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Cigarettes — Determination of ammonia in cigarette mainstream smoke using ion chromatography

Cigarettes — Dosage de l'ammoniac dans le courant principal de la fumée de cigarette par chromatographie par échange d'ions

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ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Fax: +41 22 749 09 47
Email: copyright@iso.org
Website: www.iso.org

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Contents

Page

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 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.

Introduction

The CORESTA Smoke Analytes Sub-Group[1)] conducted a survey among its members and determined that most laboratories used a method involving ion chromatography for the determination of ammonia in cigarette mainstream smoke. Two alternative trapping systems were used, either with a combination of glass fibre filter pad followed by impinger traps or with an impregnated glass fibre filter pad followed by a glass fibre filter pad.

A CORESTA recommended method (CRM) was written[1] on the basis of the results obtained in an interlaboratory study conducted in 2015 involving 17 laboratories from 8 countries using cigarettes manufactured from a range of blend styles[2]. The results demonstrated equivalency of the data obtained by using both trapping systems. It was observed that the method is not applicable to dark-air cured blended cigarettes.

This document is based upon the CRM 83 and includes statistical evaluations carried out according to ISO 5725-1 and ISO 5725-2.

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 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.

1) Until 2017, the sub-group has been previously known as CORESTA Special Analytes Sub-Group.

Cigarettes — Determination of ammonia in cigarette mainstream smoke using ion chromatography

WARNING — The use of this document involves 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 quantification of ammonia by ion chromatography in mainstream smoke using ISO 3308 smoking parameters.

This method is applicable to cigarettes with ammonia yields between 1 µg/cigarette and 30 µg/cigarette. It is not applicable for the determination of ammonia in dark-air cured cigarettes.

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

Ammonia is collected by passing the mainstream smoke of cigarettes through either

a) a glass fibre filter pad as specified in ISO 3308 followed by impinger traps containing dilute sulphuric acid (trapping system 1 in the document), or

b) an impregnated glass fibre filter pad followed by an untreated glass fibre filter pad (trapping system 2 in the document).

The glass fibre filter pad is extracted either with the impinger solutions (trapping system 1) or with dilute hydrochloric acid (trapping system 2). The obtained solutions are analysed by ion chromatography using an external standard calibration.

5 Apparatus

The usual laboratory apparatus for use in preparation of samples, solutions and standards and, in particular, the following.

5.1 Routine analytical cigarette-smoking machine, complying with the requirements of ISO 3308.

5.2 Impinger trapping system, capable of being connected in series.

5.3 High performance liquid chromatography system, consisting of a conductivity detector and conductivity suppressor, an eluent degassing unit, a gradient pump, an autosampler with sampling loop and cooling unit, a data collection system.

5.4 Cation exchange analytical column, e.g. Dionex™ IonPac[2)] CS16 IC or equivalent.

5.5 Disposable guard column, e.g. Dionex™ IonPac CG16[2)] or equivalent.

5.6 Analytical balance, suitable for measuring to the nearest 0,1 mg.

5.7 Glassware, Erlenmeyer flasks of appropriate volumes with ground glass stoppers.

5.8 Polypropylene, tubing, volumetric flasks (25 ml, 50 ml, 100 ml and 1 l).

5.9 Mechanical pipettes with disposable plastic tips.

6 Reagents

All reagents shall be at least of analytical reagent grade.

6.1 Ammonium sulfate [$(NH_4)_2SO_4$] > 99 % purity.

6.2 Sulphuric acid (H_2SO_4) > 96 % purity (for trapping system 1).

6.3 Hydrochloric acid (HCl) > 36,5 % to 38 % purity (for trapping system 2).

6.4 Methanesulphonic acid (MSA) > 99 % purity.

6.5 Ethanol > 99 % purity.

6.6 Deionised water, with a resistivity > 18 MΩ·cm at 25 °C.

7 Preparation

7.1 General

Polypropylene and glass containers shall be cleaned and dried in such a manner which ensures that contamination does not occur.

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2) Dionex Ion Pac is an example of a suitable product available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of this product. Equivalent products may be used if they can be shown to lead to the same results.

7.2 Preparation of solutions

7.2.1 Sulphuric acid, 0,01 M — Impinger solution (trapping system 1)

Carefully add 1,022 g of H_2SO_4 (w = 96 %) to a minimum of 500 ml of deionised water in a 1 l volumetric flask. Mix and dilute to volume with deionised water. Store the solution in a bottle at ambient temperature.

7.2.2 Sulphuric acid, 0,1 M — Solution C (Ion chromatography eluent)

Carefully add 10,22 g of H_2SO_4 (w = 96 %) to a minimum of 500 ml of deionised water in a 1 l volumetric flask. Mix and dilute to volume with deionised water.

7.2.3 MSA 0,003 M — Solution A (Ion chromatography eluent)

Carefully add 0,288 g MSA to 900 ml of deionised water in a 1 l volumetric flask. Mix and dilute to volume with deionised water.

7.2.4 Sulphuric acid, 0,01 M — Ammonium standards preparation

Carefully add 1,022 g of H_2SO_4 (w = 96 %) to a minimum of 500 ml of deionised water in a 1 l volumetric flask. Mix and dilute to volume with deionised water. Store the solution in a bottle at ambient temperature.

7.2.5 Hydrochloric acid, 0,05 M — Solution for glass fibre filter pad impregnation (trapping system 2)

Carefully add 4,3 ml of HCl (w = 36,5 % to 38 %) to a minimum of 500 ml of ethanol in a 1 l volumetric flask. Mix and dilute to volume with deionised water. Store the solution in a bottle at ambient temperature.

7.2.6 Hydrochloric acid, 0,01 M — Extraction solution (trapping system 2)

Carefully add 0,9 ml HCl (w = 36,5 % to 38 %) to a minimum of 500 ml of deionised water in a 1 l volumetric flask. Mix and dilute to volume with deionised water. Store the solution in a bottle at ambient temperature.

7.3 Preparation of standards

7.3.1 Primary ammonium stock solution

Accurately weigh 0,10 g of $(NH_4)_2SO_4$ into a 25 ml volumetric flask. Note the exact weight in order to accurately calculate the standard concentrations. Dissolve in the 0,01 M sulphuric acid solution (see 7.2.4) and dilute to volume with the same solution.

NOTE 1 The solution is stable for about 30 days when stored in a refrigerator.

NOTE 2 It corresponds approximately to a 1 000 µg/ml ammonium stock solution.

NOTE 3 A certified reference material at 1 000 µg/ml is suitable as well.

7.3.2 Calibration standards

A range of calibration standard solutions is prepared with appropriate volumes (0,02 ml to 0,20 ml) of the primary ammonium stock solution (see 7.3.1) diluted to prescribed volumes with 0,01 M sulphuric acid (see 7.2.4), according to Table 1.