Smoke Analytes Sub-Group (SMA) Annual Report 2019

CORESTA SSPT Conference

Hamburg, October 9, 2019

SMA SG Objectives

- To propose and maintain CORESTA Recommended Methods (CRMs) and related documents for the analysis of smoke constituents from combustible tobacco products.
- To organise interlaboratory testing related to Objective 1.

SMA at a glance

- **SG Coordinator**
  - Jana Jeffery, British American Tobacco Ltd, UK
- **SG Secretary**
  - Rana Tayyarah, ITG Brands LLC, USA
- **SC Liaison**
  - Martin Blumenstock, British American Tobacco Ltd, Germany
- **SG members and meetings**
  - Generally 30-40 participants
  - Usually two meetings per calendar year
  - Last meeting Richmond, Virginia, USA on 9th April 2019 (44 participants)
  - Next meeting hosted by PMI in Switzerland, week of 20th April 2020

Areas of work 2019

Area	Work items
Documents	• Technical Reports • CRM reviews • ISO standardisation support
Cigarettes	• Aromatic amines in MCS* by GC/MS (project 48) • Benzo[*a*]pyrene in MCS* by GC/MS (project 223) • Next analytes of interest – HCN, NOx
Cigars	• Collaborative study – B[*a*]P, TSNAs
Proficiency Testing	• Pilot study - HCN, NOx

**MCS – mainstream cigarette smoke*

Documents

Area	Activity	Status
Technical reports	• 2017 CS Aromatic amines by GC/MS • 2012 CS - CRM 58, CRM 70, CRM 74 • 2017 Joint experiment aromatic amines by LC-MS/MS • 2014 CS Phenols by HPLC-FLD • Comparison of TobLabNet methods and CRMs • 2018 Investigation – aromatic amines GC/MS	• Completed • Completed • Submitted* • In progress* • In progress* • Started*
Periodic CRM reviews	• CRM 78 - Phenols by HPLC-FLD • CRM 63 –TSNAs by GC/TEA	• Completed
Ad-hoc CRM review	• CRM 58 – B[a]P by GC/MS • CRM 70 – VOCs by GC/MS • CRM 74 - Carbonyls by HPLC/UV	• Completed

CS= Collaborative Study

**target completion end 2019*

ISO standardisation

- **ISO/TC 126/WG 10: Intense smoking regime**

  - **ISO 20778:2018, Cigarettes -- Routine analytical cigarette smoking machine -- Definitions and standard conditions with an intense smoking regime**

  - **ISO 20779:2018, Cigarettes -- Generation and collection of total particulate matter using a routine analytical smoking machine with an intense smoking regime**

ISO standardisation

Method	CRM	ISO smoking regime	ISO/TC 126/WG 10 ISO intense smoking regime
Selected VOCs by GC/MS	CRM 70	Published	DIS
Selected carbonyls by HPLC	CRM 74	Published	DIS
B[*a*]P by GC/MS (methanol)	CRM 58	Published	SMA project 223
B[*a*]P by GC/MS (cyclohexane)*	N/A	DIS	
TSNAs by LC-MS/MS	CRM 75	Published	DIS
Ammonia by IC	CRM 83	DIS	DIS
Phenolics by HPLC-FLD	CRM 78	DIS	DIS

**ISO/TC 126/WG 14*

Cigarettes: Aromatic amines by GC/MS (project 48)

- **Target analytes:** o-Toluidine, 2,6-Dimethylaniline, o-Anisidine, 1-Aminonaphthalene, 2-Aminonaphthalene, 3-Aminobiphenyl, 4-Aminobiphenyl

- **Full Collaborative Study, CS (2017)**
  - Results - r&R high and varied. Repeatability ranged between 15-64% of the mean and reproducibility between 32-193% of the mean

- **A small focus group established to investigate critical parameters of the method**
  - Internal standards, solvent standards, matrix effects and clean-up (2018)
  - Small scale CS on reference products (2019)

Cigarettes: Aromatic amines by GC/MS (project 48)

❖ r & R - 3R4F (ISO)

PAA	2019 Mini - Collaborative					
	n*	Mean	Repeatability		Reproducibility	
		ng/cig	ng/cig	%	ng/cig	%
o-TOL	5	37	8.2	23	35	48-95
o-ANIS	5	1.6	0.4	24	1.0	63
2,6-DMA	5	3.6	0.9	27	2.3	65
1-ANA	5	11	1.7	15	6.6	58
2-ANA	5	6.9	1.8	26	4.3	63
3-ABP	5	2.1	0.4	21	2.6	82-125
4-ABP	5	1.2	0.3	23	0.9	71

**number of data sets*

Cigarettes: Aromatic amines by GC/MS (project 48)

❖ r & R - 3R4F (ISO)

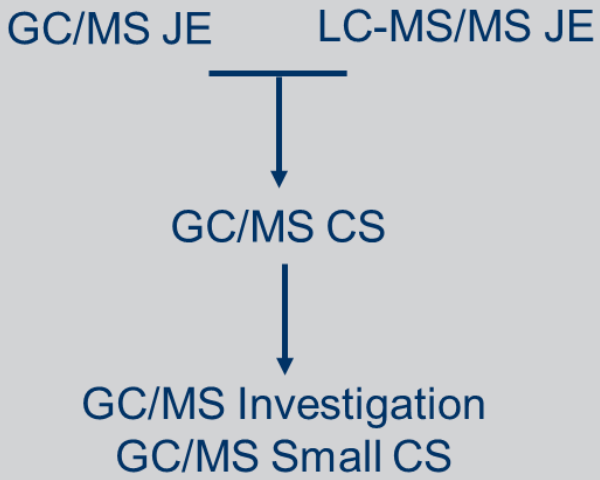
PAA	2019 Mini - Collaborative						2017 Collaborative					
	n*	Mean	Repeatability		Reproducibility		n*	Mean	Repeatability		Reproducibility	
		ng/cig	ng/cig	%	ng/cig	%		ng/cig	ng/cig	%	ng/cig	%
o-TOL	5	37	8.2	23	35	48-95	7	35	7.4	21	24	70
o-ANIS	5	1.6	0.4	24	1.0	63	8	1.5	0.4	26	1.6	101
2,6-DMA	5	3.6	0.9	27	2.3	65	6	2.8	1.2	44	1.8	63
1-ANA	5	11	1.7	15	6.6	58	8	11	2.2	20	6.6	59
2-ANA	5	6.9	1.8	26	4.3	63	9	6.2	1.8	29	4.6	75
3-ABP	5	2.1	0.4	21	2.6	82-125	10	1.7	0.5	27	1.5	91
4-ABP	5	1.2	0.3	23	0.9	71	10	1.1	0.7	64	1.4	130

**number of data sets*

Cigarettes: Aromatic amines by GC/MS (project 48)

Next steps

- Document the learnings and the method
- Project closure

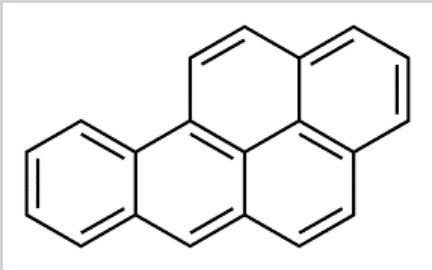


Cigarettes
Active work items

- Benzo[*a*]pyrene Collaborative Study (project 223)
- Next analytes for CRM development – HCN, NOx, PQS

B[*a*]P in mainstream smoke (project 223)

- B[*a*]P CS under intense smoking regime (ISO 20778)
- Comparison of two methods:
  - ISO 22634-1 (CRM 58) – methanol
  - ISO 22634-2 (developed in ISO TC 126/WG14) – cyclohexane
- Lead: Kentaro Eguchi



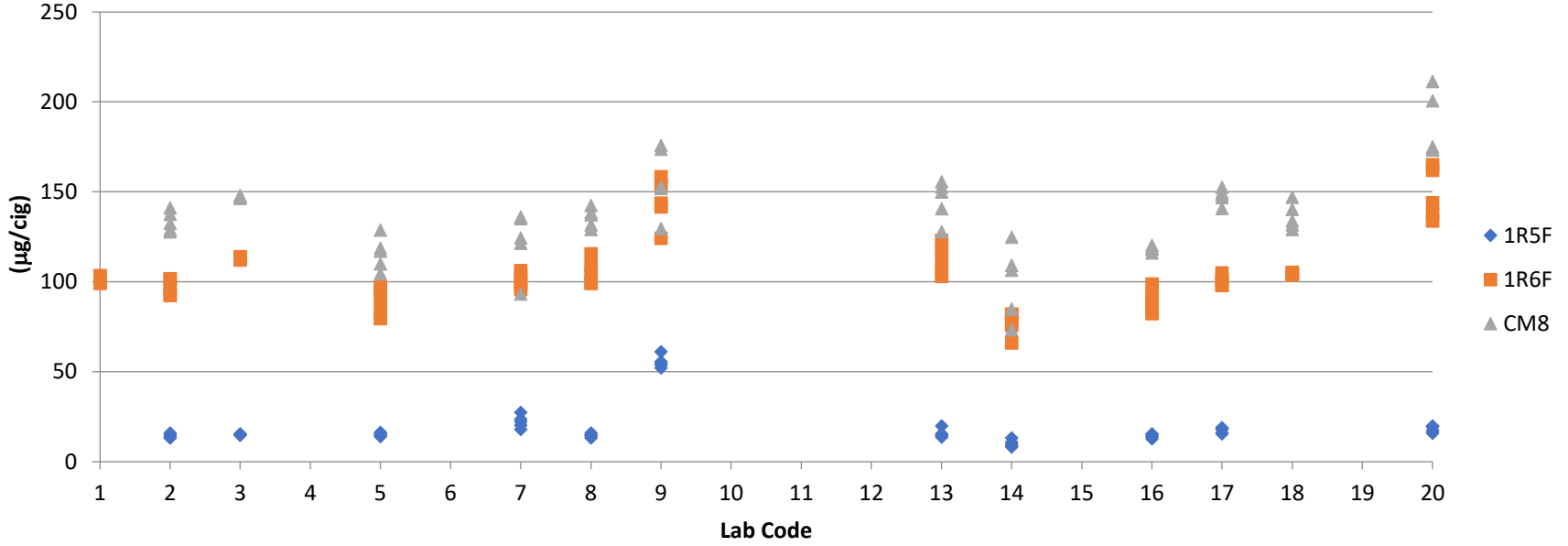
B[*a*]P in mainstream smoke (project 223)

- **Study set-up**
  - 12 laboratories
  - Six samples: 2 controls, 4 products, 5 replicates/sample
  - Methods: ISO 23906-1 and ISO 23906-2
  - Intense smoking regime (ISO 20778)
- **Timelines**
  - Samples distribution: by end 2019
  - Collaborative study: January – February 2020
  - Statistical analysis: March 2020
  - Review: next SMA meeting

New Analytes
HCN, NOX

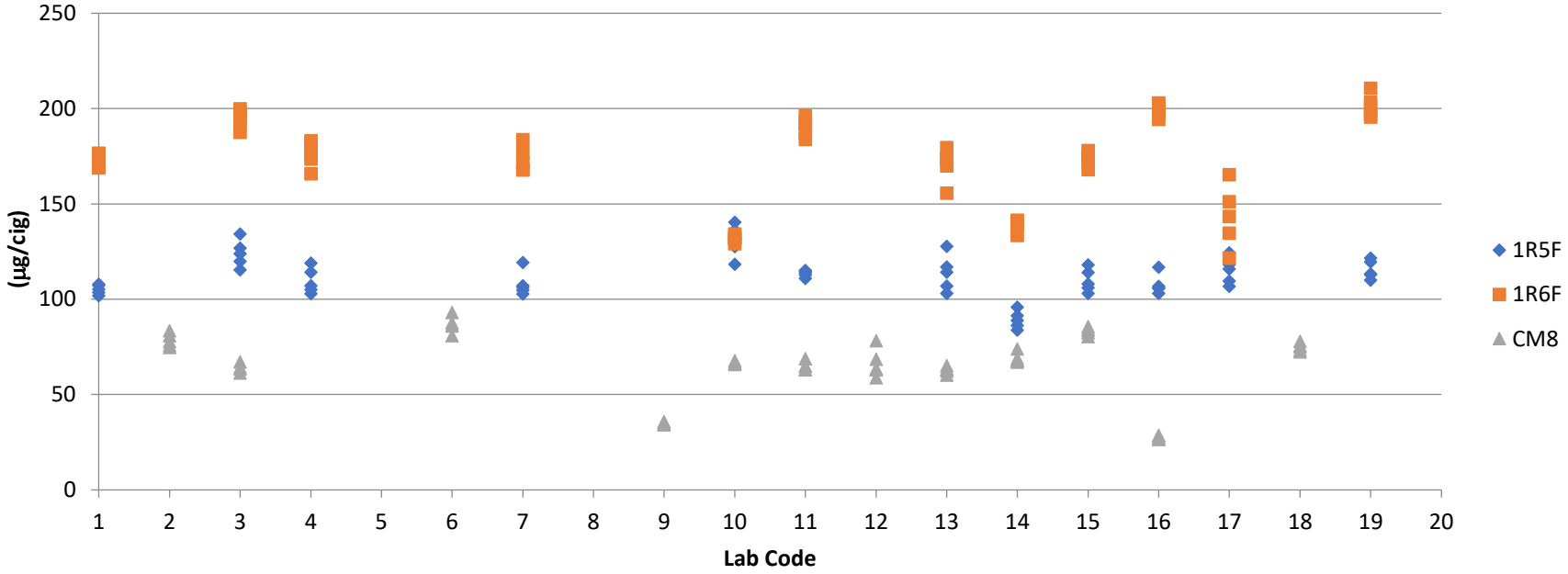
- Identified through a survey for priority analytes
- Method survey and data mining (Jannell Rowe)
  - 13 laboratories
- Proficiency Testing pilot with University of Kentucky (Rana Tayyarah, Huihua Ji)
  - 17 laboratories

PT – HCN ISO Regime



	1R5F	1R6F	CM8
Mean Values:	16 µg/cig	105 µg/cig	136 µg/cig

PT – HCN ISO Regime



Mean Values:

1R5F	1R6F	CM8
112 µg/cig	176 µg/cig	67 µg/cig

HCN in mainstream smoke

- **Study set-up**
  - In progress
- **Timelines**
  - Protocol distribution: January 2020
  - Joint Experiment: February - March 2020
  - Initial data review and analysis: spring SMA meeting

Cigarettes
Proficiency Testing

- Pilot on scope expansion successful
- In collaboration with University of Kenucky to recommend a scheme for the calendar based on the pilot
- Completion of the objective

Current activities

- Project 198 for testing CRMs for B[*a*]P and TSNAs on emissions from Kentucky reference cigars
- Timeline: 2Q 2020

Reference cigar	Description	Diameter
1C1 Cigar	Large machine made cigar	15.6
1C2 Cigar	Machine made little cigar (Filtered)	7.8
1C3 Cigarillos	Small machine made cigarillo	11.0
1C4 Cigar	Large machine made with natural wrapper	12.8

Future studies Cigarettes

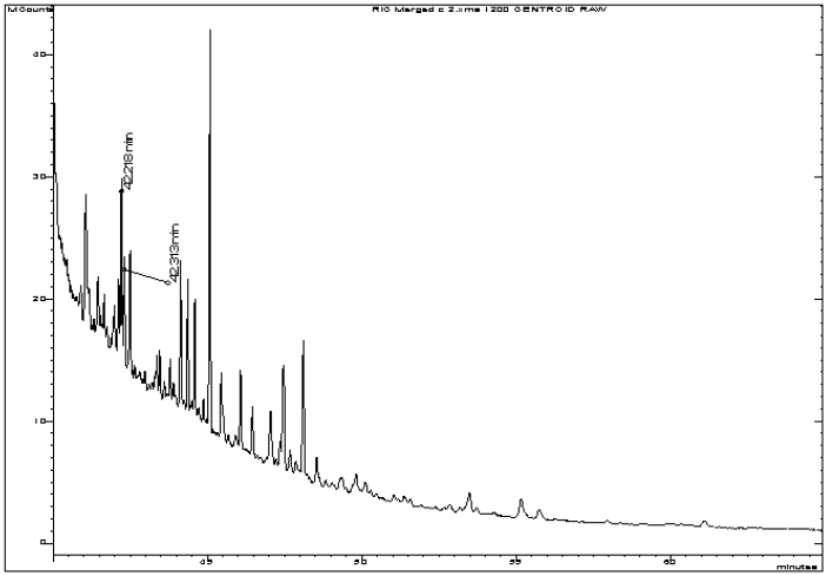
- **On-going survey for analytes of interest**
  - **NOx**
  - **PQS**
  - **B[*a*]P method improvement (CMR 58)**
  - **PAHs**

B[a]P method improvement / PAH method

- **CRM 58 improvement**
  - Selectivity/sensitivity
  - Sample preparation
  - GC column material (e.g. test PAH specific column)

- **Improved B[*a*]P method**
- **New method for a suite of PAHs**

Example of a chromatogram of a cigarette smoke extract



Future Studies Cigars

❖ Smoke methods for cigars

- ➢ Combined methods (B[*a*]P, TSNAs, VOCs)
  - ➢ Feasibility decision point for spring 2020 SMA meeting
- ➢ Analytes priorities survey

SMA celebrating 20 years!!!

Acknowledgements

- To all laboratories participating in SMA projects

- To all participants on SMA meeting, their contributions and engagement