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Cigarettes — Determination of ammonia in cigarette mainstream smoke with an intense smoking regime using ion chromatography

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Contents

Page

Foreword	iv
Introduction	v
1 Scope	1
2 Normative references	1
3 Terms and definitions	1
4 Principle	1
5 Apparatus	2
6 Reagents	2
7 Preparation	3
7.1 General.....	3
7.2 Preparation of solutions.....	3
7.2.1 Sulphuric acid, 0,01 M — Impinger solution (trapping system 1).....	3
7.2.2 Sulphuric acid, 0,1 M — Solution C (Ion Chromatography Eluent).....	3
7.2.3 MSA 0,003 M — Solution A (Ion chromatography eluent).....	3
7.2.4 Sulphuric acid, 0,01 M — Ammonium standards preparation.....	3
7.2.5 Hydrochloric acid, 0,05 M — Solution for CFP impregnation (trapping system 2).....	3
7.2.6 Hydrochloric acid, 0,01 M — Extraction solution (trapping system 2).....	3
7.3 Preparation of standards.....	3
7.3.1 Primary ammonium stock solution.....	3
7.3.2 Calibration standards.....	4
8 Sampling	4
9 Tobacco product preparation	4
10 Sample generation — Smoking of cigarettes	4
10.1 General.....	4
10.2 Smoking machine setup.....	4
10.2.1 General.....	4
10.2.2 Trapping system 1 — CFP and impinger traps.....	4
10.2.3 Trapping system 2 — Impregnated CFP.....	5
10.3 Smoking.....	6
11 Sample analysis	6
11.1 Preparation of sample.....	6
11.1.1 Extraction of CFP — Trapping system 1.....	6
11.1.2 Extraction of CFP — Trapping system 2.....	6
11.2 Determination.....	6
11.2.1 Ion chromatography operating conditions.....	6
11.2.2 Calibration.....	7
11.2.3 Calculation.....	8
12 Repeatability and reproducibility	8
12.1 General.....	8
12.2 Results of the 2015 collaborative study.....	9
13 Report	9
Annex A (informative) Examples of chromatograms	10
Bibliography	11

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).

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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 CORESTA¹⁾ Smoke Analytes Sub-Group²⁾ 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 Cambridge filter pad followed by impinger traps or with an impregnated CFP followed by a CFP.

A CORESTA recommended method (CRM) was written^[1] on the basis of the results obtained in an interlaboratory study conducted in 2015 involving 18 laboratories and 8 samples 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 was 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) Available at: www.coresta.org.

2) Until 2017, the sub-group was known as CORESTA Special Analytes Sub-Group.

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Cigarettes — Determination of ammonia in cigarette mainstream smoke with an intense smoking regime 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 20778 smoking parameters.

This method is applicable to an ammonia yield of about 10 µg/cigarette to 40 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 3402, *Tobacco and tobacco products — Atmosphere for conditioning and testing*

ISO 8243, *Cigarettes — Sampling*

ISO 20778, *Cigarettes — Routine analytical cigarette smoking machine — Definitions and standard conditions with an intense smoking regime*

ISO 20779, *Cigarettes — Generation and collection of total particulate matter using a routine analytical smoking machine with an intense smoking regime*

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 with intense smoking regime through either:

- a) a glass fibre filter pad as specified in ISO 20779 (e.g. Cambridge filter pad, CFP) 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 pads are 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 20778.

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

5.3 Ion 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 IonPac³⁾ CS16 IC or equivalent.

5.5 Guard column, e.g. Dionex IonPac CG16 or equivalent.

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

5.7 Plasticware, 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 ($(\text{NH}_4)_2\text{SO}_4$) > 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 M Ω .cm at 25 °C.

3) 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.

7 Preparation

7.1 General

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

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 minimum 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 minimum 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 CFP impregnation (trapping system 2)

Carefully add 4,3 ml of HCl ($w = 36,5 \%$ to 38%) to 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 minimum 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 $(\text{NH}_4)_2\text{SO}_4$ 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 $\mu\text{g}/\text{ml}$ ammonium stock solution.

NOTE 3 A certified reference material at 1 000 $\mu\text{g}/\text{ml}$ is suitable as well.