

Methods for

Chemical analysis of tobacco and tobacco products —

**Part 14: Determination of total and
nicotine-free dry particulate matter
using a routine analytical smoking
machine**

Committees responsible for this British Standard

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Department of Trade and Industry (Laboratory of the Government Chemist)
Tobacco Advisory Council

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National foreword

This Part of BS 5202 has been prepared under the direction of the Agriculture and Food Standards Policy Committee. It is identical with ISO 4387:1991 “Cigarettes — Determination of total and nicotine-free dry particulate matter using a routine analytical smoking machine”, published by the International Organization for Standardization (ISO), and in the preparation of which the United Kingdom played a full part.

Cross-references

International Standard	Corresponding British Standard
ISO 2971:1987	BS 5381 <i>Methods for determination of physical properties of tobacco and tobacco products</i> Part 2:1988 <i>Nominal diameter of cigarettes and filters (pneumatic method)</i> (Identical)
ISO 3308:1991	BS 5668 <i>Routine analytical cigarette smoking machine</i> Part 1:1992 <i>Specification and standard conditions</i> (Identical)
ISO 3400:1989	BS 5202 <i>Methods for chemical analysis of tobacco and tobacco products</i> Part 7:1990 <i>Determination of alkaloids in smoke condensate of cigarettes (spectrometric method)</i> (Identical)
ISO 3402:1991	BS 5743:1991 <i>Specification for atmosphere for conditioning and testing tobacco and tobacco products</i> (Identical)
ISO 6565:1983	BS 5381 <i>Methods for determination of physical properties of tobacco and tobacco products</i> Part 5:1983 <i>Draw resistance of cigarettes and filter rods</i> (Identical)
ISO 8243:1991	BS 6245 <i>Sampling tobacco and tobacco products</i> Part 3:1992 <i>Methods of sampling cigarettes</i> (Identical) BS 5202 <i>Methods for chemical analysis of tobacco and tobacco products</i>
ISO 10315:1991	Part 12:1991 <i>Determination of nicotine in smoke condensate of cigarettes (gas-chromatographic method)</i> (Identical)
ISO 10362-1:1991	Part 13:1991 <i>Determination of water in smoke condensate of cigarettes (gas-chromatographic method)</i> (Identical)

The Technical Committee has reviewed the provisions of ISO 6488:1981, and has decided that they are acceptable for use in conjunction with this standard.

A British Standard does not purport to include all the necessary provisions of a contract. Users of British Standards are responsible for their correct application.

Compliance with a British Standard does not of itself confer immunity from legal obligations.

Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, pages 1 to 14, an inside back cover and a back cover.

This standard has been updated (see copyright date) and may have had amendments incorporated. This will be indicated in the amendment table on the inside front cover.

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 to complicate its composition further.

The quantitative measurement of nicotine-free dry particulate matter (NFDPM, sometimes 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 upon 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.

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.

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 standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 2971:1987, *Cigarettes and filters — Determination of nominal diameter — Pneumatic method*.

ISO 3308:1991, *Routine analytical cigarette-smoking machine — Definitions and standard conditions*¹⁾.

ISO 3402:1991, *Tobacco and tobacco products — Atmosphere for conditioning and testing*.

ISO 6488:1981, *Tobacco — Determination of water content (Reference method)*.

ISO 6565:1983, *Tobacco and tobacco products — Draw resistance of cigarettes and filter rods — Definitions, standard conditions and general aspects*.

ISO 8243:1991, *Cigarettes — Sampling*.

ISO 10315:1991, *Cigarettes — Determination of nicotine in smoke condensates — Gas-chromatographic method*.

ISO 10362-1:1991, *Cigarettes — Determination of water in smoke condensates — Part 1: Gas-chromatographic method*.

¹⁾ To be published.

3 Definitions

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

3.1

total particulate matter; crude smoke condensate

that portion of the mainstream smoke which is trapped in the smoke trap, expressed as milligrams per cigarette (mg/cig.)

3.2

dry particulate matter; dry smoke condensate

the total particulate matter after deduction of its water content, expressed as milligrams per cigarette (mg/cig.)

3.3

nicotine-free dry particulate matter; nicotine-free dry smoke condensate

the dry particulate matter after deduction of its nicotine content, expressed as milligrams per cigarette (mg/cig.)

3.4

clearing puff

any puff taken after the cigarette has been extinguished or removed from the cigarette holder

3.5

smoking process

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

3.6

smoking run

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

3.7

laboratory sample

the sample intended for laboratory inspection or testing and which is representative of the gross sample or the sub-period sample

3.8

conditioning sample

the cigarettes selected from the test sample for conditioning prior to tests for particulate matter yield

3.9

test sample

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

3.10

test portion

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

3.11

conditioned sample

conditioned cigarettes smoked for particulate matter yield tests

4 Principle

Sampling of the test cigarettes. Conditioning of the test cigarettes. Smoking of the test cigarettes on an automatic smoking machine with simultaneous collection of total particulate matter in a glass fibre filter trap. Gravimetric determination of the mass of total particulate matter so collected. Extraction of the total particulate matter from the trap for the determination of the water and nicotine contents by gas chromatography.

NOTE 1 In the countries that are not in a position to use the 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 a Karl Fischer method²⁾. 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 with a resolution of 0,1 ml.

5.3 *Apparatus* for the determination of puff duration and frequency.

5.4 *Analytical balance*, with a resolution of 0,1 mg.

5.5 *Draw resistance testing equipment*

5.6 *Conditioning enclosure*, carefully maintained under the conditions specified in ISO 3402.

5.7 *Length-measuring device*, suitable for measuring to the nearest 0,5 mm.

5.8 *Apparatus for the determination of diameter*, in accordance with ISO 2971.

5.9 *Sealing device*, end caps made from the same non-hygroscopic and chemically inert material as the filter holder.

5.10 *Gloves*, cotton or non-talc surgical.

²⁾ ISO/TC 126 is at present examining a working draft for the Karl Fischer method of water determination.