has been used as a parameter. The relation of σ_{geo} to σ is defined as:

$$\sigma_{geo} = \cdot e^{\sigma_x} \qquad \text{(A.4)}$$

A.3 Limits for acceptable distributions

Table A.1 — Limits and tolerances

Parameter	Mean	Upper limit	Lower limit	
Soot aerosol mass concentration	Stable	+5 %	−5 %	
Soot aerosol concentration		10^7/cm^3		
Mean diameter of the permitted soot particle	95 nm	+10 nm	−10 nm	
Geometric standard deviation of mean diameter	1,75	+10 %	−10 %	
Mass count index			8 × 10^8 parts/mg	

Annex B
(informative)

Handling and use of contaminant

Tables B.1 to B.4 present health hazards in handling soot test aerosol.

Table B.1 — Physical and safety data

B.1.2	Density	(1,5 - 1,7) g/cm^3
B.1.4	Solubility in water	Insoluble
B.1.5	Hazardous reactions	Cardiovascular diseases, cancer, and respiratory diseases

Table B.2 — Protective measures for storage and handling

B.2.1	Industrial hygiene	Carbon monoxide is generated by incomplete combustion. There shall be sufficient ventilation in closed rooms.
B.2.2	Fire and explosion	Flammable
B.2.3	Industrial hygiene	Hazardous condition occurs if respiratory system is exposed to airborne particulates. A respirator or dust mask is recommended.
B.2.4	Disposal	Be aware of possible local regulations regarding disposal.

Table B.3 — Measures in case of accident or fire

B.3.1	Spillage or leakage	Vacuum or gather spilled material in a manner which does not create airborne dust.
B.3.2	Fire	No reaction with water or foam firefighting measures

Table B.4 — Health aspects

B.4.1	Toxicity	Moderately toxic as an acute irritating dust. The prolonged inhalation of dusts may result in the development of a disabling pulmonary fibrosis. Carbon monoxide is generated by incomplete combustion. There shall be sufficient ventilation in closed rooms.
B.4.2	Abrasiveness	Protective goggles should be worn to protect eyes. If eye contact occurs, flush eyes with plenty of water.
B.4.3	First aid	Eyes: rinse immediately with water. If irritation occurs, seek medical advice. Skin: wash with soap and water. If irritation develops, seek medical advice.
B.4.4	Industrial hygiene	

NOTE National regulations can vary from one country to another.

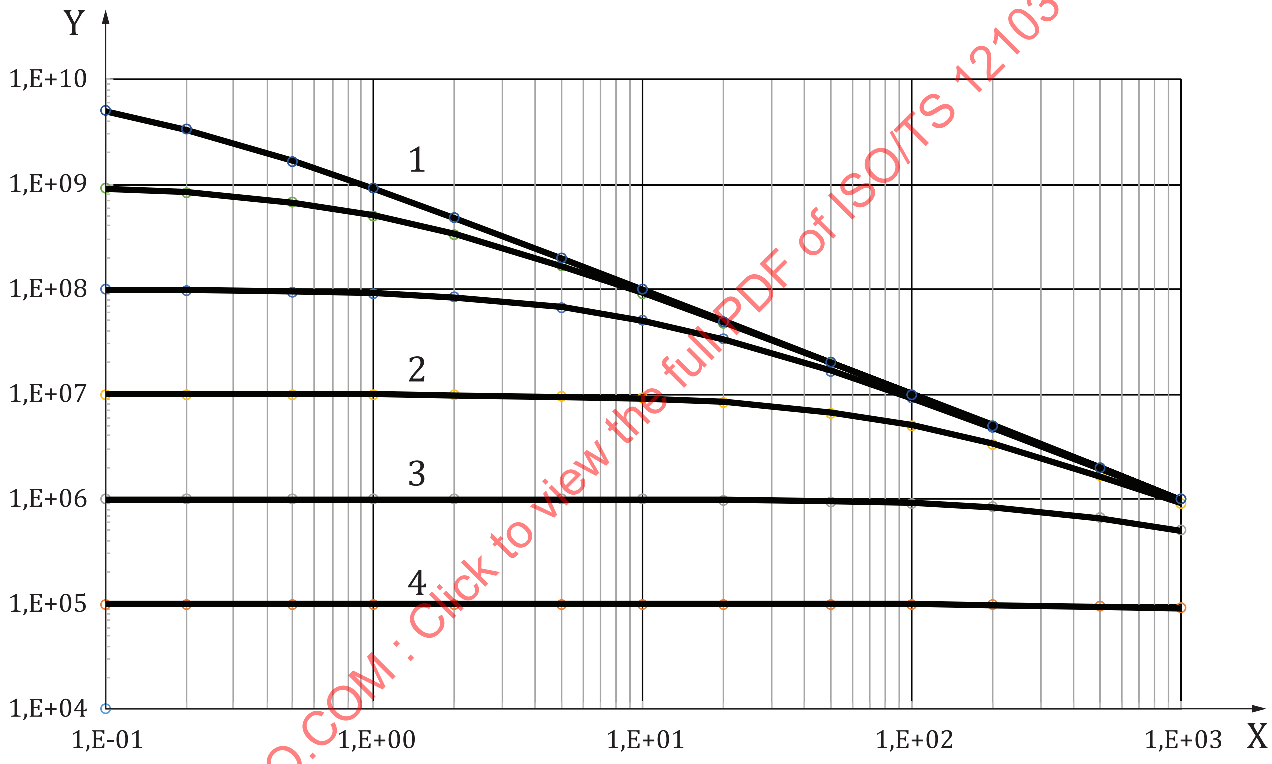
Annex C

(informative)

Concentration

Guideline for maximum number concentration

Contaminant concentration in the test system shall be monitored to minimize the time for which the contaminant reaches the unit under test. Figure C.1 shows the concentration by number versus time, to use during testing.



Key

X time (sec)

Y concentration by number [N(cm^{-3})]

1 initial concentration 10^{10} cm^{-3}

2 initial concentration 10^{7} cm^{-3}

3 initial concentration 10^{6} cm^{-3}

4 initial concentration 10^{5} cm^{-3}

Figure C.1 — Maximum number concentration

The particle losses due to coagulation at high concentrations are described by Formula (C.1):

$$\frac{C_n}{C_{n0}} = \frac{1}{\left(1 + K \cdot C_{n0} \cdot t\right)} \tag{C.1}$$

where

C_n is the particle concentration by number;

C_{n0} is the initial concentration by number;

t is the time;

K Is the coagulation constant.

Based on a particle size distribution with a mean value of 0,1 µm the half-life period (Halbwertszeit) can be specified, see Table C.1.

Table C.1 — Half-life period for particle size distribution with mean value of 0,1 µm

Initial concentration [cm^{-3}]	Half-life period $t_{1/2}$
10^{10}	2 ms
10^9	20 ms
10^8	12 s
10^7	2 min
10^6	20 min

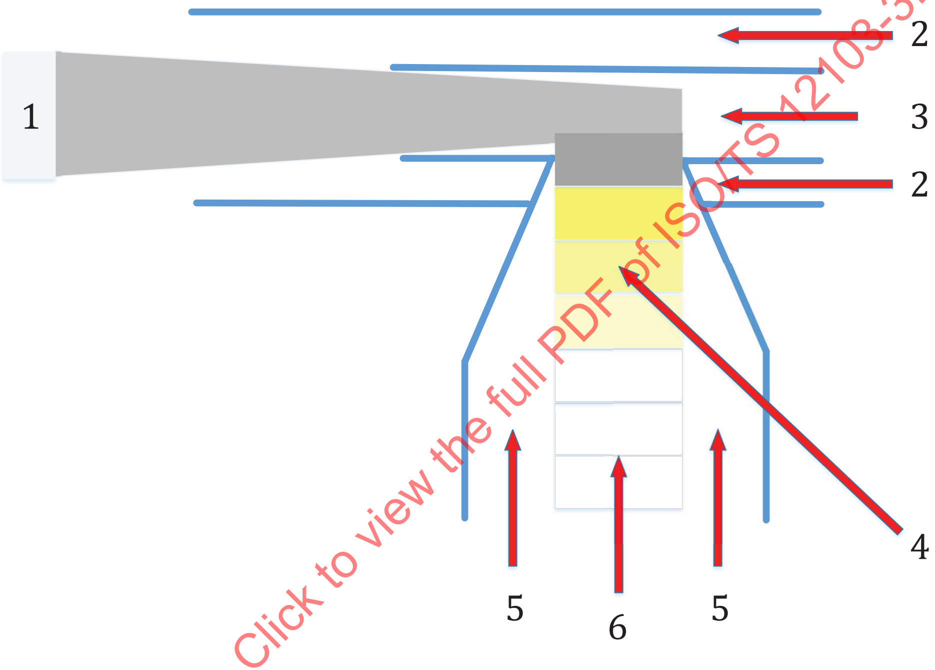
Annex D

(informative)

Flame operation and morphology

D.1 Flame operation

Figure D.1 shows the general set up of a diffusion flame.



Key

1 particle output
2 dilution gases
3 quenching gases
4 flame
5 air
6 gaseous fuel

Figure D.1 — Diffusion flame example

D.2 Morphology

Examples of a soot contaminant of different soot generation methods (SEM analysed) are included in Figures D.2 and D.3.

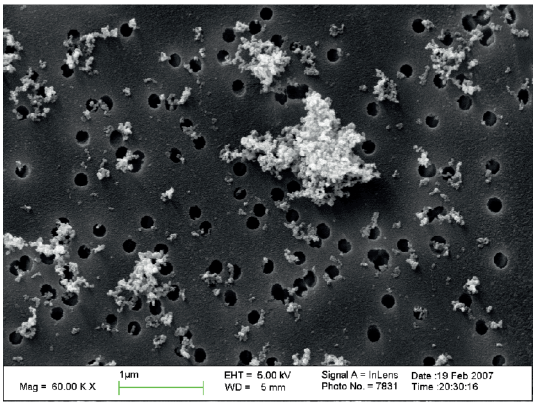


Figure D.2 — Soot from a diesel engine

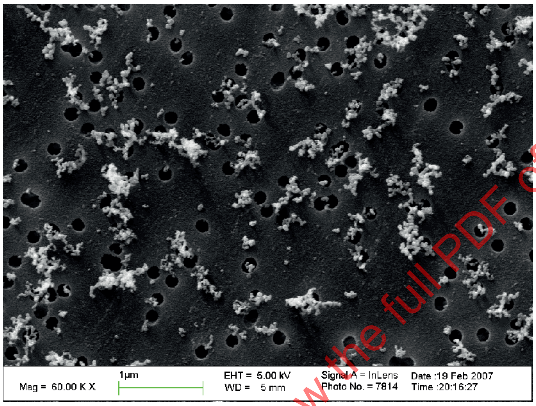


Figure D.3 — Soot particles generated by a diffusion flame, using ethylene

Annex E
(normative)

Thermal-optical transmission analysis

E.1 Description of the thermal-optical transmission analysis

Thermal-optical transmission is a conventional method for determining the carbonaceous aerosol fraction and for classifying it into organic carbon (OC) and elemental carbon (EC) by thermal treatment of a filter probe. In thermal-optical analyses, the carbonaceous material in aerosol particles deposited on a quartz-fibre filter is thermally desorbed according to a prescribed temperature protocol, first in an inert atmosphere (He) and then in an oxidizing atmosphere (He/O_2).

NOTE OC would desorb in the inert gas stream while EC would combust in the oxidizing atmosphere at high temperature. In reality however, thermally unstable organic compounds pyrolyse in the He-mode to form pyrolytic carbon (PC) which then usually desorbs off the filter in the oxidizing atmosphere, like native EC. Incorrectly accounting for PC formation can very significantly bias the discrimination between OC and EC. To correct for pyrolysis, the optical properties of the sample are monitored before and during the analysis with a laser beam. As PC absorbs the laser light, light transmission and reflectance decrease while OC chars in the He-mode. When PC and EC are released from the filter in the He/O_2-mode, transmission and reflectance increase again and the point at which the transmission and reflectance reach the pre-pyrolysis value is used to discriminate OC and EC (split point).

Several different thermal protocols are used with this analysis method. These protocols define the number of temperature steps, the temperatures and the time of each step. For the analysis of the aerosol described in this document, the protocol NIOSH 700+ shall be used.

E.2 Description of filter, conditioning and storage

High purity, high efficiency, binder free quartz-fibre filters shall be used.

Filters shall be cleaned, preconditioned in a muffle furnace operated at (800–900) °C for (1–2) h before use, to remove contaminated OC. Preconditioned filters are mostly available from analysis laboratories.

To avoid contamination, preconditioned filters shall be used within several days and stored in a suitable closed case under absence of air in the meantime.

Loaded filter after aerosol sampling shall also to be stored under absence of air and analysed as soon as possible, but within several days at last.

For each analysis campaign two blind filters shall be analysed at least. All of the filters used to evaluate the thermal-optical transmission shall be from the same charge and preconditioning and stored the same way.

E.3 Description of sampling

A sample shall be taken at the outlet of the soot generator. The sampling line shall be designed without restrictions and sharp changes in the flow direction to avoid particle losses. It shall take account on isokinetic sampling ideally. Sampling line shall not be heated. The temperature of the sample flow shall be no lower than ambient temperature and shall not exceed 80 °C when passing the filter.

For each operating point, two filter samples have to be analysed at least.

Sample flow should not exceed a rate of 6,2 cm/s through the filter.