

No: T-115
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Page: 1 of 5

1 SCOPE OF APPLICATIONS

- 1.1** Applicable to the collection and quantitation of the tar, water, nicotine and CO contents of mainstream tobacco smoke from cigarettes, cigarette equivalents, bidis and cigars.

2 NORMATIVE REFERENCES

- 2.1** International Organization for Standardization (ISO) Methods:
- 2.1.1** ISO 3308:1991 Cigarettes – Routine Analytical Cigarette-Smoking Machine - Definitions and Standard Conditions, 1991-10-15.
 - 2.1.2** ISO 3402:1991 Tobacco and Tobacco Products – Atmospheres for Conditioning and Testing, 1991-07-01.
 - 2.1.3** ISO 4387:1991 Cigarettes – Determination of total and nicotine-free dry matter using a routine analytical smoking machine, 1991-10-15.
 - 2.1.4** ISO 8243:1991 Cigarettes – Sampling, 1991-10-15.
 - 2.1.5** ISO 8454:1995 Cigarettes – Determination of carbon monoxide in the vapour phase of cigarette smoke – NDIR method, 1991-11-15.
 - 2.1.6** ISO 10315:1991 Cigarettes – Determination of nicotine in smoke condensates – Gas-chromatographic method, 1991-08-01.
 - 2.1.7** ISO 10362-1:1991 Cigarettes – Determination of water in smoke condensates - Part 1: Gas-chromatographic method, 1991-09-15.
- 2.2** CAN/CGSB-176.1-92 - Preparation of Cigarettes from Cigarette Tobacco for Testing. National Standard of Canada. Canadian General Standards Board, December 1992.
- 2.3** Health Canada Test Method T-401 – Preparation of cigarettes from packaged leaf tobacco for testing, 1999-12-31.

3 DEFINITIONS

- 3.1** Cigarette Equivalents - cigarettes prepared from either leaf tobacco (see Health Canada Test Method T-401) or other cured and processed tobacco by incorporation into cigarette tubes (see CAN/CGSB-176.1-92).
- 3.2** **Cigars**
- 3.2.1** Small cigars: defined as weighing between 1.3 and 2.5 g, 70-120 mm in length and diameter less than 17 mm.

3.2.2 Regular cigars: defined as weighing between 5 and 17 g, 110-150 mm in length, diameter 17 mm or less.

3.2.3 Premium cigars: defined as having diameters ranging from 12-23 mm and lengths between 127 and 214 mm.

3.3 Smoking Conditions for Cigars

3.3.1 Small cigars - These products are tested under standard ISO conditions as described in ISO 3308:1991.

3.3.2 Regular and premium cigars - These products are tested under Cooperation Centre for Scientific Research Relative to Tobacco (CORESTA) conditions as described in the CORESTA Informational Bulletin 1, 31-34 (1974). The puff volume will be 20 mL, puff duration 1.5 seconds, and puff frequency 40 seconds. The cigar is smoked to a butt length of 33 mm.

3.4 Smoke Train – The path followed by tobacco smoke after exiting the tobacco product. This often includes arrangements of traps and solutions specific to individual test methods.

3.5 Tar – Nicotine-free dry particulate matter (NFDPM).

3.6 Intense Smoking Conditions - See ISO 3308:1991 Sections 4.2 - 4.4 with the following values; puff duration; 2.0 seconds; puff volume, 55 mL; puff frequency, 30 seconds. Filter and tipping paper shall be wrapped by a single layer of "invisible" tape, such as Highland™ brand, 19.0 mm wide or equivalent.

3.7 Breakthrough – results from exceeding the capacity of the glass fibre filter disc as evidenced by brown stains on the side of the filter disc that is remote from the cigarette being smoked.

4 METHOD SUMMARY

Five conditioned cigarettes are smoked per port, using an automated 20-port constant volume smoking machine equipped with a CO analyzer, onto a conditioned, pre-weighed glass fiber filter disc (pad). The gas phase is collected in a Vapour Phase (VP) collection bag and then introduced into a Non-Dispersive Infra-Red analyzer (NDIR) and the % CO determined. The pad is then re-weighed and the difference is the Total Particulate Matter (TPM). The pad is extracted with Isopropanol (IPA) containing the internal standards, and the extract analyzed for nicotine and water by gas chromatography. The tar value is determined by subtracting the water and nicotine from the TPM.

Note: The testing and evaluation of certain products against this test method may require the use of materials and or equipment that could potentially be hazardous and this document does not purport to address all the safety aspects associated with its use. Anyone using this test method has the responsibility to consult with the appropriate authorities and to establish health and safety practices in conjunction with any existing applicable regulatory requirements prior to its use.

5 APPARATUS AND EQUIPMENT

- 5.1** All apparatus and equipment shall be as those set out in the appropriate standards referenced in section 2.

6 REAGENTS AND SUPPLIES

6.1 All reagents shall be, at the least, recognized as analytical reagent grade in quality.

6.2 All reagents and supplies shall be as those set out in the appropriate standards referenced in section 2 with the following addition:

6.2.1 Anethole (purity at least 99 %) may also be used as an internal standard for the determination of nicotine as set out in ISO 10315:1991, Section 4.4.

7 PREPARATION OF GLASSWARE

7.1 Glassware should be cleaned and dried in such a manner to ensure that contamination from glassware does not occur.

8 PREPARATION OF SOLUTIONS

8.1 Prepare solutions required for analysis, as set out in the appropriate standards referenced in section 2, in accordance with good laboratory practice.

9 PREPARATION OF STANDARDS

9.1 Prepare standards required for analysis, as set out in the appropriate standards referenced in section 2, in accordance with good laboratory practice.

10 SAMPLING

10.1 The sampling of cigarettes for the purpose of testing shall be in accord with ISO 8243:1991.

10.2 The sampling of leaf tobacco or other cured or processed tobacco for the purpose of testing shall be in accord with Appendix A of CAN/CGSB-176.

11 TOBACCO PRODUCT PREPARATION

11.1 Product shall be conditioned for at least 48 hours and to a maximum of 10 days at 22°C and 60% relative humidity as set out in ISO 3402:1991.

11.2 Cigarettes, small cigars, bidis and cigarette equivalents shall be marked for butt length as per ISO 4387:1991; cigars shall be marked for butt length as per CORESTA conditions (section 3.2.2).

11.3 Cigarettes to be smoked under intense smoking conditions shall be taped as described in section 3.6.

12 SMOKING MACHINE PREPARATION

12.1 Ambient Conditions

12.1.1 The ambient conditions for smoking shall be as those specified in ISO 3308:1991.

12.2 Machine Conditions

12.2.1 The smoking machine specifications to be used for cigarettes, small cigars and cigarette equivalents are as set out in ISO 3308:1991.

12.2.2 The smoking machine specifications to be used for bidis are as set out in ISO 3308:1991 with the following modifications: puff volume, 45 mL; puff frequency, 30 seconds.

12.2.3 The smoking machine specifications to be used for regular and premium cigars are as set out by CORESTA (3.3.2).

13 SAMPLE GENERATION

13.1 Cigarettes, bidis, cigarette equivalents, or cigars shall be smoked under the conditions specified in section 12.2 and TPM shall be collected as specified in ISO 4387:1991.

14 SAMPLE ANALYSIS**14.1 Determination of Tar, Water, Nicotine and Carbon Monoxide**

14.1.1 The determination of the total particulate matter present in the smoke is set out in ISO 4387:1991.

14.1.2 The water content of the total particulate matter shall be determined by gas chromatography as set out in ISO 10362-1:1991.

14.1.3 The nicotine content of the total particulate matter shall be determined by gas chromatography as set out in ISO 10315:1991.

14.1.4 The measurement of carbon monoxide present in the vapour phase of smoke shall be determined by NDIR analyzer as set out in ISO 8454:1995.

14.1.5 The tar content is determined by subtracting the water content (14.1.2) and the nicotine content (14.1.3) from the total particulate matter (14.1.1).

14.2 Calculations

14.2.1 See the appropriate ISO standards referenced in section 13.1.

15 QUALITY CONTROL

15.1 A Laboratory Reagent Blank (LRB) is used to monitor the level of water and nicotine contamination in the reagents and supplies such as glassware and pads. Although nicotine should not be detected in these blanks, there is always some water due to its presence in the extraction solution and the conditioned pad.

15.2 A Laboratory Fortified Blank (LFB) is used to evaluate the extent of potential analyte loss during the extraction process. An LFB should be run whenever there is a question about the validity of results.

15.3 A Laboratory Fortified Matrix (LFM) is used to determine the extent of potential analyte loss as a result of the analysis process and to determine potential matrix effects. An LFM should be run whenever there is a question about the validity of results.

15.4 Method detection limit (MDL)/Limit of Quantitation (LOQ)

This involves the use of either a test material with a low level of the analyte or the lowest standard. The standard deviation is then determined and the MDL is determined to be three times the standard deviation. The LOQ is determined to be ten times the standard deviation.

15.5 Stability of Reagents and Samples

15.5.1 Because of the presence of water vapour in the air, each sample vial should only be injected once and then discarded.

15.5.2 Standards should be wrapped with a laboratory film (such as Parafilm®) and kept in the dark. They are stable for approximately one week. As with the samples, each vial should only be used once for water determination.

15.5.3 Extraction solution is stable but can become contaminated with water over time. For this reason, and to ensure nicotine calibration remains constant, fresh standards for nicotine and water should be made weekly.

16 MODIFICATIONS FOR INTENSE SMOKING CONDITIONS

16.1 Three conditioned cigarettes are to be smoked per port under intense smoking conditions.