



DETERMINATION OF AEROSOL DELIVERIES OF THS 2.2 STICKS WHEN RE-USED

PROGRAM NAME	PRODUCT DEVELOPMENT
PROJECT NAME	THS 2.2 PRODUCT DEVELOPMENT
WBS NUMBER	J-1002RDA0016/0001/0001
OWNER & APPROVER	CYRIL JEANNET
AUTHOR	RITA HAJDU SCHENK
DATE OF ISSUE	16-Aug-2017

Confidentiality Statement

Certain data and information contained in this document constitute trade secrets and confidential commercial information. The legal protections that apply to trade secrets and confidential information are hereby claimed under the applicable provisions of law. No part of this document may be publicly disclosed without the written consent of Philip Morris International Inc. of which Philip Morris Products SA is an affiliate.



Contents

1 Introduction	4
2 Methods.....	4
2.1 Design.....	4
2.2 Samples	5
2.3 Procedure.....	5
2.3.1 Sample Conditioning.....	5
2.3.2 Aerosol Generation	5
2.3.3 Analysis.....	6
2.4 Instruments and Materials.....	6
2.5 Data Processing.....	6
3 Results	6
3.1 Determination of nicotine, water, glycerin, TPM, NFDPM, CO and triacetin	6
3.2 Determination of phenolic compounds	9
4 Conclusions.....	11
5 Abbreviations	11
6 Review and Approval	12
7 Document version	12

Confidentiality Statement

Certain data and information contained in this document constitute trade secrets and confidential commercial information. The legal protections that apply to trade secrets and confidential information are hereby claimed under the applicable provisions of law. No part of this document may be publicly disclosed without the written consent of Philip Morris International Inc. of which Philip Morris Products SA is an affiliate.



Table 1: Design of aerosol collection of the samples on the smoking machine	4
Table 2: Nicotine, water, glycerin, TPM, NFDPM, CO and triacetin on fresh sticks	7
Table 3: Nicotine, water, glycerin, TPM, NFDPM, CO and triacetin on re-used sticks	7
Table 4: Phenol, o-cresol, m-cresol, p-cresol, catechol, resorcinol and hydroquinone on fresh sticks	9
Table 5: Phenol, o-cresol, m-cresol, p-cresol, catechol, resorcinol and hydroquinone re-used sticks	9
Figure 1: Nicotine, water, glycerin, TPM, NFDPM, CO and triacetin	8
Figure 2: Phenol, o-cresol, m-cresol, p-cresol, catechol, resorcinol and hydroquinone	10

Confidentiality Statement

Certain data and information contained in this document constitute trade secrets and confidential commercial information. The legal protections that apply to trade secrets and confidential information are hereby claimed under the applicable provisions of law. No part of this document may be publicly disclosed without the written consent of Philip Morris International Inc. of which Philip Morris Products SA is an affiliate.



1 Introduction

The objective of this study was to:

- Determine ISO parameters, glycerin and phenols yields in aerosol of THS 2.2 sticks when re-used a second time.
- Compare deliveries of re-used sticks with the fresh stick.

Basic aerosol parameters (TPM, nicotine, water, CO, NFDMP, glycerin and triacetin) were selected to evaluate the performance and overheating of THS 2.2 sticks when re-used a second time. The level of phenolic compounds were as well determined as indicator of the aerosol filtration efficiency of the PLA and cellulose acetate mouth piece filter combination, as the former tends to melt during product use.

2 Methods

2.1 Design

Four replicates of heat stick of B-12480 were generated on an aerosol collection plan on four different ports of a KC linear smoking machine.

The smoked sticks were kept and re-used on a second aerosol collection plan on the same machine and same ports ([Table 1](#)).

Table 1 – Design of aerosol collection of the samples on the smoking machine

Port	Replicate	Aerosol collection plan 1	Aerosol collection plan 2
		Sample ID	Sample ID
x	1	B-13680	Used sticks of B-13680
y	2	B-13680	Used sticks of B-13680
z	3	B-13680	Used sticks of B-13680
w	4	B-13680	Used sticks of B-13680

The following compounds were analyzed in the aerosol of the two samples:

- TPM, nicotine, water, CO, NFDMP, glycerin and triacetin
- phenol, o-cresol, m-cresol, p-cresol, hydroquinone, resorcinol and catechol

Confidentiality Statement

Certain data and information contained in this document constitute trade secrets and confidential commercial information. The legal protections that apply to trade secrets and confidential information are hereby claimed under the applicable provisions of law. No part of this document may be publicly disclosed without the written consent of Philip Morris International Inc. of which Philip Morris Products SA is an affiliate.



2.2 Samples

- Batch of heat stick pack: B-12480 (DII Ron)
- Batch of device: B-13022

2.3 Procedure

2.3.1 Sample Conditioning

Prior to smoking, 3R4F monitor cigarettes were conditioned following ISO standard 3402 (1999), at least 48h at target conditions of $22\pm 1^{\circ}\text{C}$ and $60\pm 3\%$ RH according to the following instructions (WKIs):

- PMI_RD_WKI_000489 Reception and preparation of test and reference item
- PMI_RD_WKI_000380 Préparation des moniteurs pour le fumage
- PMI_RD_WKI_000399 Blocage de la ventilation du papier de bout des cigarettes

2.3.2 Aerosol Generation

The aerosol generation and sample analyses were conducted by Product Testing labs.

Aerosol generation was conducted at $22\pm 2^{\circ}\text{C}$ and $60\pm 5\%$ RH in the Aerosol Collection Lab applying Health Canada (HC) smoking regimen:

- Puff volume: 55 mL
- Puff duration: 2.0 s
- Puff interval: 30 s
- Number of puffs: 12
- Puff profile: bell shaped

Aerosol generation was performed according to the following work instructions:

- PMI_RD_WKI_000530 Trappage en phase particulière pour la détermination des constituants de l'aérosol

Confidentiality Statement

Certain data and information contained in this document constitute trade secrets and confidential commercial information. The legal protections that apply to trade secrets and confidential information are hereby claimed under the applicable provisions of law. No part of this document may be publicly disclosed without the written consent of Philip Morris International Inc. of which Philip Morris Products SA is an affiliate.



- PMI_RD_WKI_000078 Trappage des phénols, acétamides et acrylamides sur les produits non conventionnels (R2540.M052)
- PMI_RD_WKI_000858 Nombre d'articles à fumer et répliques selon le type de fumage et de machine à fumer (R2540.F0001)

2.3.3 Analysis

The generated aerosols have been analyzed according the following WKIs:

- PMI_RD_WKI_000966 Analyse de l'eau, la nicotine et la triacétine dans les condensats de fumée par GC-FID/TCD
- PMI_RD_WKI_000990 Analyse de la glycérine et du menthol dans les condensats de fumée par GC-FID
- PMI_RD_WKI_000379 Analysis of 7 phenolic compounds in aerosol

2.4 Instruments and Materials

As detailed in the appropriate WKIs.

2.5 Data Processing

Validated excel files were used for data treatments as described in the respective WKIs.

Results are available in the SDMS and RDLIMS under project: RLS-ZRH-2015-23.

3 Results

3.1 Determination of nicotine, water, glycerin, TPM, NFDPM, CO and triacetin

Results are depicted in [Table 2](#) and [Table 3](#) and [Figure 1](#).

Confidentiality Statement

Certain data and information contained in this document constitute trade secrets and confidential commercial information. The legal protections that apply to trade secrets and confidential information are hereby claimed under the applicable provisions of law. No part of this document may be publicly disclosed without the written consent of Philip Morris International Inc. of which Philip Morris Products SA is an affiliate.



Table 2: Nicotine, water, glycerin, TPM, NFDPM, CO and triacetin on fresh sticks

		B-12480_Comparability DII Ron batch						
		Nicotine	Water	Glycerin	TPM	NFDPM	CO	Triacetin
		mg/stick						
AVG		1.25	40.2	4.9	54.0	12.6	0.69	0.56
Median		1.24	40.4	5.0	54.3	12.2	0.62	0.56
stdev		0.05	1.9	0.4	1.3	1.9	0.19	0.03
n		4	4	4	4	4	4	4
Uncertainty of mean		0.08	3.0	0.6	2.1	3.1	0.31	0.04
CV		4.2%	4.7%	8.1%	2.5%	15.3%	28.4%	4.7%
Uncertainty of indiv.		0.17	6.02	1.25	4.25	6.12	0.62	0.08
LIMS no.	Replicate							
1381011	rep 1	1.24	42.2	5.1	54.5	11.0	0.62	0.57
	rep 2	1.20	39.9	4.3	52.2	11.1	0.53	0.54
	rep 3	1.24	37.7	4.9	54.0	15.0	0.97	0.53
	rep 4	1.33	40.8	5.2	55.4	13.3	0.62	0.59

Table 3: Nicotine, water, glycerin, TPM, NFDPM, CO and triacetin on re-used sticks

		Re-used B-12480_Comparability DII Ron batch						
		Nicotine	Water	Glycerin	TPM	NFDPM	CO	Triacetin
		mg/stick						
AVG		0.21	5.4	0.6	6.7	1.1	0.37	0.12
Median		0.21	5.5	0.6	6.8	1.1	0.35	0.13
stdev		0.02	0.3	0.0	0.3	0.1	0.05	0.02
n		4	4	4	4	4	4	4
Uncertainty of mean		0.03	0.5	0.0	0.5	0.2	0.07	0.04
CV		9.6%	5.5%	5.1%	5.1%	10.6%	12.1%	20.5%
Uncertainty of indiv.		0.06	0.94	0.09	1.09	0.38	0.14	0.08
LIMS no.	Replicate							
1381014	rep 1	0.19	5.0	0.5	6.2	1.1	0.35	0.12
	rep 2	0.22	5.6	0.6	6.8	1.0	0.35	0.13
	rep 3	0.18	5.4	0.6	6.8	1.2	0.44	0.08
	rep 4	0.22	5.6	0.6	7.0	1.2	0.35	0.14

Nicotine, water, glycerin, TPM, NFDPM, CO and triacetin deliveries are much lower on the re-used sticks compared to fresh sticks.

In the re-used sticks, the nicotine delivery is around 0.21 mg/stick which is just above the LOQ (0.16 mg/stick) and is approx. 6 times lower than in the fresh sticks. Glycerin is around 0.6 mg/stick,

Confidentiality Statement

Certain data and information contained in this document constitute trade secrets and confidential commercial information. The legal protections that apply to trade secrets and confidential information are hereby claimed under the applicable provisions of law. No part of this document may be publicly disclosed without the written consent of Philip Morris International Inc. of which Philip Morris Products SA is an affiliate.



TPM ~7 mg/stick and water ~5 mg/stick, which represent approximately 8 times lower deliveries compared to the fresh sticks.

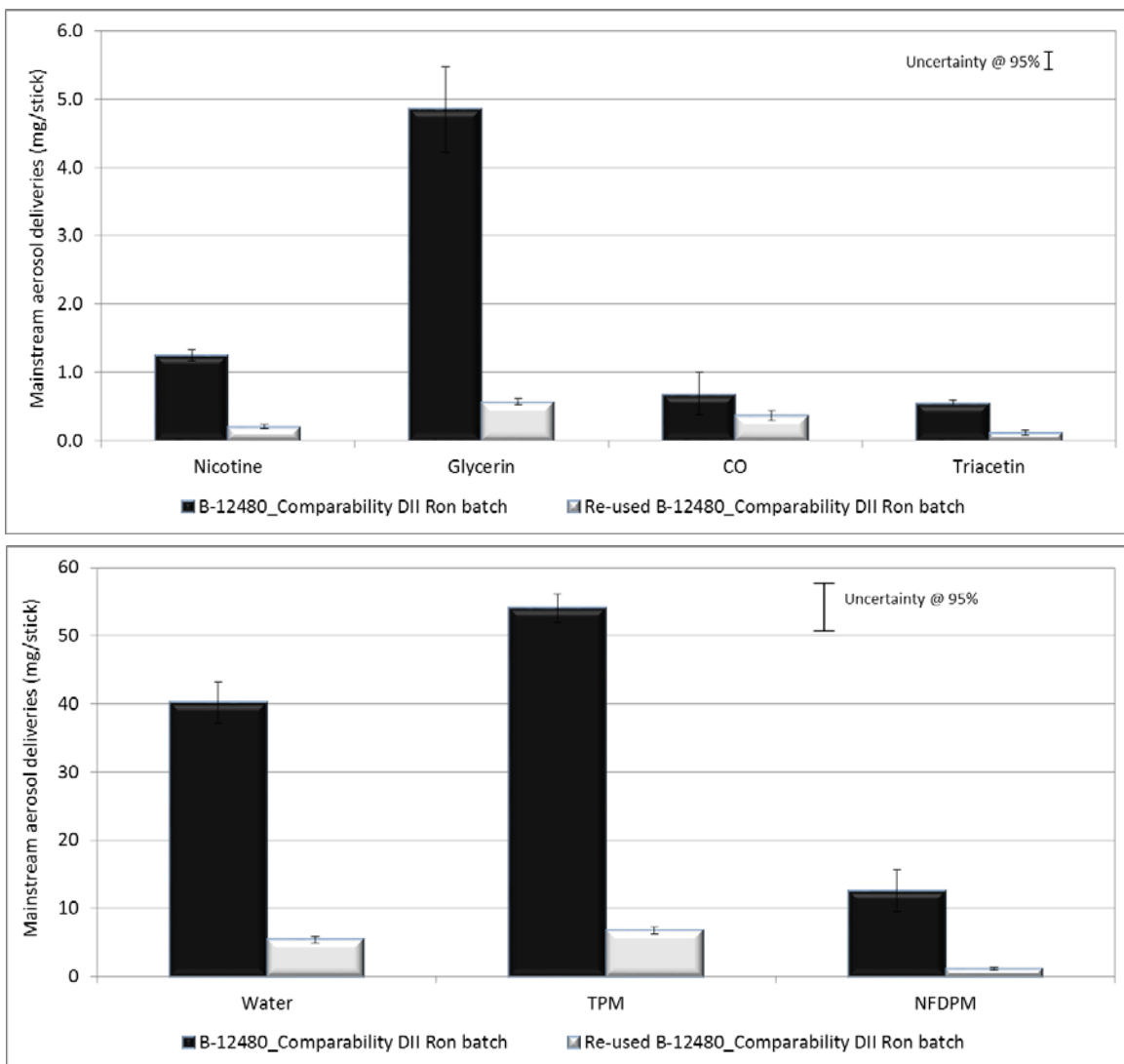


Figure 1– Nicotine, water, glycerin, TPM, NFDPM, CO and triacetin

Confidentiality Statement

Certain data and information contained in this document constitute trade secrets and confidential commercial information. The legal protections that apply to trade secrets and confidential information are hereby claimed under the applicable provisions of law. No part of this document may be publicly disclosed without the written consent of Philip Morris International Inc. of which Philip Morris Products SA is an affiliate.



3.2 Determination of phenolic compounds

Results are depicted in [Table 4](#) and [Table 5](#) and [Figure 2](#).

Table 4: Phenol, o-cresol, m-cresol, p-cresol, catechol, resorcinol and hydroquinone on fresh sticks

		B-12480 Comparability DII Ron batch						
		phenol	o-cresol	m-cresol	p-cresol	catechol	resorcinol	hydroquinone
		ug/stick						
AVG		1.68	0.12	0.05	0.09	17.5	0.05	8.01
Median		1.63	0.12	0.05	0.08	17.7	0.05	8.13
stdev		0.23	0.02	0.01	0.02	0.9	0.00	0.49
n		4	4	4	4	4	4	4
Uncertainty of mean		0.36	0.04	0.01	0.03	1.4	0.01	0.78
CV		13.6%	18.9%	17.9%	20.2%	5.0%	8.0%	6.1%
Uncertainty of indiv.		0.72	0.08	0.03	0.06	2.77	0.01	1.56
LIMS no.	Replicate							
1381010	rep 1	1.69	0.16	0.06	0.11	18.3	0.06	8.40
	rep 2	1.56	0.11	0.04	0.07	17.4	0.05	7.87
	rep 3	1.99	0.13	0.05	0.09	18.0	0.06	8.40
	rep 4	1.47	0.10	0.04	0.07	16.3	0.05	7.38

Table 5: Phenol, o-cresol, m-cresol, p-cresol, catechol, resorcinol and hydroquinone re-used sticks

		Re-used B-12480 Comparability DII Ron batch						
		phenol	o-cresol	m-cresol	p-cresol	catechol	resorcinol	hydroquinone
		ug/stick						
AVG		0.65	0.05	0.02	0.03	3.2	0.01	1.71
Median		0.65	0.05	0.02	0.03	3.2	0.01	1.73
stdev		0.06	0.00	0.00	0.00	0.3	0.00	0.15
n		4	4	4	4	4	2	4
Uncertainty of mean		0.09	0.00	0.00	0.00	0.5	0.00	0.24
CV		9.0%	5.5%	7.2%	6.8%	10.5%	0.1%	9.0%
Uncertainty of indiv.		0.19	0.01	0.00	0.01	1.06	0.00	0.49
LIMS no.	Replicate							
1381013	rep 1	0.65	0.05	0.02	0.04	3.2	0.003 < x < 0.012	1.86
	rep 2	0.72	0.06	0.02	0.04	2.8	0.003 < x < 0.012	1.53
	rep 3	0.58	0.05	0.02	0.03	3.2	0.01	1.65
	rep 4	0.65	0.05	0.02	0.03	3.6	0.01	1.82

The level of phenols in the aerosol of the re-used sticks were found to be as well lower compared to the fresh sticks.

Confidentiality Statement

Certain data and information contained in this document constitute trade secrets and confidential commercial information. The legal protections that apply to trade secrets and confidential information are hereby claimed under the applicable provisions of law. No part of this document may be publicly disclosed without the written consent of Philip Morris International Inc. of which Philip Morris Products SA is an affiliate.



In re-used sticks, 2 values are below LOQ (0.012 ug/stick) for resorcinol and 2 are just above the LOQ. Phenol and cresols levels are approx. 2.5 times lower compared to fresh sticks, while catechol, resorcinol and hydroquinone approx. 4-5 times.

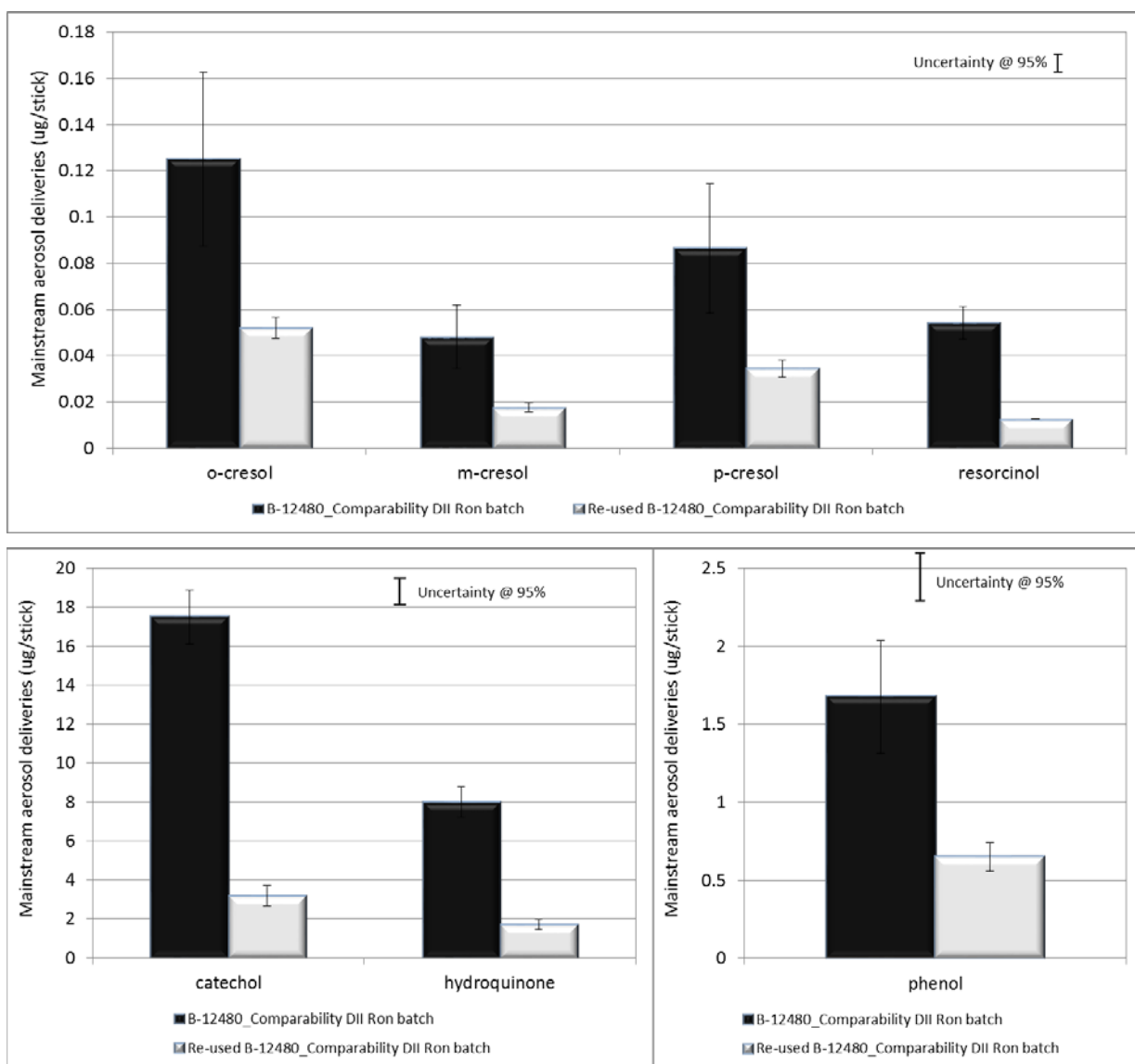


Figure 2– Phenol, o-cresol, m-cresol, p-cresol, catechol, resorcinol and hydroquinone

Confidentiality Statement

Certain data and information contained in this document constitute trade secrets and confidential commercial information. The legal protections that apply to trade secrets and confidential information are hereby claimed under the applicable provisions of law. No part of this document may be publicly disclosed without the written consent of Philip Morris International Inc. of which Philip Morris Products SA is an affiliate.



4 Conclusions

Re-used THS 2.2 sticks generate very low deliveries in terms of nicotine, glycerin, TPM, water, CO and triacetin. There are no indications of overheating. The available amount of aerosol formers, glycerin and water, probably decreases after the first use in the tobacco plug and the remaining amount generates only very low levels in the aerosol.

Consequently, phenols yields are as well reduced in the aerosol compared to a fresh stick.

5 Abbreviations

Abbreviation/Term	Explanation
AVG	Average
CO	Carbon Monoxide
CV	Coefficient of Variation
FPD	Functional Product Design
EDMS	Electronic Document Management System
ISO	International Organization for Standardization
LOQ	Limit of Quantification
NFDPM	Nicotine Dry Free Particulate Matter
PLA	Polylactic acid
RDLIMS	Laboratory Information System
SDMS	Scientific Data Management System
THS	Tobacco Heating System
TPM	Total Particulate Matter
WKI	Work Instruction

Confidentiality Statement

Certain data and information contained in this document constitute trade secrets and confidential commercial information. The legal protections that apply to trade secrets and confidential information are hereby claimed under the applicable provisions of law. No part of this document may be publicly disclosed without the written consent of Philip Morris International Inc. of which Philip Morris Products SA is an affiliate.



6 Review and Approval

This document has been approved by:

Name	Justification / Function	Date / Signature
Rita Hajdu Schenk	Signed as Author / Scientist Product Characterization	16 Aug. 2017
Cyril Jeannet	Signed As Owner and Approver / Manager Product Characterization	16-Aug-2017
Pascale Berbeyer	Signed as Quality Assurance / QA Analyst	16 Aug 2017

7 Document version

Version	Description	Change type
1.0	Initial version	NA
2.0	Alignment of THS 2.2 terminology and added paper signatures	Minor

Confidentiality Statement

Certain data and information contained in this document constitute trade secrets and confidential commercial information. The legal protections that apply to trade secrets and confidential information are hereby claimed under the applicable provisions of law. No part of this document may be publicly disclosed without the written consent of Philip Morris International Inc. of which Philip Morris Products SA is an affiliate.