Comparative Smoke Analysis of CORESTA Monitoring Cigarettes

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ABSTRACT : This experiment was conducted as a part of Asia collaborative study on purpose of verifying the difference between CM6 and CM7 including 3R4F for reference. It carried out using various analytical categories for example, main stream, sidestream and Av. smoke. Additional analysis such as physical properties, blending ratio, combustibility and general leaf component analysis also implemented in order to investigate the difference. We complied with ISO standard and CORESTA recommended method during analytical operating procedures. In this study, we described that comparative analytical result for CM6 and CM7 known as reference or monitoring cigarettes including 3R4F for reference. All sample cigarettes were conditioned at 22°C, 60% relative humidity for 48 hours. Av. Smoke, MS and SS smoke analysis were performed over five times with two smoking condition, ISO and Health Canada with the exception of Av. smoke analysis. We complied with ISO standard method during analytical operating procedures. And, we conducted additional analysis, such as physical properties, blending ratio, combustibility and leaf component analysis also in order to investigate the difference.

In conclusion, we found out some differences between CORESTA monitoring cigarette No. 6 and No 7. The smoke components such as total particulate matters, NFDPM, nicotine and carbon monoxide contents of CM7 were a little lower than CM6. And, these phenomena were the same as not only main stream smoke but also side stream smoke and Av. smoke. This tendency was consistent with ISO and Health Canada smoking condition. Besides, leaf constituents' color of CM7 was darker than CM6. In case of combustibility, it showed short combustion time approximately 30 seconds.

Key words : monitoring cigarettes, mainstream, sidestream and Av. smoke

MATERIALS AND METHODS

Cigarettes & sample selection

Sample cigarettes were CM6 and CM7 known as monitoring cigarettes has been produced according to the provisions given in the International Standard, ISO 16055. And analyzed in annual surveys organized by the CORESTA "Routine Analytical Chemistry Sub-Group" by laboratories each equipped to analyze according to ISO 3308, 3402, 4387, 8243, 8454, 10315 and 10362. As for 3R4F, it was used for reference. All sample cigarettes were selected randomly and were conditioned at twenty-two plus minus one degrees Celsius, sixty plus minus two percent relative humidity for forty eight hours in

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accordance with ISO and CORESTA recommended method. And then, smoke analyses were carried out. Atmospheric conditions and sample regime specification were showed in table 1.

 Table
 1. Atmospheric conditions and sample

 regime
 specification for smoking analysis

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~	Atmospheric	conditions
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	Conditioning Atmosphere*	Test Atmosphere
Temperature	$22~°C~\pm~1~°C$	$22~°C~\pm~2~°C$
Relative Humidity	(60 ±2) %	(60 ± 5) %

* Samples should be conditioned for at least 48 hours but no more than 10 days.

> Sample regime specification

Puff Volume (mL)	Puff Frequency* (s)	Puff Duration (s)	Vent Blocking (%)	Descrip tion
35 ± 0.3	$60~\pm~0.5$	2 ± 0.2	0	ISO
$55~\pm~0.5$	$30~\pm~1.0$	2 ± 0.2	100	Intense

*Time from the start of one puff to the start of the next puff.

Smoking machines and sample preparation

Three types of smoke analysis method were used such as mainstream, sidestream and Av. smoke. Using a smoking machine such as, RM 200 for mainstream smoke, SM 405-SV for sidestream smoke and RM 20D for Av. smoke respectively the cigarette samples were smoked in with CORESTA recommended accordance methods and then TPM were collected. In addition, the standard butt length to which cigarettes has been marked the greatest among of the following three cases for instance, 23 mm, length of filter + 8 mm and length of overwrap + 3 mm. Standard butt length selection criteria are the same as Table 2. In addition, many other things with regard to standard conditions, sample cigarette conditioning, total particulate matter(TPM) and nicotine free dried particulate matter(NFDPM), carbon monoxide, nicotine and water quantitative analysis method were complied with ISO and Health Canada method.

Experimental work flow by smoke analysis type

Fig. 1 shows roughly work flow for mainstream smoke analysis. As for extraction step, iso-propanol was used for working solution. And, n-heptadecane and ethanol were used as internal standard, namely n-heptadecane was necessary for nicotine quantification. In the same way, ethanol was used for water contents quantification.

Sidestream smoke analysis procedure was so cumbersome and complicated. There were many apparatus mounted and set-up required, such as fishtail chimney, impinged trap set, and cigarette holder for MS/SS with cambridge filter pad(CFP), conical flask for CFP infusion after smoking and one-mark flask for capturing chimney inner wall

Table 2. Standard butt length selection criteria by CRM

		CM6			CM7			3R4F	
Length of Filter	21 mm	+ 8mm	= 29 mm	21 mm	+ 8mm	= 29 mm	27 mm	+ 8mm	= 35 mm
Length of Overwrap	29 mm	+ 3mm	= 32 mm	29 mm	+ 3mm	= 32 mm	32 mm	+ 3mm	= 35 mm
Ultimate Butt Length		32 mm			32 mm	L		35 mm	
Butt Length Selection Criteria*	- length of filter + 8 mm			nm					

• The standard butt length to which cigarettes shall be marked shall be the greatest of the following three lengths.

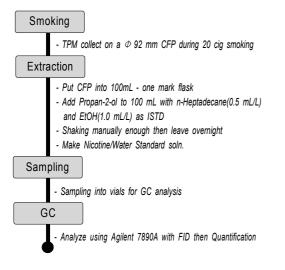


Fig. 1. Experimental work flow about mainstream smoke.

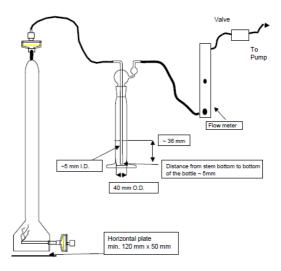
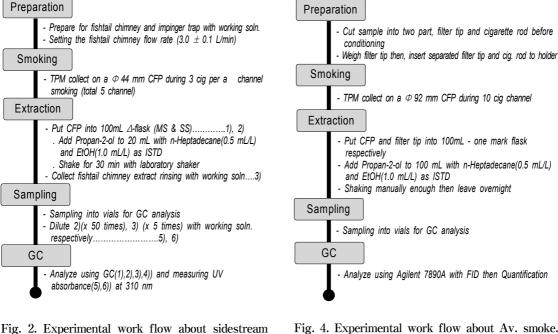


Fig. 3. Schematic diagram for sidestream smoke collection system with impinger in place.



- Analyze using Agilent 7890A with FID then Quantification

Fig. 4. Experimental work flow about Av. smoke.

In addition, fishtail chimney rinsing extract. flow rate must be checked previously. Fig. 2 and Fig. 3 displays work flow for sidestream

smoke.

smoke analysis procedure and schematic diagram for sidestream smoke collection system in separately.

Av. smoke analysis procedure had a big difference comparing with others that cutting sample into two parts, filter tip and cigarette rod before conditioning. The other processes were like same ways. That was what should be dealt with additional analysis about filter tip. In this part, it should be conducted measuring for filter tip's weight about before and after smoking. That had the three steps as follows.

Step 1. Cut sample cigarette into two part, filter tip and cigarette rod before conditioning

Step 2. Weigh filter tip and then, insert separated filter tip and cigarette rod into cigarette holder

Step 3. TPM collect on a 92 mm diameter CFP during ten cigarette smoking

Leaf components and physical properties analysis

We conducted additional analysis, such as physical properties, blending ratio, combustibility and leaf component analysis also in order to investigate the difference. In case of physical properties, tobacco rod length, cut tobacco weight, circumference, (un)encapsulated pressure drop(UPD and EPD) of tobacco rod tobacco rod hardness, moisture content of cut tobacco, whiteness, opacity, thickness and porosity of cigarette paper are included. Total alkaloid, total nitrogen, total sugar, nitrate and chloride were analyzed with continuous flow analyzer for leaf component analysis.

RESULTS AND DISSUSSION

Smoke analysis results

Standard curves for nicotine and water contents quantification at ISO and intense smoking condition were acquired within appropriate reliable range. Fig. 5 demonstrated reliabilities about standard curves for GC analysis that all of r-square values were higher than zero point three nine.

At comparative analysis of mainstream smoke

for three types of sample cigarettes, NFDPM, nicotine, carbon monoxide and puff count of CM7 showed a little lower than CM6. This tendency was same at ISO and Health Canada condition. On the contrary, sidestream smoke of CM7 displayed that the contents of four items equal or a little higher than CM6. On intense condition, carbon monoxide content was showed as same. But, puff count of CM7 was approximately one-puff lower than CM6. In terms of Av. smoke, CM7 was lower than CM6 in relation to NFDPM, nicotine and carbon monoxide as well. Fig. 6 through fig. 9 show each result.

Physical properties analysis results

Fig. 10 explain the basic construction about Especially, we found that three samples. cigarette paper porosity of CM7 was lower than CM6. As for physical properties, CM7 was lower than CM6 to encapsulated pressure drop(EPD)of filter tip and hardness of cigarette. Whereas CM7 is higher than CM6 to(un)encapsulated pressure drop(UPD and EPD) of cigarette and unencapsulated pressure drop of cigarette rod. And, they showed similar tendency to the weight of cigarette and cut tobacco. In case of combustibility, CM7 was more rapid than CM6. The other items of physical properties were also analyzed such as whiteness, opacity and thickness of cigarette paper. And they showed similar tendency. The color of cut tobacco constituent with naked eye for CM7 was darker than CM 6.

With regard to leaf blending, CM6 and CM7 had not reconstituted tobacco leaf when comparing with 3R4F. In addition, CM7 was darker than CM6 at the color of cut tobacco constituent. Table 3 and table 4 displays those results in detail.

Leaf components analysis results

Table 5 shows the tobacco leaf components about three samples. CM7 was lower than CM6 to total alkaloid, total sugar and chloride

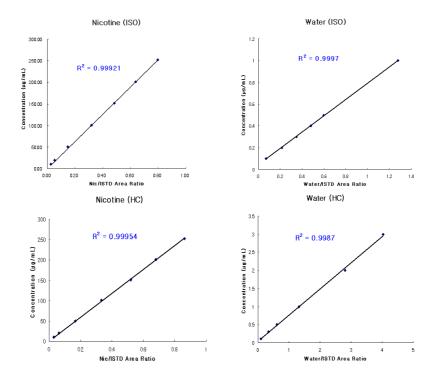


Fig. 5. Standard curves for GC analysis in nicotine and water contents quantification.

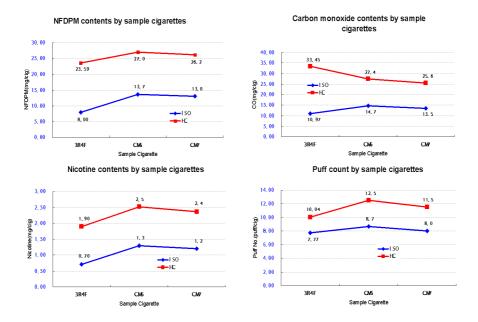


Fig. 6. Comparative analysis of mainstream for three sample cigarettes about NFDPM, nicotine, carbon monoxide and puff count under the ISO and Health Canada condition.

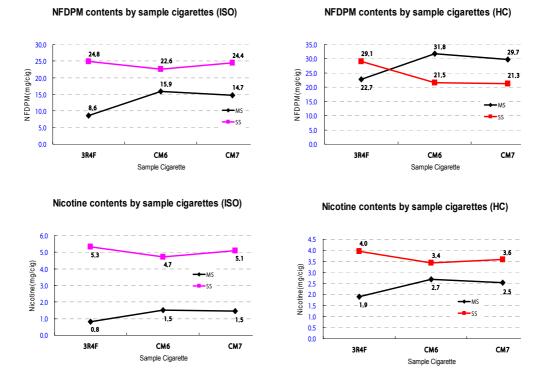


Fig. 7. Comparative analysis between mainstream and sidestream for three sample cigarettes about NFDPM and nicotine under the ISO and Health Canada condition.

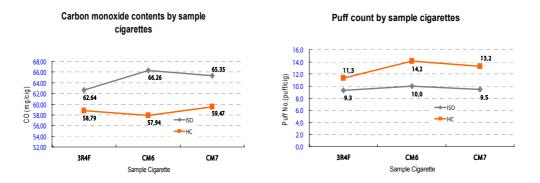


Fig. 8. Comparative analysis of sidestream for three sample cigarettes about carbon monoxide and puff count under the ISO and Health Canada condition.

including sugar by nicotine ratio. Whereas CM7 was higher than CM6 regarding as nitrate and total nitrogen with total nitrogen by nicotine ratio. Besides, CM 6 and CM 7 showed higher

contents in terms of nitrogen, total sugar and sugar by nicotine ratio when comparing with 3R4F. In relation to nitrate and chloride, those tendencies were found out to the contrary.

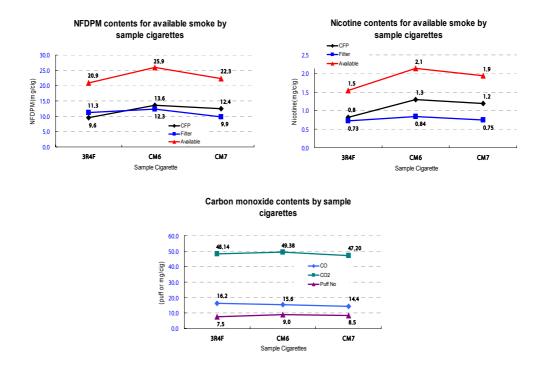


Fig. 9. Comparative analysis of Av. smoke for three sample cigarettes about NFDPM, nicotine and carbon monoxide by CFP, cigarette filter and Av. smoke.

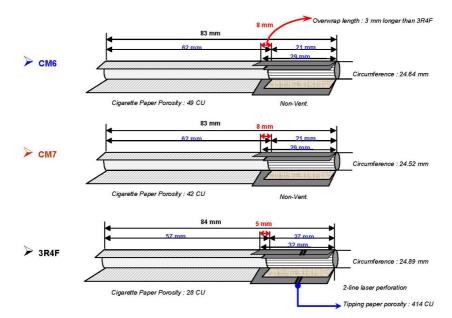


Fig. 10. Basic construction of three sample cigarettes.

Items Sample Cig	CM6	CM7	3R4F	Unit
Cigarette Weight	976	958	1,043	mg/cig.
Cut Tobacco Weight	761	756	759	mg/cig.
Encapsulated Pressure Drop of Cigarette	145	151	184	mmH ₂ O
Unencapsulated Pressure Drop of Cigarette	145	151	138	mmH ₂ O
Unencapsulated Pressure Drop of Cigarette Rod	58	65	58	mmH ₂ O
Encapsulated Pressure Drop of Filter	80	75	119	mmH ₂ O
Hardness	82.2	78.3	80.0	%
Moisture Contents of Cut Tobacco	12.3	13.9	12.6	%
Combustibility	7 min 15 s	6 min 49 s	6 min 3 s	min.sec / 30 mm

Table 3. Analytical results about physical properties and leaf blending constituent by three sample cigarettes

Table 4. Analytical results about physical properties and leaf blending constituent by three sample cigarettes

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Encapsulate	ed Pressure Drop of Filter	80	75	119	mmH ₂ O
Hardness		82.2	78.3	80.0	%
Moisture C	ontents of Cut Tobacco	12.3	13.9	12.6	%
	Whiteness	88.9	88.6	79.3	%
Cig. Paper	Opacity	79.1	80.9	79.3	%
т арст	Thickness	34	34	39	μm
	Flue cured	Detected	Detected	Detected	%
	Burley	Detected	Detected	Detected	%
Leaf Blending	Oriental	Questionable	Questionable	Questionable	%
	Reconstituted Tobacco Leaf	$N.D^{1)}$	N.D	Detected	%
	Expanded Tobacco	N.D	N.D	N.D	%
	Expanded Stem	N.D	N.D	N.D	%

1) N.D : Not Detected

Items Sample Cig	CM6	CM7	3R4F	Unit
Nicotine	2.25	2.21	2.08	%
Total Sugar	16.2	14.6	10.7	%
Nitrate	0.17	0.22	1.12	%
Chloride	0.52	0.47	0.77	%
Total Nitrogen	2.47	2.76	2.83	%
S/N ratio	7.22	6.62	5.15	-
Total N./Nic. ratio	1.10	1.25	1.36	-

Table 5. Analytical results about general leaf component by three sample cigarettes

CONCLUSIONS

In this study, we found out some differences between CORESTA monitoring cigarette No. 6 and No 7. This experiment was conducted as a part of Asia collaborative study on purpose of verifying the difference between CM6 and CM7 including 3R4F for reference. It carried out using various analytical categories for example, main stream, sidestream and Av. smoke as shown summarized fig. 11 through 13.

Additional analysis such as physical properties, blending ratio, combustibility and general leaf component analysis also implemented in order to investigate the difference. Av. smoke, mainstream and sidestream smoke analysis were performed over five times with two smoking condition, ISO and Health Canada with the exception of Av. smoke analysis. The smoke components such as total particulate matters, NFDPM, nicotine and carbon monoxide contents of CM7 were a little lower than CM6. And, this phenomenon was the same as not only main stream smoke but also side stream smoke and Av. smoke. This tendency was consistent with ISO and Health Canada Besides, the cut tobacco smoking condition. constituent's color with naked eye for CM7 was darker than CM 6. In case of combustibility, it showed short combustion time approximately 30 seconds.

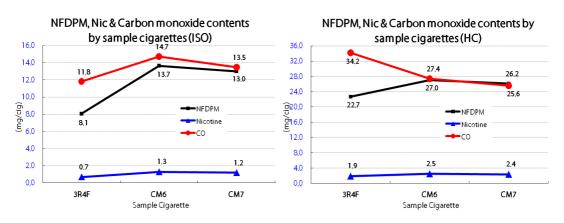


Fig. 11. Overall results of mainstream smoke for three sample cigarettes at two smoking condition.

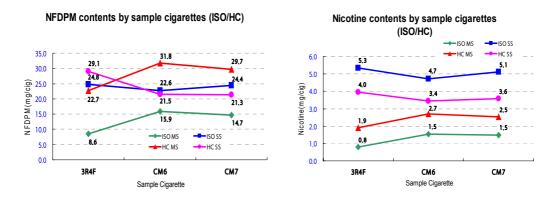


Fig. 12. Overall results of sidestream smoke for three sample cigarettes at two smoking condition.

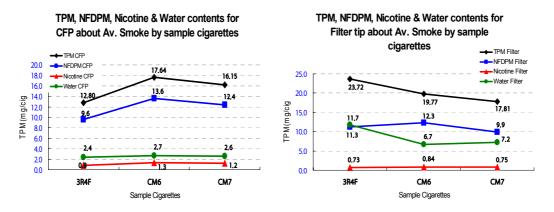


Fig. 13. Overall results of Av. smoke for three sample cigarettes at ISO smoking condition.

REFERENCES

- CORESTA Recommended Method No.21 (1991) Atmosphere for Conditioning and Testing Tobacco and Tobacco Products
- CORESTA Recommended Method No.54 (2002) Determination of nicotine and NFDPM in sidestream smoke using a fishtail chimney and a routine analytical / linear smoking machine
- CORESTA Recommended Method No.55 (2002) Determination of carbon monoxide in the vapor phase of cigarette sidestream smoke using a fishtail chimney and a routine analytical / linear smoking machine

- Health Canada Method T-115 paragraph 16.1 (1999) Ventilation zone blocking, puffing parameters and number of test pieces smoked.
- ISO 10315 (2000) Cigarettes_determination of nicotine in smoke condensates_Gas chromatographic method
- ISO 10362-1 (1999) Cigarettes_determination of water in smoke condensates_Part 1: Gas chromatographic method
- ISO 3308 (2000) Routine analytical cigarette smoking machine – definitions and standard conditions
- ISO 3402 (1999) Tobacco and tobacco products atmosphere for conditioning and testing

- ISO 4387 (2000) Cigarettes_determination of total and nicotine-free dry particulate matter using a routine analytical smoking machine
- ISO 8454 (2007) Cigarettes_determination of carbon monoxide in the vapour phase of cigarette smoke_NDIR method