



Results of the 2013 CORESTA Part Filter Method ring trial and Comparison with the 2012 ring trial

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Objective of the Ring Trial

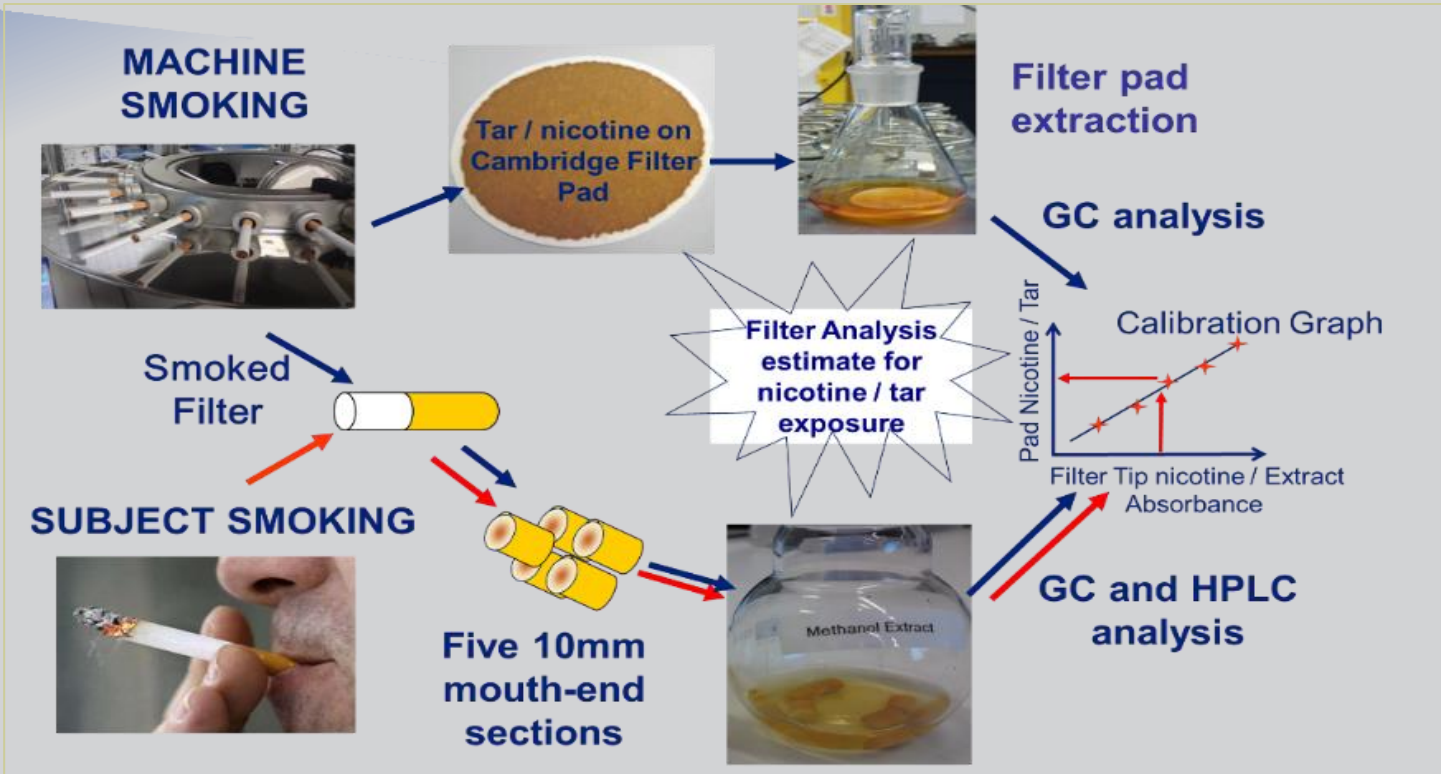
To gain a measure of the *repeatability* and *reproducibility* of the Part-Filter Method to estimate nicotine and tar yields across laboratories

Repeatability (r) = within lab variation

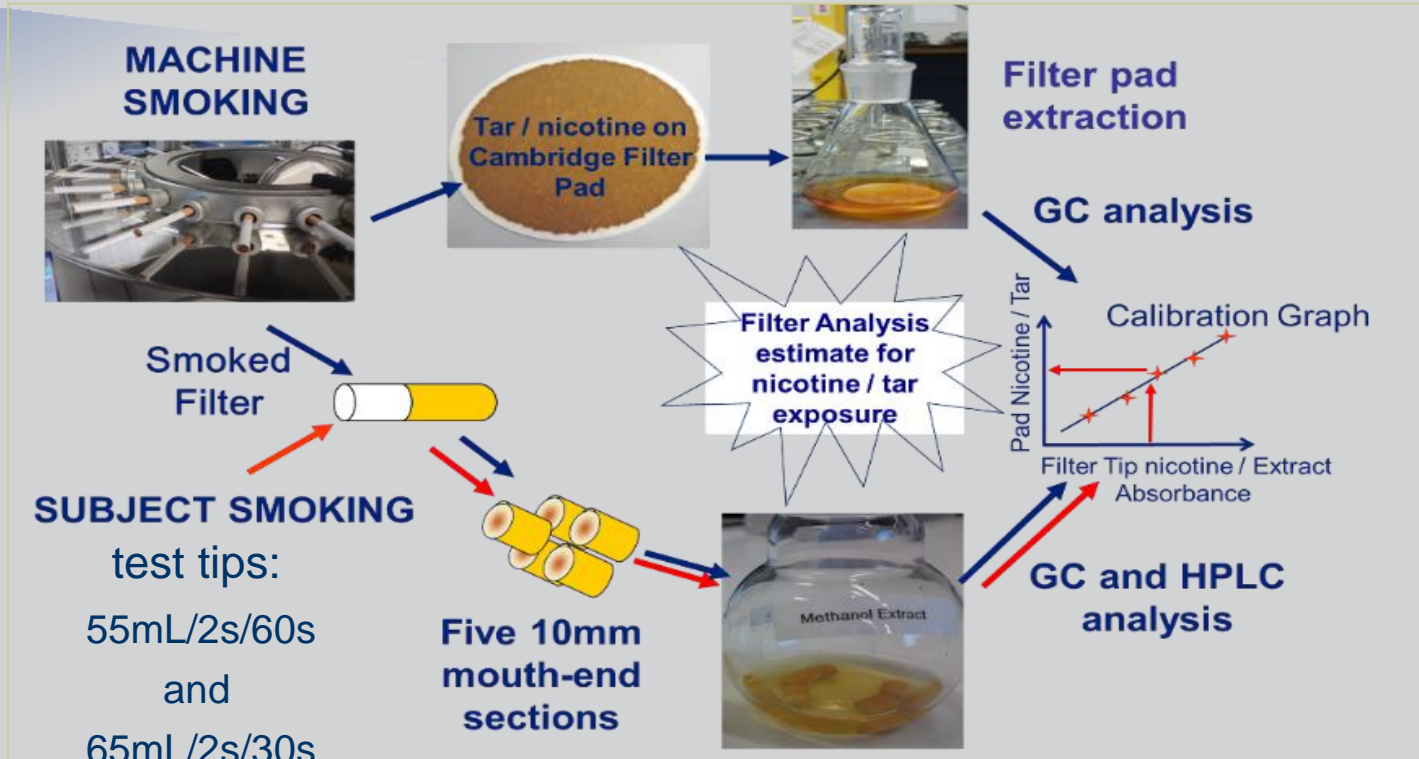
Reproducibility (R) = within and between lab variation

Reach a position on whether the method should be written up as a CORESTA Recommended Method (CRM)

Diagrammatic of the PFM (Part-Filter Method)



Diagrammatic of the PFM (Part-Filter Method)





2013 Ring Trial – Eight laboratories returned valid data

BAT (Germany) GmbH, Bayreuth, Germany

Essentra PLC (formerly FILTRONA), Jarrow, UK

Japan Tobacco Inc, Tokyo, Japan

KT&G Central Research Institute, Daejeon, ROK

Labstat International ULC, Kitchener, Canada

BAT Souza Cruz, Porto Allegra, Brazil

BAT GR&D, Southampton, UK *

Imperial Tobacco Group, Fleury-les-Aubrais, France **

* study co-ordination

** statistical analysis



2013 Results: repeatability (r) and Reproducibility (R) for Nicotine

Estimated nicotine yield from <u>tip nicotine</u>	Number of labs included in statistical evaluation	mean (mg/cig)	r (mg/cig)	R (mg/cig)	CV r (%)	CV R (%)
1mg ISO 'tar' - Low Regime	8	0.29	0.05	0.10	6.0	12.5
1mg ISO 'tar' - High Regime	8	0.70	0.08	0.22	4.2	11.2
10mg ISO 'tar' - Low Regime	8	1.01	0.16	0.29	5.6	10.1
10mg ISO 'tar' - High Regime	7	1.81	0.17	0.23	3.4	4.5

Low test regime: 55mL/2s/60s

High test regime: 65mL/2s/30s



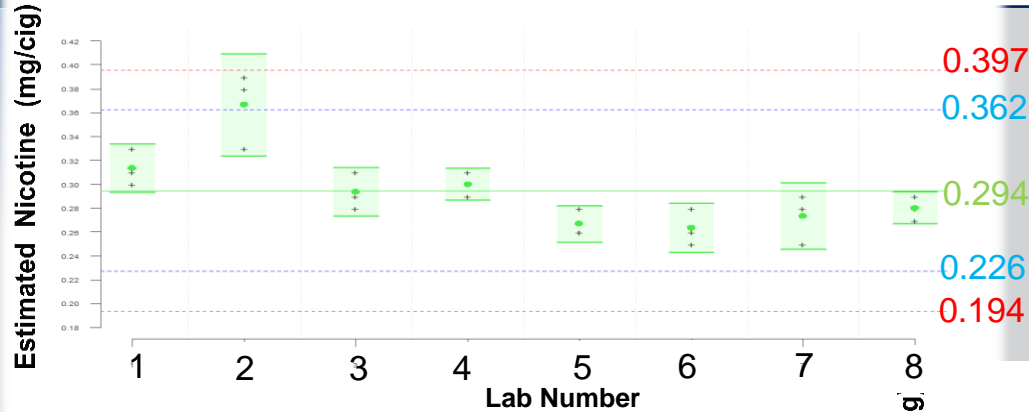
2013 Results: repeatability (r) and Reproducibility (R) for NFDPM

Estimated NFDPM yield from <u>tip UV absorbance</u>	Number of labs included in statistical evaluation	mean (mg/cig)	r (mg/cig)	R (mg/cig)	CV r (%)	CV R (%)
1mg ISO 'tar' - Low Regime	7	3.07	0.79	2.52	9.1	29.0
1mg ISO 'tar' - High Regime	7	7.58	1.10	7.36	5.1	34.3
10mg ISO 'tar' - Low Regime	7	13.58	1.38	7.48	3.6	19.5
10mg ISO 'tar' -High Regime	7	24.56	1.68	15.25	2.4	22.0

Estimated NFDPM yield from <u>tip solanesol</u>	Number of labs included in statistical evaluation	mean (mg/cig)	r (mg/cig)	R (mg/cig)	CV r (%)	CV R (%)
1mg ISO 'tar' - Low Regime	5	3.24	0.64	1.07	6.9	11.7
1mg ISO 'tar' - High Regime	4	7.30	0.50	1.77	2.4	8.6
10mg ISO 'tar' - Low Regime	5	13.87	3.21	4.90	8.2	12.5
10mg ISO 'tar' -High Regime	5	25.15	3.07	6.17	4.3	8.7

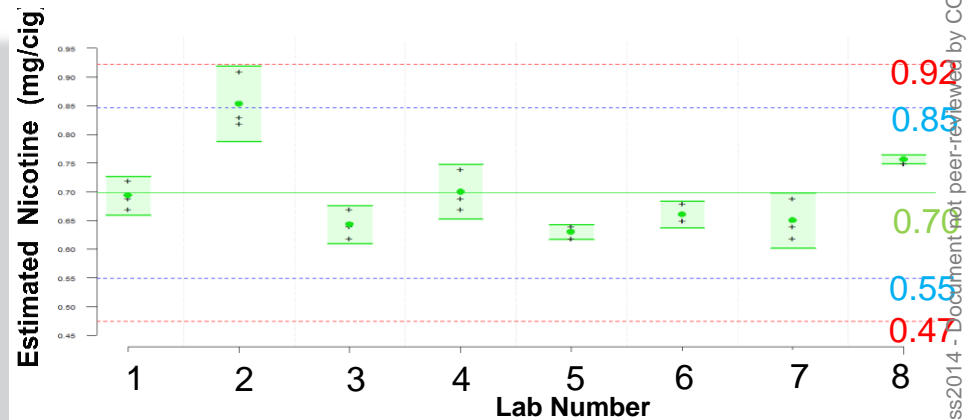


Estimated nicotine yield from tip nicotine: 1mg product



← low regime smoking
8 labs, 0 excluded as outlier

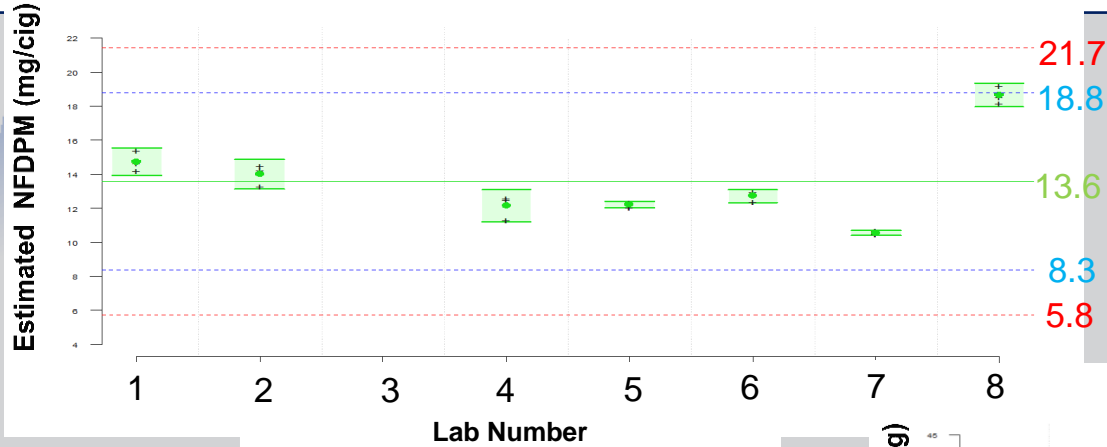
high regime smoking →
8 labs, 0 excluded as outlier



10mg product – similar results, one laboratory excluded as outlier

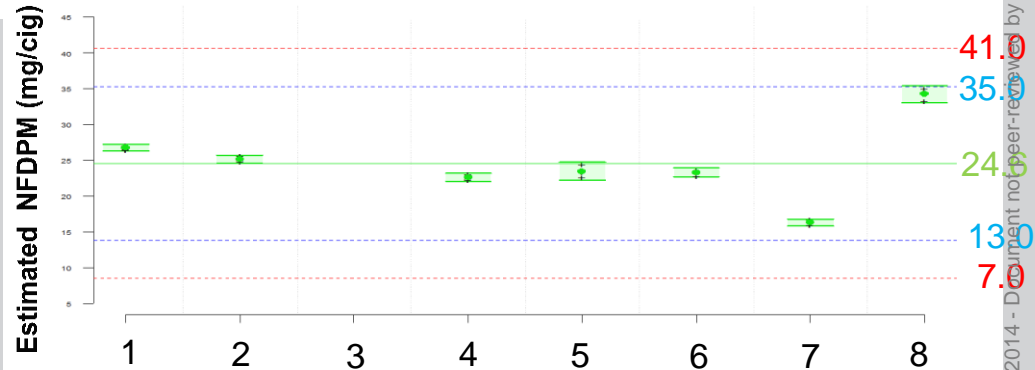


Estimated NFDPM by tip UV absorbance: 10mg product



← low regime smoking
7 labs, 0 excluded as outlier

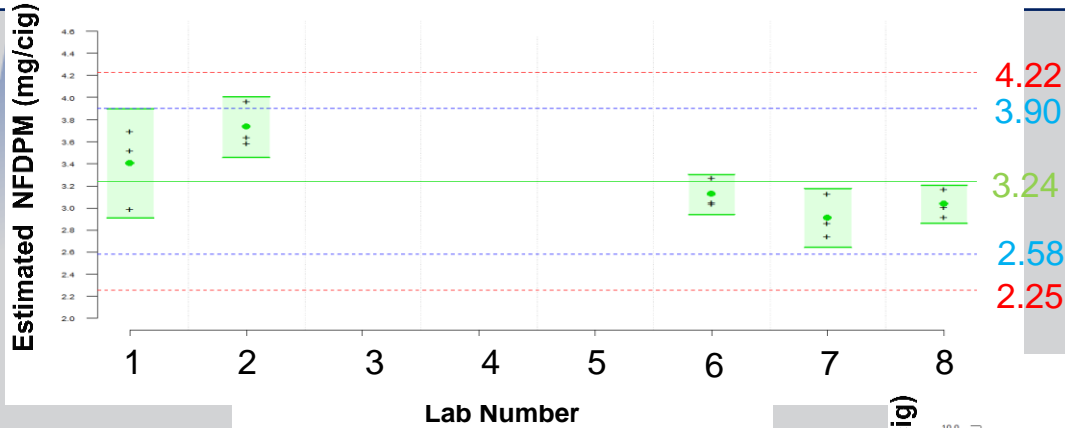
high regime smoking →
7 labs, 0 excluded as outlier



1mg product – similar results, no laboratory excluded as outlier

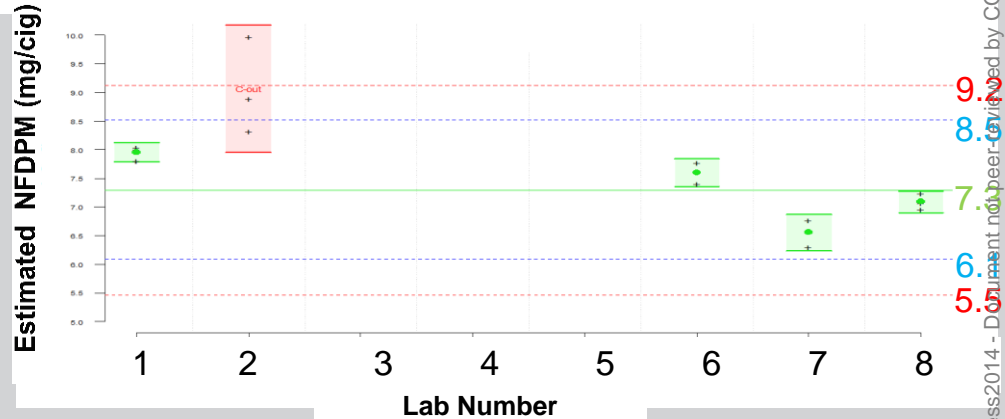


Estimated NFDPM from tip solanesol: 1mg product



← low regime smoking
5 labs, 0 excluded as outlier

high regime smoking
5 labs, 1 excluded as outlier →



10mg product – similar results, no laboratory excluded as outlier



Comparison of two ring trials

		<u>2012</u>			<u>2013</u>		
		Mean (mg/cig)	CV r (%)	CV R(%)	Mean (mg/cig)	CV r (%)	CV R(%)
nicotine by tip nicotine	1mg tar Low	0.11	16.3%	23.5%	0.29	6.0%	12.5%
	1mg tar High	0.35	3.9%	24.5%	0.70	4.2%	11.2%
	10mg tar Low	0.89	4.0%	14.6%	1.01	5.6%	10.1%
	10mg tar High	1.13	4.2%	13.4%	1.81	3.4%	4.5%
NFDPM by tip UV	1mg tar Low	0.98	16.8%	48.4%	3.07	9.1%	29.0%
	1mg tar High	3.43	7.4%	18.0%	7.58	5.1%	34.3%
	10mg tar Low	10.91	3.9%	6.2%	13.58	3.6%	19.5%
	10mg tar High	13.35	3.4%	8.5%	24.56	2.4%	22.0%
NFDPM by tip solanesol	1mg tar Low	1.06	16.2%	57.8%	3.24	6.9%	11.7%
	1mg tar High	3.74	4.2%	15.5%	7.30	2.4%	8.6%
	10mg tar Low	11.75	5.6%	5.6%	13.87	8.2%	12.5%
	10mg tar High	14.31	6.2%	12.8%	25.15	4.3%	8.7%



Key differences between the two ring trials - 2012 and 2013

- ❖ Different test tips regimes
 - Low test regime: 40mL/2s/60s → 55mL/2s/60s
 - High test regime: 65mL/1s/50s → 65mL/2s/30s

- ❖ Smoking calibration regimes simplified → no vents blocked regimes included, 4 puff regime included

- ❖ Differences in cohort of laboratories taking part



Acceptability Criteria

According to AIAG* a general rule for the *variation* introduced by the measurement system is:

- ❖ Under 10 percent reproducibility variation is acceptable
- ❖ 10 percent to 30 percent variation suggests that the system may be acceptable
- ❖ Over 30 percent variation is considered unacceptable, and you should improve the measurement system

*AIAG Automotive Industry Action Group



2013 Ring Trial Summary

- ❖ CV for repeatability (within lab variation) for estimated nicotine and NFDPM remains satisfactory, showed some improvement compared with 2012 ring trial
- ❖ CV for reproducibility for estimated nicotine were in the range 5 to 13%, compared with 13 to 25% in 2012 – a considerable improvement. Indicates method is under control
- ❖ CV for reproducibility for estimated NFDPM by UV were in the range 20 to 34%, compared to 6 to 48% previously. Indicates this measurement requires further attention

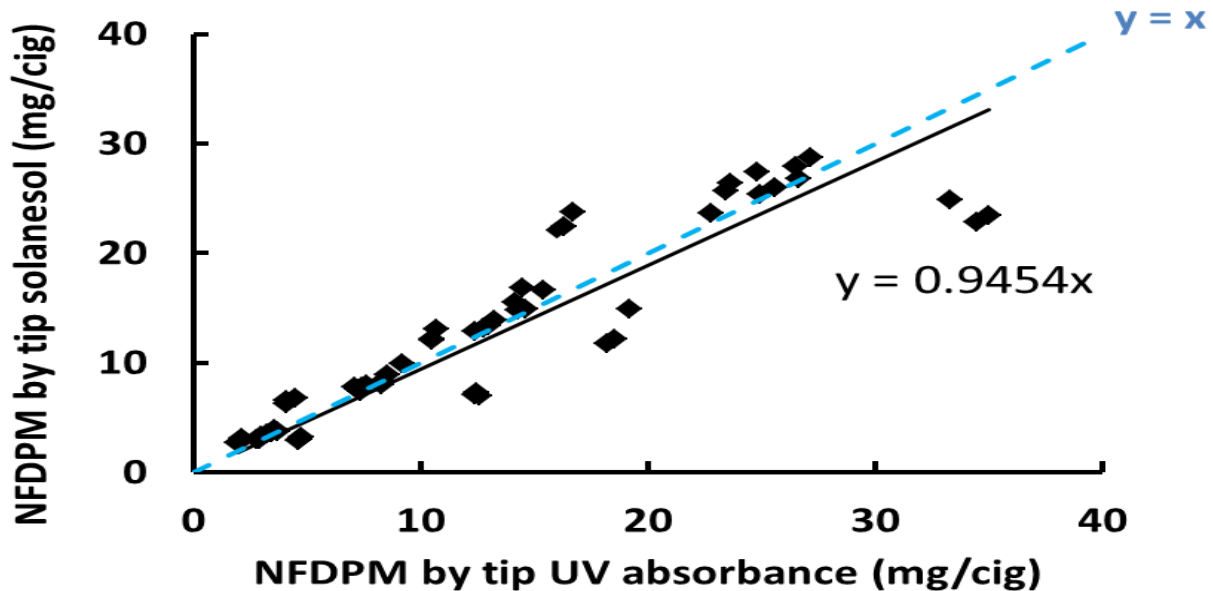


2013 Ring Trial Summary

- ❖ Estimated NFDPM values by tip solanesol are similar to estimated NFDPM by tip UV absorbance
- ❖ CV for reproducibility for estimated NFDPM by tip solanesol were in the range 9 to 13%, compared to 6 to 58% previously
- ❖ There is evidence that estimation of NFDPM is best completed using tip solanesol measurements
- ❖ However only five laboratories submitted data for NFDPM by tip solanesol



Estimated NFDPM by tip solanesol is similar to estimated NFDPM by tip UV absorbance



Data were shown to be normally distributed



Smoking behaviour sub group requested:

- ❖ CORESTA Recommended Method is written based on method for estimated nicotine yield by tip nicotine
- ❖ CORESTA Recommended Method is produced based on method for estimated NFDPM by tip solanesol
- ❖ Unable to recommend progression of estimated NFDPM by tip UV



Smoking behaviour sub group requested:

- ❖ Draft CRM will be forwarded to the Scientific Commission by end 2014
- ❖ A manuscript based on the ring trial results will be distributed within the Smoking Behaviour sub-group (by May 2015 meeting) followed by the Scientific Commission for intended publication in a peer-reviewed journal



*We wish to extend our thanks and appreciation
to all the participating laboratories*

Thank you for your kind attention

Questions welcomed !