

CORESTA TASK FORCE ON SIDESTREAM SMOKE: REVIEW OF ACTIVITIES

Period of activities: September 2001 – September 2002

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1. INTRODUCTION

The Sidestream Task Force was created in March 1999 with the objective of developing a method for the collection and evaluation of sidestream NFDPM (“tar”) nicotine and carbon monoxide. The Task Force members further accepted that carbon dioxide would also be measured by those able to do so, although this would remain optional and not part of the objective.

In total, about 35 participants, representing over 20 research entities, have registered in the Task Force, and a total of 16 laboratories actively participated in collaborative trials or associated studies over its period of activity.

2. SUMMARY

The Task Force met 3 times over the considered period: in Zurich (November 2001), and in Paris (March 2002 and June 2002), while a smaller committee had to meet on several occasions to prepare the draft methods for the group and to discuss and perform specific statistical evaluations. The Task Force activity during this period covered essentially 5 phases

- Evaluate the collaborative study results, and issue a draft Coresta Recommended method (CRM 54) for the determination of nicotine and NFDPM yields in sidestream smoke.
- Determine the leading sources of discrepancy in the analytical determinations of the CO yields among the participating laboratories. From the results of the failed collaborative study, more robust procedures were established that could be followed by the different participating laboratories.
- Select and distribute the sets of cigarettes to be used for the tests, and perform a full collaborative study on the determinations of the CO (and optionally CO₂) yield in sidestream smoke following the new protocol.
- Based on the evaluation of the collaborative study results, issue a draft Coresta Recommended method (CRM 55) for the determination of CO yields in sidestream smoke.
- Address the issue of recommending a monitor for the statistical control of the determinations and provide initial data for the recommended monitor (CM4).

3. FIRST ATTEMPT AT A CONCLUSIVE COLLABORATIVE STUDY: RESULTS AND DISCUSSION

The determinations being incompatible, 2 separate protocols for the sidestream smoke yields determination had been drafted and accepted by the group after extensive review, one for NFDPM and nicotine, and the second one for carbon monoxide and optionally carbon dioxide. Fifteen laboratories agreed to participate in the study aimed at determining the repeatability (r) and reproducibility (R) associated with these protocols.

A set of 7 cigarettes was selected, that would present the range of situations that could be encountered in the analysis. These were the CM3 monitor cigarette, a commercial low-sidestream product, a commercial 120-mm slim cigarette, a commercial air-cured cigarette, a commercial flue-cured cigarette, and 2 commercial American blend cigarettes with 9 and 3-mg mainstream tar deliveries (coded M, A, B, C, D, E and F respectively). Eight determinations (from 3 cigarettes each) were required from each cigarette for each analyte.

The r and R calculated from the consolidated results after full statistical analysis for the “tar” and nicotine determinations led the group to accept the proposed protocol and a method was issued. Data are given in Table 1 below. A preliminary set of results had already been reported in the previous yearly report. This is the final data set. The draft Coresta Recommended Method was assigned the number CRM 54 and was submitted to the Scientific Commission for approval.

CIGARETTE CODE		Total NFDPM		Total Nicotine	
		(mg/cig)	(%)	(mg/cig)	(%)
A	r	2.66	19.0%	0.58	14.1%
	R	4.64	33.2%	1.07	26.0%
	Mean	13.97		4.11	
B	r	3.74	13.8%	0.59	10.7%
	R	4.89	18.0%	1.07	19.5%
	Mean	27.14		5.51	
C	r	3.15	15.5%	0.41	14.2%
	R	5.27	25.8%	0.67	23.4%
	Mean	20.40		2.88	
D	r	2.96	13.4%	0.75	12.9%
	R	4.39	19.9%	0.83	14.3%
	Mean	22.04		5.79	
E	r	3.88	14.2%	0.74	14.5%
	R	6.15	22.6%	0.77	15.2%
	Mean	27.25		5.06	
F	r	3.84	17.8%	0.67	13.3%
	R	5.11	23.7%	1.23	24.6%
	Mean	21.58		5.00	
M	r	3.84	14.2%	0.68	13.6%
	R	5.44	20.1%	1.04	20.8%
	Mean	27.04		5.02	

Table 1: Summary of the results of the first conclusive collaborative study for the determination of sidestream smoke NFDPM and nicotine yields.

The r and R calculated from the carbon monoxide (and dioxide) data showed that the reproducibility was much higher for these determinations, and this fact spoke highly for the need to tighten the protocol and further investigate the sources of this variability. It was therefore decided that

1. sources of variability should be identified
2. corrective actions should be documented by a revision of the protocol
3. a pre-test should verify that the corrective actions had been successful
4. and only then could a second conclusive collaborative trial be attempted

4. MODIFICATIONS OF SPECIFIC POINTS OF THE CO PROCEDURE

A questionnaire was circulated among the participants, who were also requested to perform a short experiment on the CM3 monitor, by which they would report the average weight of the equilibrated cigarettes they were testing and their static burn rate under the conditions of sidestream smoke collection they had established.

These tests did not result in a modification of the protocol. Yet, performing them led some participating laboratories to implement minor modifications to the way they performed cigarette equilibration or they set up their collection systems.

More importantly, it was also found that testing the collection setup for leaks on a regular basis was essential, as most of the lines are under reduced pressure and any leak would lead to a dilution of the sample being analyzed. A procedure was therefore included in the protocol to this effect.

In order to ensure that the procedure was fully under control among the different laboratories and that the added leak check was performed correctly before engaging into a second collaborative study, a pre-test was performed on that same CM3 cigarette only. The results of the pre-test are given in table 2 below, that is set to allow a comparison of the sidestream CO yields means, r and R measured for this same CM3 cigarette by the Task Force members on the occasion of different studies : Initial collaborative study performed early in the method development, first (failed) collaborative study, and present pre-trial.

CM3 Monitor	No. of Labs	Means (mg/cig)	r (mg/cig)	R (mg/cig)
Initial Coll. Study	9	50.22	10.29	38.79
1st Coll. Study	14	57.86	10.45	18.07
Pre-test	10	55.86	7.64	10.77

Table 2: Comparison of the results of the different collaborative studies for the determination of the CM3 sidestream smoke CO yield

Note that only 10 participants could perform the last pre-test in view of the limited time allowed for the implementation of the newly introduced procedures, and this could have produced a self selection of the most experienced laboratories. These results confirmed, however, that a substantial improvement in both intra-laboratory and inter-laboratory variability had been achieved by the proposed actions.

A second attempt could thus be made to perform a conclusive collaborative study for the determination of carbon monoxide (and optionally dioxide) yields in sidestream smoke.

5. CONCLUSIVE COLLABORATIVE STUDY FOR CARBON MONOXIDE AND DIOXIDE

The same set of 7 cigarettes that had been selected for the first attempt towards a conclusive study was used for the second collaborative study.

The r and R calculated from the consolidated results after full statistical analysis are given in Tables 3 and 4 below for CO and CO₂ determinations respectively.

Cigarette	Mean (mg/cig)	r (mg/cig)	R (mg/cig)	r (%)	R (%)
A	53.96	7.06	14.27	13.1 %	26.4 %
B	60.50	7.45	14.94	12.3 %	24.7 %
C	55.93	6.92	13.46	12.4 %	24.1 %
D	58.66	6.18	11.69	10.5 %	19.9 %
E	50.87	5.63	9.39	11.1 %	18.5 %
F	50.17	5.71	10.55	11.4 %	21.0 %
M	58.51	6.40	12.57	10.9 %	21.5 %

Table 3: Summary of the results of the second conclusive collaborative study for the determination of sidestream smoke CO yields

Cigarette	Mean (mg/cig)	r (mg/cig)	R (mg/cig)	r (%)	R (%)
A	450.2	52.5	173.1	11.7%	38.5%
B	459.6	58.6	120.1	12.8%	26.1%
C	385.2	34.2	133.4	8.9%	34.6%
D	425.3	48.2	166.8	11.3%	39.2%
E	399.5	38.6	120.1	9.7%	30.1%
F	404.9	44.2	153.1	10.9%	37.8%
M	420.0	57.1	137.7	13.6%	32.8%

Table 4: Summary of the results of the second conclusive collaborative study for the determination of sidestream smoke CO₂ yields

Considering the results of this study performed by the same 15 laboratories as the previous one, the CM3 data compared favorably with those of the pre-test (compare with Table 2). R was now slightly higher, but r was slightly lower than in the pre-test performed by a more limited number of laboratories.

The mean CO yield was very close to the one found in the first study (58.5 vs. 57.9 mg/cig) - though slightly higher because of the suppression of leaks.

It was concluded that the variability had been reduced to a very acceptable level, and these results led the group to accept the proposed protocol as a base for a Coresta Recommended Method.

The method was written and reviewed, and this draft Coresta Recommended Method was assigned the number CRM 55 and was submitted to the Scientific Commission for approval. This method only refers to CO analysis, but it does include in the form of notes all the additional information that is needed for using it for CO₂ determination.

6. SIDESTREAM YIELDS DETERMINATION FOR THE CM4 MONITOR

Last, the group determined that it should recommend that the sidestream yields of a monitor cigarette be measured through a collective undertaking and made available to the laboratories using this monitor.

This was accepted by the Scientific Commission, and, as an initiating step, the group performed another determination of the sidestream NTDPM, nicotine and CO yields of both the CM3 and the newly released CM4 monitors. Although the exercise is essentially completed, some results are still missing.

When complete, the results will be communicated to the coordinator of the Routine Analytical Chemistry Sub-group.

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