TECHNICAL REPORT

ISO/TR 19478-2

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ISO and Health Canada intense smoking parameters —

Part 2: Examination of factors contributing to variability in the routine measurement of TPM, water and NFDPM smoke yields of cigarettes

Paramètres de fumage ISO et Santé Canada Intense —

Partie 2: Examen des facteurs contribuant à la variabilité des mesures de routine de MPT, d'eau et de MPAEN dans la fumée de cigarette

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Foreword

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The committee responsible for this document is ISO/TC 126, *Tobacco and tobacco products*.

ISO/TR 19478 consists of the following parts, under the general title *ISO and Health Canada intense smoking parameters*:

— *Part 1: Results of an international machine smoking study*

— *Part 2: Examination of factors contributing to variability in the routine measurement of TPM, water and NFDPM smoke yields of cigarettes*

Introduction

ISO/TC 126 Working Group 10 (WG 10) was established by ISO/TC 126 in 2007 in response to a New Work Item Proposal by the British Standards Institution (BSI) for the development of a new regime for the machine smoking of cigarettes that was more intense than the then current ISO 3308:2000, and a subsequent questionnaire sent to TC 126 members. Twenty out of 26 members of ISO/TC 126 voted in favour of the following option:

"*to install a Working Group 10 dealing with an 'Intense Smoking Regime' which shall start with the preparatory work. WHO is invited to participate with their technical experts. No draft Standard is expected to be presented by this group until the future method proposal of WHO has been taken into consideration*".

The third session of the Conference of the Parties (COP) to the World Health Organization (WHO) Framework Convention on Tobacco Control Durban, South Africa, 17 to 22 November 2008, requested the Convention Secretariat to invite the WHO's Tobacco Free Initiative (TFI) to undertake the following task:

"*validate, within five years, the analytical chemical methods for testing and measuring the cigarette contents and emissions identified as priorities in the progress report of the working group 1 using the two smoking regimens set out in paragraph 18 of that report, and inform the Conference of the Parties through the Convention Secretariat on a regular basis of the progress made.*"

The two smoking regimens were specified in paragraph 18 of the report of the COP working group (FCTC/COP/3/6) as follows:

Smoking regimen	Puff volume (ml)	Puff frequency	Ventilation holes
ISO 3308:2000, Routine analytical cigarette-smoking machine — Definitions and standard conditions	35	Once every 60 s	No modifications
Same as ISO 3308:2000 but modified as indicated.	55	Once every 30 s	All ventilation holes must be blocked with Mylar adhesive tape.

The two regimes were those specified in ISO 3308 and by Health Canada in Method T-115. At the early meetings of WG 10, some new human smoking studies were presented and are included in Annex A for completeness of reporting, but WG 10 never considered the correlation with machine smoking regimes in detail as this brief had previously been given to ISO/TC 126/WG 9 and WG 9 had produced a comprehensive report, ISO/TR 17219:2013.

The WHO TFI requested the WHO Tobacco Laboratory Network (TobLabNet) to carry out the practical work of validating the two smoking regimes. In 2008, TobLabNet organized and carried out a collaborative test to measure the tar, nicotine and carbon monoxide yields of cigarettes when using the Health Canada Intense (HCI) regime. The collaborative test involved 14 laboratories smoking five products (three reference cigarettes/monitor test pieces and two commercial products). Details of this collaborative were supplied to ISO/TC 126/WG 10.

WG 10 had expressed a willingness from its inception to participate with the WHO groups in the development of an intense smoking regime but had not been invited to do so. It, therefore, decided at its fifth meeting in December 2009 to undertake a collaborative study to measure the tar, nicotine and carbon monoxide yields of cigarettes using both the ISO 3308:2000 and Health Canada intense smoking regimes. A steering group was established and the laboratory work was carried out in 2010 involving 35 laboratories smoking 10 products (eight commercial and two reference cigarettes/monitor test piece). A final report on the study was approved by WG 10 and subsequently converted to a Technical Report, ISO/TR 19478-1. ISO/TR 19478-1 provided a basic analysis of the study data, drawing conclusions about the possible sources of the increased variability associated with the HCI regime.

These conclusions provided the basis for the additional studies reported here and instigated to provide a more complete understanding of how the smoke yield changes with increasing smoking intensity.

ISO and Health Canada intense smoking parameters —

Part 2:
Examination of factors contributing to variability in the routine measurement of TPM, water and NFDPM smoke yields of cigarettes

1 Scope

This part of ISO/TR 19478 extends the analysis reported in ISO/TR 19478-1:2014 and reports additional studies focused on the conclusions i) and j) from that Technical Report. It identifies and assesses factors impacting on the measurement of smoke TPM, NFDPM, nicotine, water, and carbon monoxide yields when increasing the intensity of the puffing regime from that specified in ISO 3308:2000 to the regime specified in Health Canada Method T-115.

2 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

2.1
cigarette coal
carbonised burning tip of a tobacco rod

2.2
ISO regime
puffing regime when taking one puff of 35 ml volume and 2 s duration every 60 s as defined in ISO 3308:2000

2.3
Health Canada Intense regime
HCI regime
puffing regime, first described by Health Canada, when taking one puff of 55 ml volume and 2 s duration every 30 s with 100 % of the ventilation zone on the cigarette filter blocked

2.4
linear (smoking) machine
smoking machine complying with the requirements of ISO 3308:2000 with each cigarette holder directly coupled to a CFH (smoke trap)

Note 1 to entry: The CFH is coupled via a port to its own suction mechanism and held in a fixed position while each cigarette is smoked. The most common configuration has 20 ports in line.

2.5
rotary (smoking) machine
smoking machine complying with the requirements of ISO 3308:2000 with each cigarette holder coupled sequentially via a port to a single CFH (smoke trap) and suction mechanism

Note 1 to entry: The most common configuration has 20 ports on a carousel sharing a single CFH and suction mechanism.

2.6
Massachussetts regime
MA regime
puffing regime, used in Massachusetts, USA, when taking one puff of 45 ml volume and 2 s duration every 30 s with 50 % of the ventilation zone on the cigarette filter blocked

2.7
option B regime
puffing regime proposed by ISO/TC 126 Working Group 9 when taking one puff of 60 ml volume and 2 s duration every 30 s with 50 % of the ventilation zone on the cigarette filter blocked

3 Abbreviated terms

CFH	Cambridge Filter Holder
CFP	Cambridge Filter Pad
COP	Conference of the Parties to the World Health Organization Framework Convention on Tobacco Control
NFDPM or tar	Nicotine Free Dry Particulate Matter
TNCO	tar, nicotine and carbon monoxide where tar is specifically nicotine free dry particulate matter (NFDPM)
TPM	total particulate matter
RH	relative humidity
SVD	Saturated Vapour Density
TobLabNet	World Health Organization Tobacco Laboratory Network
WHO	World Health Organization
ΔT	Reduction in the time to smoke a cigarette due to puffing, calculated as: (Time to smoulder burn the cigarette) - (Time to burn the cigarette when puffing)

4 Principle

Following the analysis of the data from the TNCO study (ISO/TR 19478-1), WG 10 decided to review the impact of increasing puffing intensity on the measurement of TPM, NFDPM, nicotine, water and carbon monoxide yields from cigarettes with particular emphasis on differences resulting from the use of rotary or linear smoking machines. The Ad Hoc Group (AHG) of WG 10 was set up to give focus to the review process. The membership of the AHG necessarily included representatives of the manufacturers of smoking machines as well as those WG 10 members who wished to be actively involved in further studies. The review was carried out with particular reference to the conclusions from the WG 10 TNCO study which compared smoke yield data for the ISO regime, as specified in ISO 3308:2000 and ISO 4387, with that for the HCI regime, as specified in the Health Canada Method T-115. The AHG first identified a number of differences in design features between rotary and linear machines with the potential to alter the collection of smoke condensate and so increase the variation in smoke yield measurements. Individual AHG members then used their expertise to create protocols to evaluate these factors in their respective laboratories.

Conclusions i) and j) of ISO/TR 19478-1:2014 provided the focus for the work of the AHG, although other issues were also considered. Clauses 5 and 6 provide a summary of the understanding developed within WG 10 and the AHG of the issues identified in conclusions i) and j).

The studies providing the background to this part of ISO/TR 19478 are listed in Annex A together with a summary of the content of each and the meeting at which they were presented. Annex B provides a list of all meetings of WG 10 and the Ad Hoc Group of WG 10 until the end of 2013.

5 The influence of smoking intensity on the yield and composition of cigarette smoke

5.1 General

The results of the collaborative study described in ISO/TR 19478-1 had shown the reproducibility of the NFDPM yield measurements from 10 products collected under the HCI smoking regime and measured in many laboratories, were worse than when using the ISO regime. This finding was supported by other collaborative studies run by the WHO TobLabNet and by CORESTA[1] as summarized in A.11. The range of products to which this conclusion can be applied was widened to include products of 16 mm to 18 mm in circumference (super slims) in a further small study (A.20). The ISO WG 10 TNCO study data set has also been used to discuss the problems with statistical outlier analysis when combining data from rotary and linear machines which give different measured water yields.[2][3]

Apart from the increased puff volume and frequency specified for the HCI regime, it is also necessary to block 100 % of the ventilation on the cigarette filter. The Health Canada Method T-115 specifies overwrapping the filter with “invisible” tape (adhesive cellophane tape) to block the ventilation holes in the cigarette filter but special cigarette holders have also been developed to achieve the same outcome. The overwrapping of filters with adhesive tape was investigated (A.19) to eliminate it as a potential cause of the increased measurement variability and another study (A.15) showed that taping and using specially designed vent blocking cigarette holders gave similar yields.

In order that the increased yield resulting from the much increased smoking intensity of the HCI regime does not overload the CFP when using a linear machine with the 44 mm CFP (ISO 4387 specifies a maximum load of 150 mg), three cigarettes are smoked per smoking run rather than five. Another study (A.23) confirmed the 150 mg limit for the ISO regime but found it could be doubled when using the HCI regime.

5.2 A review of information relevant to conclusion i) of ISO/TR 19478-1:2014

Conclusion i) stated,

“As expected from previous studies, the water yields were disproportionately higher than other measured smoke parameters under the HCI regime. This water effect is a contributory factor to the increases in R values, but the magnitude of its contribution is uncertain.”

Conclusion i) set the need to better understand how the smoke yield changed with smoking intensity, both in magnitude and composition. In particular, to investigate the cause of greatly increased smoke water content.

It can be seen (Table 1) from the TPM and water yields for the 10 products tested in the WG 10 study that there is a consistency to the proportions of nicotine and water in the TPM for all 10 products and both smoking regimes. The most important feature of the data is the increase in the average water yield from approximately 10 % of the TPM for the ISO regime to almost 30 % for the HCI regime. It is by far the most abundant component of the TPM with nicotine being only 5 % of the TPM for the HCI regime. This finding signals a potential measurement problem using the normal ISO procedures for TPM and water measurement as the collection system, the CFP held in the CFH, is specifically designed for collecting particulate material. If a major proportion of the smoke water is in the vapour phase, the collection efficiency of the CFP/CFH unit will be compromised, as will the subsequent measurement of the smoke water yield.

Table 1 — TPM, water and nicotine yields under the ISO and HCI smoking regime

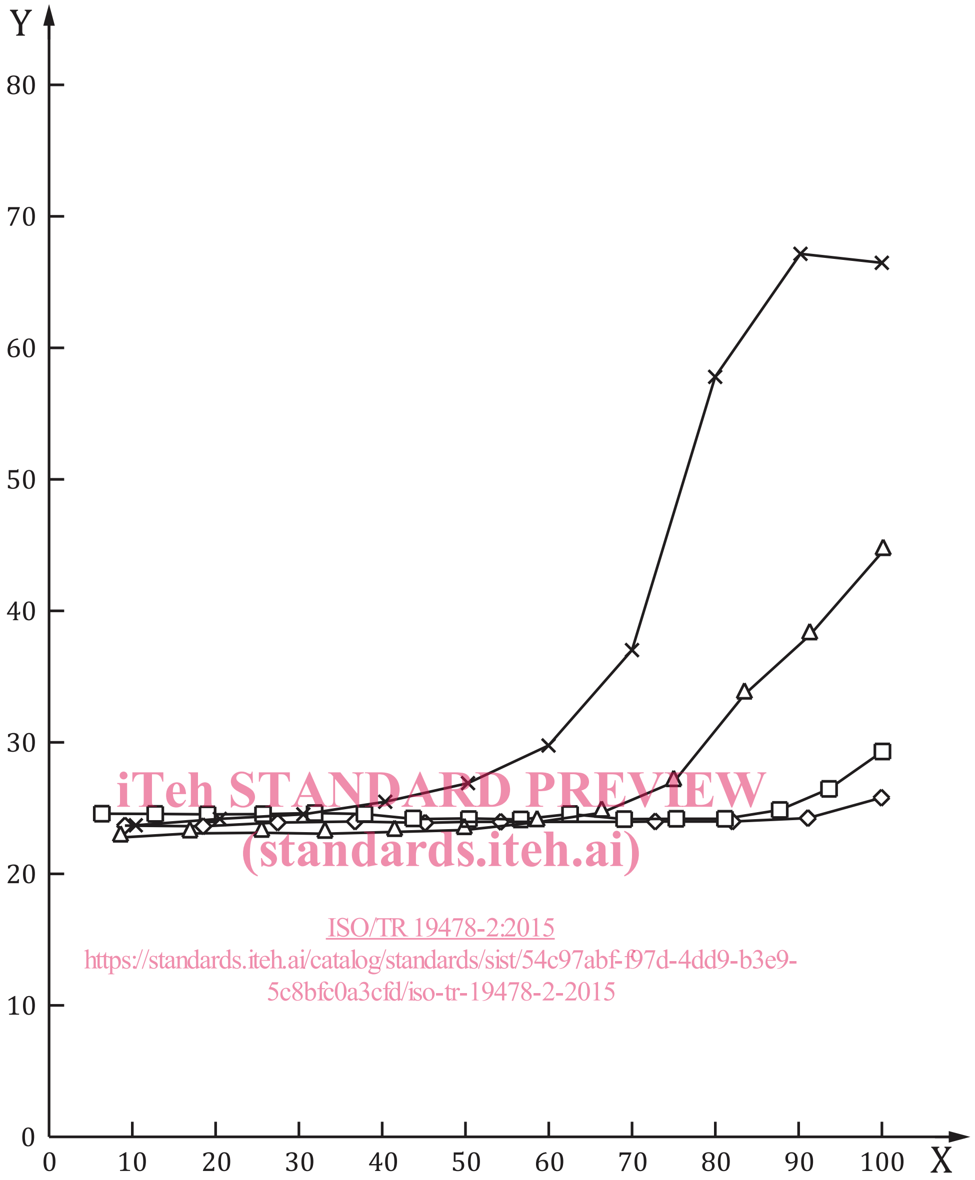
Product code	ISO regime					HCI regime				
	TPM	Water		Nicotine		TPM	Water		Nicotine	
	mg/cig	mg/cig	% of TPM	mg/cig	% of TPM	mg/cig	mg/cig	% of TPM	mg/cig	% of TPM
A	1,28	0,10	7,5	0,11	8,4	25,41	7,35	28,9	1,27	5,0
B	5,21	0,40	7,6	0,39	7,5	31,26	9,39	30,0	1,34	4,3
C	10,81	1,26	11,6	0,68	6,3	39,84	11,87	29,8	1,79	4,5
D	10,03	0,93	9,2	0,82	8,1	39,07	11,19	28,6	2,11	5,4
E	11,54	1,10	9,5	0,66	5,7	29,71	6,06	20,4	1,41	4,7
F	10,65	1,12	10,5	0,75	7,1	43,69	13,42	30,7	2,07	4,7
G	12,05	1,51	12,5	0,83	6,9	43,43	14,16	32,6	2,09	4,8
H	11,08	1,06	9,6	0,67	6,0	40,09	11,47	28,6	1,68	4,2
I	2,08	0,19	9,1	0,15	7,4	27,05	9,03	33,4	0,99	3,7
J	17,30	1,69	9,7	1,37	7,9	41,18	10,11	24,5	2,68	6,5
Mean	**9,20**	**0,93**	**9,7**	**0,64**	**7,1**	**36,07**	**10,40**	**28,8**	**1,74**	**4,8**

The understanding of subsequent subclauses will be aided by an understanding of the nature of cigarette smoke and the formation process in the burning tobacco rod. After lighting, the tobacco rod forms the coal, a carbonised section at its tip, which then promotes the continuous burning of the remaining tobacco through the heat liberated from the oxidation of the carbon. Studies[4] have established thermal profiles in the region of the cigarette coal which show that the temperature at the char line on the cigarette paper is approximately 450 °C with the tobacco temperature rapidly dropping to about 300 °C within 3 mm to 4 mm. The tobacco in this region is denuded of volatile components which evaporate and migrate along the tobacco rod away from the hot coal. They then cool with some condensing to form a smoke aerosol in equilibrium with the remaining vapour cloud. At the same time, some smoke components will condense onto the tobacco as well as diffusing through the cigarette paper to be lost to the surrounding environment.

During puffing, volatile compounds are transferred and deposited further along the tobacco rod. This deposited material is partly lost by diffusion through the cigarette paper between puffs with the remaining material accumulating with successive puffs until the char line reaches it. If this occurs during a puff, the material becomes part of the smoke yield from that puff so increasing the yield from the puff. This transfer process has been demonstrated by following changes in the density of the tobacco rod during smoking.[5]

5.3 Puff by puff smoke temperature measurements

The first of a number of relevant studies (A.4) presented to WG 10 provided temperature data for individual puffs as a cigarette was smoked. The data was for peak temperatures at two positions in the cigarette filter and for four puffing regimes, ISO, HCI, MA and Option B from the report of ISO/TC 126/WG 9 (ISO/TR 17219:2013) The temperatures measured 5 mm from the mouth end of the filter of a 1 mg tar cigarette (ISO) are shown in Figure 1.



Key

X proportion of cigarette burnt (%)

Y maximum temperature in filter (°C)

—◇— ISO regime

—□— MA regime

—△— WG 9 (B) regime

—×— HCI regime

Figure 1 — Temperatures 5 mm from mouth end of cigarette filter (A.4)

Figure 1 shows that the temperature of the smoke as it leaves the cigarette filter is near the ambient temperature of 22 °C for the early puffs. This indicates the heat exchange with the tobacco and filter material, together with mixing with air drawn into the cigarette through the cigarette paper and filter, is sufficient to cool the smoke to the temperature of the test environment.

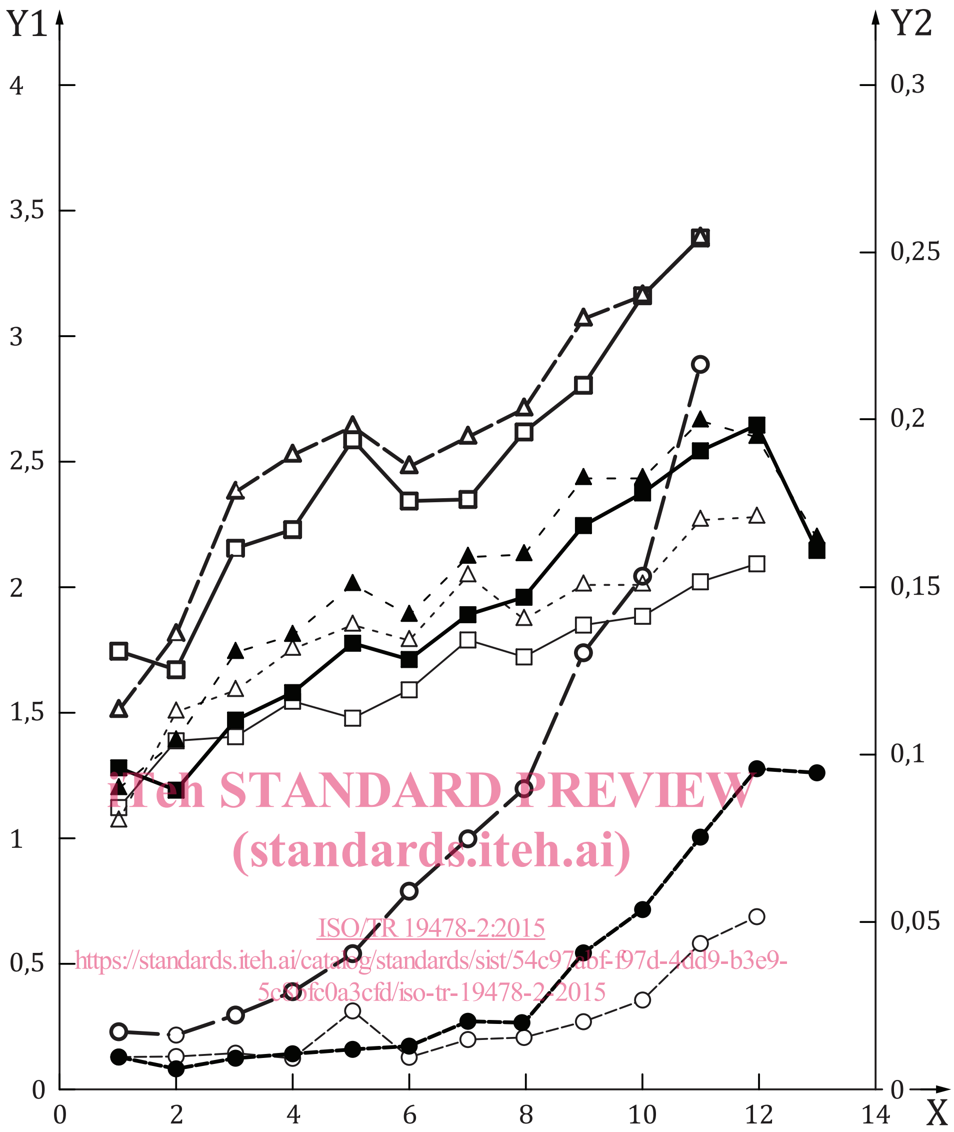
As further puffs are taken and the tobacco rod burns down, the temperature rises above 22 °C. The more intense the smoking regime, the earlier this occurs during the smoking process and the greater is the increase in temperature above the ambient level.

These temperature measurements are highly significant when considering the formation of the smoke aerosol. Smoke is initially formed as a complex mixture of hot vapours and gases just behind the cigarette coal. It then becomes an aerosol as it is drawn through the tobacco rod and cools. The partitioning of compounds between the vapour and particulate phases will continue to change until it reaches a fixed temperature. Since the collection and measuring system is held at 22 °C, it is desirable for the smoke to leave the cigarette at this temperature to prevent further changes during collection. This will not happen when using very intense smoking regimes since the temperature measurements in Figure 1 indicate that the smoke from the later puffs is considerably above 22 °C.

5.4 Puff by puff smoke yields

It has been shown that the temperature of smoke leaving a cigarette can be above the ambient temperature for the later puffs, particularly when the intensity of the smoking regime is increased. As the formation of smoke from a vapour cloud behind the cigarette coal is primarily driven by the temperature change, it seems probable that the smoke composition will also change during the smoking of a cigarette. A change in smoke composition linked to its exit temperature from the cigarette should be apparent from reviewing the puff by puff smoke yield. Suitable single puff yield data[7] was made available to the Ad Hoc Group. The smoke yield measurements were for the 1R4F reference cigarette using a smoking regime with puffs of 60 ml volume and 2 s duration being taken every 30 s. The level of ventilation at the cigarette filter was also varied by blocking 0 %, 50 % or 100 % of the ventilation holes.

The NFDPM, nicotine and water yields for individual puffs are given in Figure 2. The data in Figure 2 show that the yields of the three smoke components increase both as the cigarette is consumed during smoking and also as a greater proportion of the filter ventilation holes are blocked. The general increase in smoke yield as the tobacco rod is burnt is expected and due to reduced filtration and ventilation in the shorter tobacco rod. The rate of increase in yield for NFDPM and nicotine is similar for successive puffs and for each of the three levels of vent blocking. The water yields show a distinctly different pattern with the rate of increase being much greater as the cigarette is consumed, and the rate is much increased as a greater percentage of the vents are blocked.



Key

X puff number

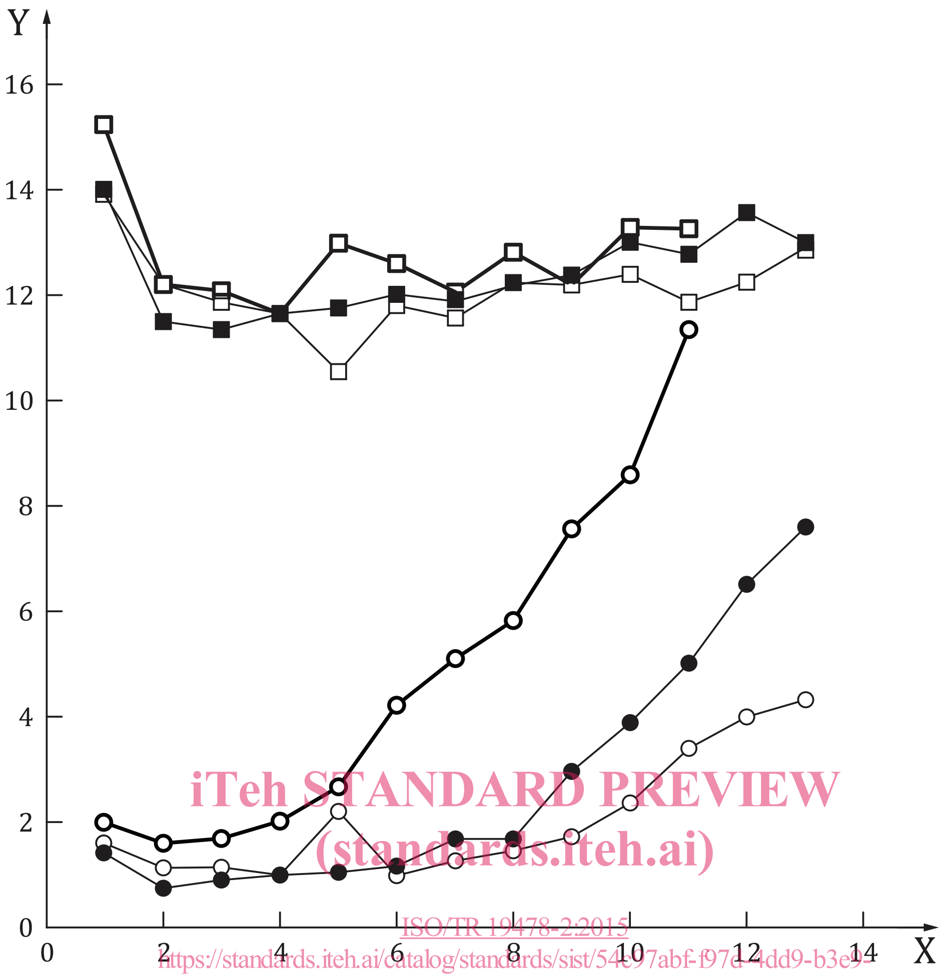
Y1 NFDPM and water yield (mg/puff)

Y2 nicotine yield (mg/puff)

- NFDPM-0 % VB
- water-0 % VB
- nicotine-0 % VB
- NF DPM-50 % VB
- water-50 % VB
- nicotine-50 % VB
- NF DPM-100 % VB
- water-100 % VB
- nicotine-100 % VB

Figure 2 — Puff by puff yields from 1R4F reference cigarette[7]

These patterns of change are more clearly shown by taking the ratio of the NFDPM and water yields to those for nicotine as shown in Figure 3.



Key

X puff number

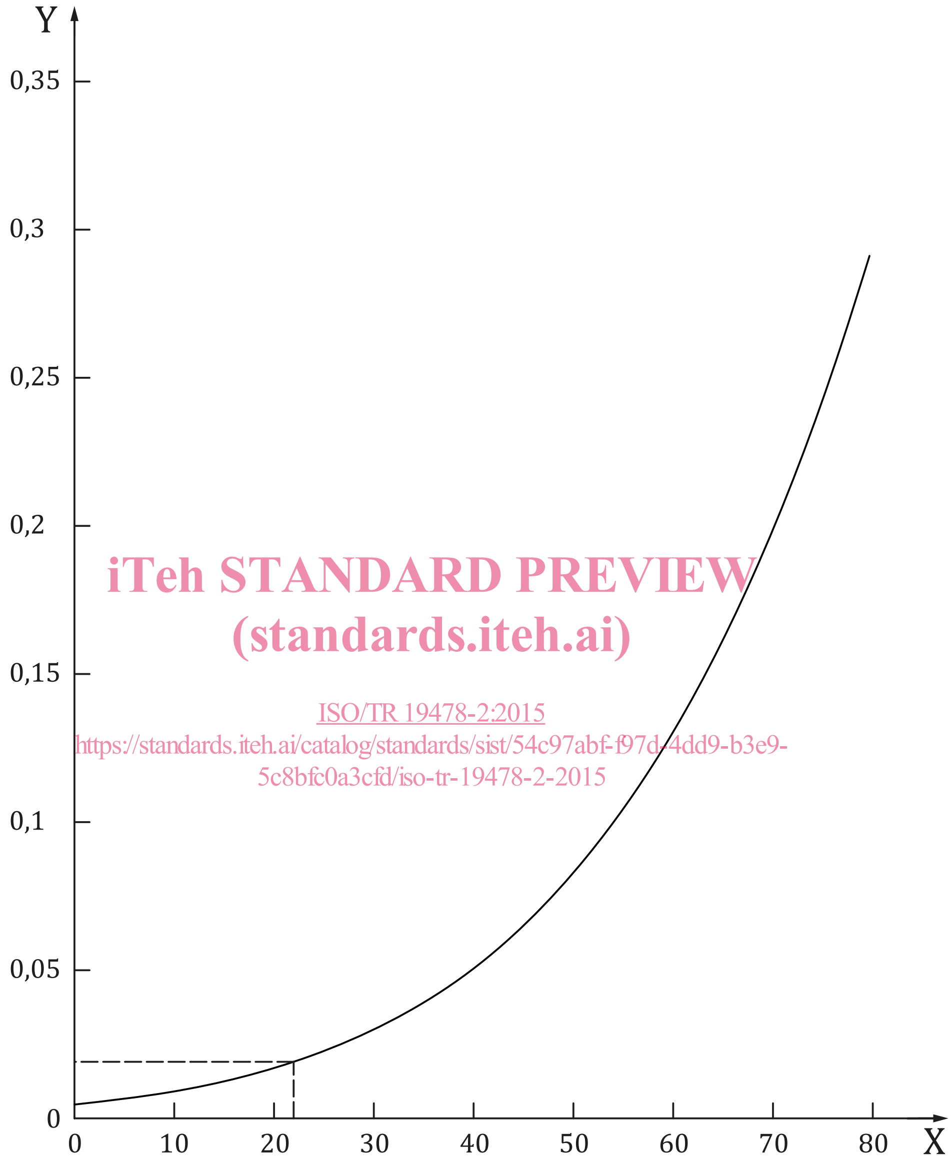
Y yield as a ratio to nicotine

- water-0 % VB
- NFDPM-0 % VB
- water-50 % VB
- NF DPM-50 % VB
- water-100 % VB
- NF DPM-100 % VB

Figure 3 — Puff by puff yields of NFDPM and water as a ratio to nicotine yield[7]

The NFDPM to nicotine ratios in Figure 3 show that the relative yield of these smoke components is little changed during the smoking of a cigarette or by decreasing the filter ventilation. In contrast, the water to nicotine ratios show that water yields are relatively unchanged for the initial puffs but then increase rapidly. The water yield also increases as the filter ventilation is reduced by vent blocking.

The puff by puff water yields cannot be directly correlated with the temperature measurements in 5.3 as the cigarettes tested were not of the same design, but a mechanism to link the increased smoke water content with an increase smoke temperature can be proposed. Tobacco naturally absorbs water from the environment to a level of approximately 10 % by weight when stored at a temperature of 22 °C and a 60 % relative humidity. When a cigarette is lit, the heat from the coal drives a cloud of volatile tobacco components down the tobacco rod where they cool and condense to form the smoke aerosol. Water vapour will be the biggest vapour phase smoke component due to the release of the water absorbed by tobacco and the additional amount produced as a combustion product. Previously unpublished work (A.9) measured 200 mg to 300 mg of water per cigarette in the sidestream smoke from 84 mm long filter cigarettes. With such large amounts of water present when smoke is generated, it seems inevitable that the concentration will remain high, at or near the saturated vapour density (SVD) of water, as it cools continuously while travelling through the tobacco rod. Increasing puffing intensity will increase the heat transfer from the cigarette coal into the tobacco rod as well as reducing the residence time of the smoke. As a consequence, the smoke will cool to a lesser extent and leave the cigarette at a higher temperature. As shown in Figure 4, the SVD increases at a much greater rate than the temperature so allowing the smoke to carry much greater quantities of vapour phase water.



Key

X temperature °C

Y saturated vapour density mg/ml

NOTE SVD at 22 °C = 0,0194 mg/ml.

Figure 4 — Change in the saturated vapour density of water with temperature

Although the temperature data from 5.3 cannot be directly correlated with the puff by puff yields discussed in this clause, it is of interest to calculate the temperatures necessary to produce the measured water yields in order to compare with those in 5.3. The relationship between temperature and SVD in Figure 4 can be used to convert the puff by puff water yields in Figure 2 to equivalent temperatures after the yields are converted to concentrations in mg/ml by dividing by the puff volume. When calculating the temperatures, it is assumed that the water vapour will always be present as a