EXPERIMENTAL REPORT

DROPLET SIZE DISTRIBUTION FOR P1 THS 2.2 AND MENTHOL (VINNY MINT HIGH) TEST ITEMS USING A VALIDATED PIXE CASCADE IMPACTOR

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1 APPROVAL AND VERIFICATION

Name	Justification	Date / Signature
Pratte, Pascal	approved by author, Manager Aerosol Physics	29 Sept. 2016 Pascal Pratte
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2 ABSTRACT

The current report describes the measurement of Droplet Size Distribution (DSD) data for P1 THS2.2 (DV.000174) and menthol test items. From the experiments, it was shown that aerosols generated from three tested batches were respirable, meaning smaller than 2.5 µm in Mass Median Aerodynamic Diameter (MMAD).

3 INTRODUCTION

The Droplet Size Distribution (DSD) influences the deposition pattern in the respiratory tract of inhaled aerosols. This assay is used to determine the DSD of the aerosol generated from P1 THS 2.2 device using menthol test items (see Table 1). This assay is based on the use of a cascade impactor to gravimetrically determine the DSD. This instrument was chosen due to its large DSD size range (60nm – 16µm).

4 RESEARCH QUESTION

Is the DSD within the respirable range for P1 device using menthol test items (THS 2.2 DORADO I VINNY MINT HIGH)?

5 PLAN

5.1 Approach

The experimental approach is described in details in the work instruction named "Droplet Size Determination using the PIXE Cascade Impactor."

5.2 Experiment

Experiments were performed by the Analytical Laboratory Team on August 23rd, August 24th and September 09th 2016.

5.3 Design

At the beginning of each experimental day, a suitability test was performed to verify that the PIXE cascade impactor is functioning in a correct manner. To this end, a 3R4F cigarette monitor was used using the Health Canada regimen. The weight of the impaction plates, before and after the experiment, were recorded in a validated form. On this form, the pass criterion for the suitability test is met when the mass fraction for plate 2 is comprised between 0.70 and 1.00 (unitless). When the criteria are fullfiled the PIXE cascade impactor is disassembled and cleaned. Subsequently, the plates are weighed, reassembled and the PIXE cascade impactor connected to a Programmable Dual Syringe Pump (PDSP). The aerosol generation was performed under Health Canada regimen and the resulting loaded plates weighed upon aerosol collection. The mass of the plates, prior and after the aerosol collection, were recorded on a dedicated validated form.

5.4 Samples

Prior conducting the aerosol generation experiment, samples were conditioned according to ISO conditions for P1 menthol test items. Ten replicates were performed for three distinct batches over three different days. The laboratory condition set during the aerosol generation were maintained at 22°C ± 2°C and 60% ± 5% relative humidity. The aerosol was generated under Health Canada smoking regimen (Health Canada, T-115, 1999).

Table 1: Test item description

Product/ test item	Batch number (Stick/Pack)	Additional information
THS 2.2 DORADO I VINNY MINT HIGH	B-31061 / 41.2572271	**10 replicates per batch**
	B-31062 / 41.2572272	
	B-31063 / 41.2572273	

5.5 Instruments and Materials

Measurements and analyses were performed based on the working instruction named "Droplet Size Determination using the PIXE Cascade Impactor."

5.6 Procedure

In a first step, before conducting the experiments according to Table 1, pre-experiments were performed to determine the overloading threshold (maximum number of puffs collected without overloading the PIXE cascade impactor). The test items was connected to the inlet of the PDSP and the aerosol generation started. The first 5 puffs were exhausted to the evacuation port. Iteratively, an increasing number of puffs were collected in the PIXE cascade impactor. The PIXE cascade impactor was dismantled while visual inspection was used to determine whether the PIXE cascade impactor was overloaded. An overloaded impaction plate is recognized when aerosol particle/droplet has not only been precipitated on the impaction plate and also on the nozzle of the impactor segment located directly above the respective impaction plate. If at least one impactor plate is overloaded, the pre-experiment is repeated by reducing the number of collected puff by one in an iterative manner. In the case the impactor is not overloaded, the pre-experiment is repeated with one additional puff in an iterative manner. This means that the procedure was repeated until the maximum number of puffs is reached without overloading the PIXE cascade impactor.

5.7 Data Processing

The calculation of the MMAD and GSD is performed using SAS software version 9.2. For this a nonlinear regression analysis of the normalized mass fraction nMfi vs the respective Mid-Point Diameter Di using the model function (Hinds 1999, p. 92) is used based on the minimum squared errors.

5.8 Respirability acceptance criteria based on aerosol measured MMAD and GSD

The key objective of performing these tests was to ensure that aerosols are respirable which means that more than 80% of the total inhaled aerosol is transferred in the human lung alveolar region. Table 2 summarizes the different (MMAD, GSD) couples for which the aerosol is respirable. Each member of each couple represents the maximum value for which the aerosol is respirable. For example, for a GSD of 1.6, the MMAD should not exceed 0.9 µm in order to classify the aerosol as respirable, considering the method variability (see Table 2, right column). For a perfectly mono-disperse aerosol (GSD = 1.0), the maximum possible MMAD characterizing it respirable is 2.4 µm.

Table 2: MMAD and GSD upper limit values for which the aerosol is respirable (assuming lognormal distribution)

Theoretical upper limit values		Upper limit values for MMAD taking into account method variability
MMAD [µm]	GSD [-]	MMAD [µm]
2.5	1.0	2.4
2.1	1.1	2.0
1.7	1.2	1.6
1.5	1.3	1.4
1.3	1.4	1.2
1.1	1.5	1.0
1.0	1.6	0.9
0.9	1.7	0.8
0.8	1.8	0.7
0.7	1.9	0.6
0.6	2.1	0.5
0.5	2.3	0.4
0.4	2.6	0.3
0.3	3.0	0.2

6 RESULTS

From the obtained DSD, the MMAD and the GSD values were calculated and reported in the current section. Briefly, the MMAD is a representative size parameter of the DSD whereas the GSD quantifies how the mass spreads around the MMAD. By definition, a GSD between 1.0 and 1.2 characterizes a mono-disperse distribution otherwise above this range, it is a poly-disperse distribution. The calculated MMAD and GSD values obtained for the three testing days are summarized in Table 3, 4 and 5. From the Tables, MMADs were found to range from 0.66 to 0.90 µm while the related Geometric Standard Deviation (GSD) parameter was comprised between 1.21 and 1.47.

Table 3: MMAD and GSD values for Day 1 - 23.08.2016 (B-31061 / 41.2572271)

Replicate	MMAD [µm]	GSD [-]
1	0.88	1.23
2	0.81	1.31
3	0.83	1.31
4	0.66	1.32
5	0.66	1.31
6	0.66	1.31
7	0.67	1.33
8	0.72	1.43
9	NA	NA
10	0.73	1.39

- No sufficient mass was accumulated in the PIXE cascade impactor for replicate 9 to obtain a MMAD and a GSD value.

Table 4: MMAD and GSD values for Day 2 – 24.08.2016 (B-31062 / 41.2572272)

Replicate	MMAD [µm]	GSD [-]
1	0.86	1.27
2	0.75	1.36
3	0.81	1.32
4	0.88	1.21
5	0.88	1.14
6	NA	NA
7	0.88	1.25
8	0.88	1.27
9	0.68	1.47
10	0.74	1.36

- No sufficient mass was accumulated in the PIXE cascade impactor for replicate 6 to obtain a MMAD and a GSD value.

Table 5: MMAD and GSD values for Day 3 – 09.09.2016 (B-31063 / 41.2572273)

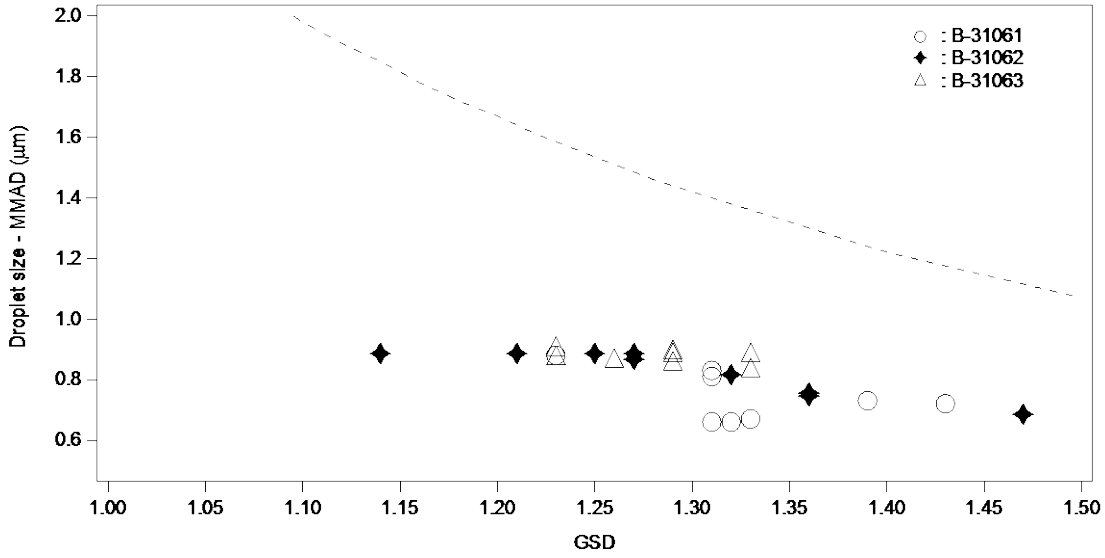
Replicate	MMAD [µm]	GSD [-]
1	0.88	1.29
2	NA	NA
3	0.83	1.33
4	0.88	1.29
5	0.85	1.29
6	0.87	1.23
7	0.86	1.26
8*	0.89	1.29
9	0.88	1.33
10	0.90	1.23

- No sufficient mass was accumulated in the PIXE cascade impactor for replicate 2 to obtain a MMAD and a GSD value.

7 DISCUSSION AND CONCLUSIONS

According to the respirability criteria defined in section 5.8, MMAD and GSD couples were plotted from Table 3, 4, 5 and compared to upper limit couples from Table 2 as shown in Figure 1. From the Figure, it is observed that the data point for the three tested batches (B-31061, B-31062, and B-31063) are below the dashed curve defining the respirability limit for an aerosol. Based on that, we conclude that aerosols generated from the three batches were respirable.

Figure 1: MMAD plotted as a function of GSD (unitless) values. The different data point symbols are linked to the three batch numbers displayed in the insert of the Figure.



8 REFERENCES

- DSD Protocol Menthol 2
- Health Canada, Official Method T-115, Determination of "Tar", Nicotine and Carbon Monoxide in Mainstream Tobacco Smoke, December 31, 1999
- Aerosol Technology,W.C. Hinds, 1999, second edition

9 REVISION HISTORY

Version N°	Description of change	Type of change
2.0	Prior Version 1.0 was signed, a typographic error was identified in the title and corrected. Consequently, Version 2.0 was issued and signed as the original version.	1

(1. Major change/new version; 2. Minor change; 3. Review without change)

10 ABBREVIATIONS

Abbreviations		
DSD	:	Droplet Size Distribution
GSD	:	Geometric Standard Deviation
MMAD	:	Mass Median Aerodynamic Diameter
P1	:	Platform 1
PDSP	:	Programmable Dual Syringe Pump
PM2.5	:	Particulate Matter
THS	:	Tobacco Heating System