

WHO TobLabNet
Official Method
SOP 01

STANDARD OPERATING PROCEDURE FOR INTENSE SMOKING OF CIGARETTES

**Tobacco Free Initiative
Tobacco Laboratory Network (TobLabNet)**



**World Health
Organization**



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**Standard operating procedure for
intense smoking of cigarettes**

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**World Health Organization
Tobacco Laboratory Network**

**Standard operating procedure for method
Intense smoking of cigarettes**

Method:	Intense smoking of cigarettes
Analytes:	Not applicable
Matrix:	Cigarettes
Last update:	April 2012



No machine smoking regimen can represent all human smoking behaviour: machine smoking testing is useful for characterizing cigarette emissions for design and regulatory purposes, but communication of machine measurements to smokers can result in misunderstanding about differences between brands in exposure and risk. Data on smoke emissions 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. Representing differences in machine measurements as differences in exposure or risk is a misuse of testing with WHO TobLabNet standards.

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FOREWORD

This document was prepared by members of the World Health Organization (WHO) Tobacco Laboratory Network (TobLabNet) as a standard operating procedure (SOP) for intense smoking of cigarettes.

INTRODUCTION

In order to establish comparable measurements for testing tobacco products globally, consensus methods are required for measuring specific contents and emissions of cigarettes. The Conference of the Parties to the WHO Framework Convention on Tobacco Control (FCTC) at its third session in Durban, South Africa, in November 2008, recalling its decisions FCTC/COP1(15) and FCTC/COP2(14) on the elaboration of guidelines for implementation of Articles 9 (*Regulation of the contents of tobacco products*) and 10 (*Regulation of tobacco product disclosures*) of the WHO FCTC, noting the information contained in the report of the working group to the third session of the Conference of the Parties on the progress of its work ... requested the Convention Secretariat to invite WHO's Tobacco Free Initiative to ... validate, within five years, the analytical chemical methods for testing and measuring cigarette contents and emissions (FCTC/COP/3/REC/1).

Using the criteria for prioritization set at its third meeting in Ottawa, Canada, in October 2006, the working group on Articles 9 and 10 identified the following contents for which methods for testing and measurement (analytical chemistry) should be validated as a priority:

- nicotine
- ammonia
- humectants (propane-1,2-diol, glycerol (propane-1,2,3-triol) and triethylene glycol (2,2-ethylenedioxybis(ethanol))).

Measurement of these contents will require validation of three methods: one for nicotine, one for ammonia and one for humectants.

Using the criteria for prioritization set at the meeting in Ottawa mentioned above, the working group identified the following emissions in mainstream smoke for which methods for testing and measurement (analytical chemistry) should be validated as a priority:

- 4-(methylnitrosamino)-1-(3-pyridyl)-1-butanone (NNK)
- N-nitrosonornicotine (NNN)
- acetaldehyde
- acrylaldehyde (acrolein)
- benzene
- benzo[a]pyrene
- 1,3-butadiene
- carbon monoxide
- formaldehyde

Measurement of these emissions with the two smoking regimens described below will require validation of five methods: one for tobacco-specific

nitrosamines (NNK and NNN), one for benzo[a]pyrene, one for aldehydes (acetaldehyde, acrolein and formaldehyde), one for volatile organic compounds (benzene and 1,3-butadiene), and one for carbon monoxide.

The table below sets out the two smoking regimens for validation of the test methods referred to above.

Smoking regimen	Puff volume (ml)	Puff frequency	Filter ventilation holes
ISO regimen: ISO 3308; <i>Routine analytical cigarette smoking machine—definitions and standard conditions</i>	35	Once every 60 s	No modifications
Intense regimen: Same as ISO 3308, but modified as indicated	55	Once every 30 s	All ventilation holes must be blocked 100% as described in 12.2 .

This SOP was prepared to describe the procedure for intense smoking of cigarettes.

1 SCOPE

This SOP describes the overall procedures for machine smoking of cigarettes under intense conditions.

Note: Training in use of the smoking machine and other analytical equipment is important for successful operation. People not experienced in operating smoking machines or in using the analytical methods for measuring tobacco product emissions and contents should be trained.

2 REFERENCES

- 2.1** ISO 3308: *Routine analytical cigarette-smoking machine—Definitions and standard conditions*.
- 2.2** ISO 4387: *Cigarettes—Determination of total and nicotine-free dry particulate matter using a routine analytical smoking machine*.
- 2.3** ISO 3402: *Tobacco and tobacco products—Atmosphere for conditioning and testing*.

3 TERMS AND DEFINITIONS

- 3.1** TPM: Total particulate matter
- 3.2** ISO regimen: Parameters used to smoke tobacco products that include a 35-ml puff volume, a 60-s puff interval, 2-s puff duration and no blocking of the filter ventilation holes
- 3.3** Intense Regimen – Parameters used to smoke tobacco products which include 55-ml puff volume, 30-s puff interval, 2-s puff duration and 100% blocking of the filter ventilation holes.
- 3.4** *Tobacco products*: Products entirely or partly made of leaf tobacco as the raw material that are manufactured to be used for smoking, sucking, chewing or snuffing (Article 1(f) of the WHO FCTC)
- 3.5** *Laboratory sample*: Sample intended for testing in a laboratory, consisting of a single type of product delivered to the laboratory at one time or within a specified period

3.6 *Test sample:* Product to be tested, taken at random from the laboratory sample. The number of products taken shall be representative of the laboratory sample.

3.7 *Test portion:* Random sample from the test sample to be used for a single determination. The number of products taken shall be representative of the test sample.

4 METHOD SUMMARY

4.1 All samples are conditioned and marked according to ISO standard procedures.

4.2 Ventilation holes are blocked 100%.

4.3 Cigarettes are smoked according to ISO standard procedures with the exception of puff volume and puff frequency.

5 SAFETY AND ENVIRONMENTAL PRECAUTIONS

5.1 Follow routine safety and environmental precautions, as in any chemical laboratory activity.

5.2 The testing and evaluation of certain products with this test method may require the use of materials or equipment that could be hazardous or harmful to the environment. This document does not purport to address all the safety aspects associated with its use. All persons using this method have the responsibility to consult the appropriate authorities and to establish health and safety practices as well as environmental precautions in conjunction with any existing, applicable regulatory requirements prior to its use.

5.3 Special care should be taken to avoid inhalation or dermal exposure to harmful chemicals. Use a chemical fume hood, and wear an appropriate laboratory coat, gloves and safety goggles when preparing or handling undiluted materials, standard solutions, extraction solutions or collected samples.

6 APPARATUS AND EQUIPMENT

Usual laboratory apparatus, in particular:

6.1 Equipment needed to condition cigarettes as specified in ISO 3402

6.2 Equipment needed to mark butt length as specified in ISO 4387

6.3 Equipment needed to cover ventilation holes for the intense regimen as specified in [section 12.2](#)

6.4 Equipment needed to perform smoking of tobacco products as specified in ISO 3308

6.5 Cellophane tape, 20 mm ($\frac{3}{4}$ ") wide, such as Scotch® tape, (3M, Maplewood, Minnesota, USA)

6.6 Cigarette holders for blocking 100% ventilation holes

7 REAGENTS AND SUPPLIES

All reagents shall be of at least analytical reagent grade unless otherwise noted. When possible, reagents are identified by their Chemical Abstracts Service (CAS) registry numbers.

8 PREPARATION OF GLASSWARE

Clean and dry glassware in a manner to ensure no contamination from residues.

9 PREPARATION OF SOLUTIONS

Not applicable

10 PREPARATION OF STANDARDS

Not applicable

11 SAMPLING

Sampling should be done as described in the specific method SOP

12 CIGARETTE PREPARATION

12.1 Mark cigarettes at a butt length in accordance with ISO 4387.

12.2 Block all ventilation holes, as specified below.

12.2.1 For the intense regimen, block filter ventilation holes completely by applying 20 mm ($\frac{3}{4}$ ") wide cellophane tape [6.5] around the entire circumference of the cigarette.

12.2.2 Measure out a length of cellophane tape of 50–55 mm.

12.2.3 Attach the cut end of the tape parallel to the long axis of the cigarette with the side of the tape within 1 mm of the mouth end of the filter (see Figure 1).

12.2.4 Carefully wrap the tape around the filter to ensure complete bonding to the paper with no wrinkles or air holes. If wrinkles or air holes appear, reject the sample and do not include it in the analysis.

12.2.5 The tape should circle the cigarette twice, with a small overlap of less than 5 mm (see Figure 2).

12.2.6 The tape should not extend beyond the mouth end of the filter.

As an alternative to tape, special holders [6.6] for blocking 100% of ventilation holes can be used.

12.3 Condition all cigarettes to be smoked in accordance with ISO 3402.

13 PREPARATION OF THE SMOKING MACHINE**13.1 Ambient conditions**

The ambient conditions for smoking are specified in ISO 3308.



Figure 1



Figure 2

13.2 Machine specifications

Follow ISO 3308 machine specifications, except the following:

- 13.2.1** For the intense regimen, set the smoking machine to draw a puff volume of 55 ± 0.1 ml.
- 13.2.2** For the intense regimen, set the smoking machine to take puffs at a frequency of 30 s.
- 13.2.3** Programme each smoking run to end when the cigarette has burnt down to the mark placed earlier [12.1].

14 SAMPLE GENERATION

Smoke a sufficient amount of product on the specified smoking machine such that breakthrough does not occur.

- 14.1** Smoke the test samples as specified in ISO 4387, and collect the analyte of interest as described in the specific SOP
- 14.2** Include at least one reference test sample for quality control.
- 14.3** When testing sample types for the first time, evaluate breakthrough. The number of cigarettes might have to be adjusted to prevent breakthrough. During determination of tar, nicotine and carbon monoxide, breakthrough of cigarette smoke occurs at TPM levels exceeding 600 mg for a 92-mm filter pad or 150 mg for a 44-mm filter pad. If breakthrough occurs, the number of cigarettes smoked onto each pad must be decreased. The breakthrough of the filter pads or other collection devices might differ, however, depending on the analyte of interest.

15 SAMPLE PREPARATION

Not applicable.

16 SAMPLE ANALYSIS

Not applicable.

17 DATA ANALYSIS AND CALCULATIONS

Not applicable.

18 SPECIAL PRECAUTIONS

None.

19 DATA REPORTING

Data will be reported as described in the specific SOP

20 QUALITY CONTROL

Quality control will be performed as described in the specific SOP

21 METHOD PERFORMANCE SPECIFICATIONS

Not applicable.

22 REPEATABILITY AND REPRODUCIBILITY

Not applicable.

23 BIBLIOGRAPHY

- 23.1** ISO 10185: Tobacco and tobacco products—Vocabulary.

