

SLOVENSKI STANDARD SIST ISO 4387:2000

01-september-2000

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Cigarettes -- Determination of total and nicotine-free dry particulate matter using a routine analytical smoking machine

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Cigarettes -- Détermination de la matière particulaire totale et de la matière particulaire anhydre et exempte de nicotine au moyen d'une machine à fumer analytique de routine

SIST ISO 4387:2000

Ta slovenski standard je istoveten z: 0512ata99300/ssi-s-4387:2000

ICS:

65.160 V[àæ\£A[àæ} afa[å^|\afa[

Tobacco, tobacco products and related equipment

SIST ISO 4387:2000

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INTERNATIONAL STANDARD

ISO 4387

Third edition 2000-04-01

Cigarettes — Determination of total and nicotine-free dry particulate matter using a routine analytical smoking machine

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ISO 4387:2000(E)

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Printed in Switzerland

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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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

International Standard ISO 4387 was prepared by Technical Committee ISO/TC 126, *Tobacco and tobacco products*.

This third edition cancels and replaces the second edition (ISO 4387:1991), which has been editorially revised.

Annex A of this International Standard is for information only iteh ai

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Introduction

Cigarettes are manufactured to close tolerances using strict quality control procedures. However, all the constituents involved in the manufacture are derived from natural products (tobacco, cigarette paper, tipping, etc.) and this results in a final product which is intrinsically variable. The complexity does not finish here because the cigarette is converted during smoking to cigarette smoke.

Cigarette smoke is a complex mixture consisting of many individual chemical constituents. These compounds exist as gases, vapours and condensed aerosol particles. Additionally, various ageing processes, together with diffusional and intersolubility effects, start occurring immediately after the formation of the smoke which further complicate its composition.

The quantitative measurement of nicotine-free dry particulate matter (NFDPM, sometime referred to as "tar") is, therefore, dependent on its arbitrary definition.

From the time that scientists have attempted to determine a value for NFDPM, many different methods have been used. However, experience has shown some procedures to be more reliable and, with these factors in mind, during 1988 and 1989, collaborative studies by Task Forces composed of members of the Cooperation Centre for Scientific Research Relative to Tobacco (CORESTA) Smoke and Technology groups have been made on the repeatability and reproducibility of the determination of total and dry particulate matter from cigarettes.

The studies show that improvements in repeatability and reproducibility result when some restrictions are placed on the wide variety of methods and practices permitted by existing standard methods. Thus, this International Standard, and the others which together form a complete set for the sampling, conditioning and determination of nicotine, water and particulate matter from cigarettes, have been produced after much cooperation and collaborative experimentation by many laboratories in many countries.

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CORESTA first published an International Standard for the machine smoking of cigarettes in 1968, and since that time many improvements in equipment as well as in procedure have been suggested.

This International Standard incorporates these improvements and consequently represents the state of the art on this subject and provides one set of procedures accepted as reference methods.

This method is a machine method and allows cigarettes to be smoked using a strictly controlled set of parameters. Thus, it enables the NFDPM and nicotine from cigarettes, when smoked by this procedure, to be compared and ranked on the basis of machine yield.

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Cigarettes — Determination of total and nicotine-free dry particulate matter using a routine analytical smoking machine

1 Scope

This International Standard specifies methods for the determination of total particulate matter and for the subsequent determination of nicotine-free dry particulate matter present in the smoke from cigarettes generated and collected using a routine analytical smoking machine.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

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ISO 2971, Cigarettes and filter rods — Determination of nominal diameter —Method using a laser beam measuring apparatus.

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ISO 3308:2000, Routine analytical cigarette-smoking machine Definitions and standard conditions.

ISO 3402, Tobacco and tobacco products — Atmosphere for conditioning and testing.

ISO 6488-1, Tobacco — Determination of water content — Part 1: Karl Fischer method.

ISO 6565, Tobacco and tobacco products — Draw resistance of cigarettes and pressure drop of filter rods — Standard conditions and measurement.

ISO 8243, Cigarettes — Sampling.

ISO 10315, Cigarettes — Determination of nicotine in smoke condensates — Gas-chromatographic method.

ISO 10362-1, Cigarettes — Determination of water in smoke condensates — Part 1: Gas-chromatographic method.

ISO 16055, Tobacco and tobacco products — Monitor test piece — Requirements and application.

3 Terms, definitions and abbreviated terms

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

3.1 total particulate matter crude smoke condensate TPM

that portion of the mainstream smoke which is trapped in the smoke trap, expressed as milligrams per cigarette

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3.2

dry particulate matter dry smoke condensate

DPM

total particulate matter after deduction of its water content, expressed as milligrams per cigarette

3.3

nicotine-free dry particulate matter nicotine-free dry smoke condensate **NFDPM**

dry particulate matter after deduction of its nicotine content, expressed as milligrams per cigarette

3.4

smoking process

use of a smoking machine to smoke cigarettes from lighting to final puff

3.5

smoking run

specific smoking process to produce such smoke from a sample of cigarettes as is necessary for the determination of the smoke components

3.6

laboratory sample

sample intended for laboratory inspection or testing and which is representative of the gross sample or the subperiod sample

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3.7

test sample

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cigarettes for test taken at random from the laboratory sample and which are representative of each of the increments making up the laboratory sample

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3.8 conditioning sample

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cigarettes selected from the test sample for conditioning prior to tests

3.9

test portion

group of cigarettes prepared for a single determination and which is a random sample from the test sample or conditioned sample, as appropriate

3.10

monitor test piece

cigarette taken from a batch specially fabricated under controlled manufacturing conditions

The cigarettes of such a batch show the greatest possible homogeneity with regard to their physical and chemical characteristics.

Principle

The test cigarettes are sampled then conditioned. The test cigarettes are smoked on an automatic smoking machine with simultaneous collection of total particulate matter in a glass fibre filter trap. If used, the consistency of the laboratory smoking process and subsequent analytical procedures are controlled by using monitor test pieces specified in ISO 16055. The mass of the total particulate matter so collected is determined gravimetrically. The total particulate matter is extracted from the trap for determination of the water and nicotine contents by gas chromatography.

NOTE In the countries that are not in a position to use gas-chromatographic methods, reference should be made to ISO 3400 for the determination of total nicotine alkaloids, and the determination of water in smoke condensate should be performed by the method described in ISO 10362-2. In such cases, values obtained for nicotine and water in smoke condensate may be used with the addition of a note made in the expression of the result.

5 Apparatus

Normal laboratory apparatus and, in particular, the following items.

- **5.1** Routine analytical cigarette-smoking machine, complying with the requirements of ISO 3308.
- **5.2** Soap bubble flow meter, graduated at 35 ml to an accuracy of \pm 0,2 ml and with a resolution of 0,1 ml.
- 5.3 Apparatus for the determination of puff duration and frequency.
- **5.4** Analytical balance, suitable for measuring to the nearest 0,1 mg.

The weighing of filter pad holders may be affected by static electricity, necessitating the use of an antistatic device.

- **5.5** Conditioning enclosure, carefully maintained under the conditions specified in ISO 3402.
- **5.6 Length-measuring device**, suitable for measuring to the nearest 0,5 mm.
- 5.7 Device for the determination of diameter, in accordance with ISO 2971.

If such apparatus is not available, the diameter may be determined from the circumference by slitting the cigarette longitudinally, removing and flattening the paper then measuring its width.

- 5.8 Smoke trap sealing device, end caps made from a non-hygroscopic and chemically inert material.
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- **5.9** Gloves, made of cotton, or the non-talc surgical type 0-4387-2000

6 Sampling

A laboratory sample (3.6) shall be taken by a sampling scheme such as one of those given in ISO 8243.

This sample will normally contain cigarettes taken from different parts of the population. Make up the test sample (3.7) required for the test by randomly selecting cigarettes from the different parts of the population represented in the laboratory sample.

7 Determination of total particulate matter

7.1 Preparation of the cigarettes for smoking

7.1.1 General

If N cigarettes of a given type are to be smoked, $C \times N$ cigarettes shall be prepared from Q cigarettes for conditioning and butt marking.

The symbols used in this clause are as follows:

N is the number of cigarettes of a given type to be smoked, resulting from sampling at one point in time or from a sub-period sample;

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