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BSI Standards Publication

**Cigarettes — Determination of selected carbonyls
in the mainstream smoke of cigarettes — Method
using high performance liquid chromatography**

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National foreword

This British Standard is the UK implementation of ISO 21160:2018.

The UK participation in its preparation was entrusted to Technical Committee AW/40, Tobacco and tobacco products.

A list of organizations represented on this committee can be obtained on request to its secretary.

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

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Cigarettes — Dosage de carbonyles sélectionnés dans le courant principal de la fumée de cigarette — Méthode par chromatographie liquide haute performance



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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).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on 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 the following URL: www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 126, *Tobacco and tobacco products*.

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

At the outset of this work, discussions in the CORESTA (www.coresta.org) Special Analytes Sub-Group (since 2017 the Sub-Group changed its name to Smoke Analytes) determined that most laboratories used a method involving derivatization of carbonyls with 2,4-dinitrophenylhydrazine (DNPH) because they considered it the most suitable. This was chosen as the basis of the CORESTA Recommended Method (CRM). The CRM comprised smoke collection in impinger traps, derivatization of carbonyls with DNPH followed by their determination using reversed phase High Performance Liquid Chromatography with Ultra Violet or Diode Array Detection (HPLC-UV or HPLC-DAD).

Initial joint experiments and ongoing discussions addressed some methodological aspects that needed to be evaluated before the drafting of a CRM. This method was produced through a collaborative study undertaken in 2010 involving 15 laboratories from 11 countries using the ISO 3308 smoking regime^[1]. Further data are provided for the same selected carbonyl compounds from 10 samples with different tar yields from the collaborative study in 2012, which involved 19 laboratories from 11 countries^[2].

This method includes recommendations about some of the critical steps that should be controlled to provide data as robust and consistent as the repeatability and reproducibility data provided in the ISO document. Statistical evaluations were carried out according to ISO 5725-1 and ISO 5725-2^{[3],[4]}.

No machine smoking regime can represent all human smoking behaviour.

- It is recommended that cigarettes also be tested under conditions of a different intensity of machine smoking than those specified in this document.
- Machine smoking testing is useful to characterize cigarette emissions for design and regulatory purposes, but communication of machine measurements to smokers can result in misunderstandings about differences in exposure and risk across brands.
- Smoke emission data from machine measurements may be used as inputs for product hazard assessment, but they are not intended to be nor are they valid as measures of human exposure or risks. Communicating differences between products in machine measurements as differences in exposure or risk is a misuse of testing using ISO standards.

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Cigarettes — Determination of selected carbonyls in the mainstream smoke of cigarettes — Method using high performance liquid chromatography

WARNING — The use of this document can involve hazardous materials, operations and equipment. This document does not purport to address all the safety problems associated with its use. It is the responsibility of the user of this document to establish appropriate safety and health practices, and determine the applicability of any other restrictions prior to use.

1 Scope

This document specifies a method for the determination of selected carbonyls (formaldehyde, acetaldehyde, acetone, acrolein, propionaldehyde, crotonaldehyde, 2-butanone and *n*-butyraldehyde) as their 2,4-dinitrophenylhydrazones in mainstream smoke using reversed phase HPLC-UV/DAD.

This method is applicable to cigarettes with nicotine-free dry particulate matter (NFDPM) yields between 1 mg/cigarette and 15 mg/cigarette using reversed phase HPLC-UV/DAD.

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 3308, *Routine analytical cigarette-smoking machine — Definitions and standard conditions*

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

ISO 4387, *Cigarettes — Determination of total and nicotine-free dry particulate matter using a routine analytical smoking machine*

ISO 8243, *Cigarettes — Sampling*

3 Terms and definitions

No terms and definitions are listed in this document.

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/>

4 Principle

Cigarettes are smoked on a standard smoking machine as specified in ISO 3308 that has been fitted with impingers, but without a glass fibre filter pad as described in ISO 3308 (Cambridge filter pad; CFP, for example of equivalent product) and the filter pad holder, under the ISO 3308 smoking regime.

The carbonyls in mainstream tobacco smoke are trapped by passing each puff through an impinger device containing an acidified solution of 2,4-dinitrophenylhydrazine (DNPH) in 1:1 acetonitrile:water.

An aliquot of the smoke extract is then syringe-filtered and diluted with 1 % tris-(hydroxymethyl)-aminomethane in aqueous acetonitrile.