Smoke Analytes Sub-Group (SMA) 2017 Report

Smoke Science and Product Technology Study Groups

Kitzbühel, October 11, 2017

Sub-Group name change

❖ **In January 2017, SG changed its name to Smoke Analtyes clearly indicate that its work is related to combustible products only**

SMA SG Objectives

❖ **To propose practical and robust recommended methods for smoke analytes for all combustible tobacco products.**

❖ **To organise and conduct periodically proficiency testing of smoke analytes other than TNCO.**

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SMA SG 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 Berlin, 2016 (34 participants)**
  - ▪ **Next meeting spring 2018**

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2017 activities

Area	Plan	Status
Documents	CRMs promotion to ISO standards • Selected carbonyls in mainstream cigarette smoke (MCS) – ISO/DIS • Selected volatiles in MCS – ISO/DIS ISO comparison study on B[a]P in MCS methods – ISO/FDIS	In progress according to ISO schedule
Cigarette projects	Project 48: Aromatic Amines Full Collaborative Study by GC-MS Aromatic Amines Pre-Study by LC-MS/MS	Completed

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Reviewed standards

- ❖ **WG14 – B[*a*]P in cigarette smoke**

  ISO/FDIS 22634-1 and ISO/FDIS 22634-2

  Reviewed by ISO secretary and is under last review

New standards

- ❖ **ISO 19290:2016 TSNAs in mainstream cigarette smoke by LC-MS/MS (based on CRM75)**

  Published

- ❖ **ISO/NP 21160 Carbonyls in mainstream cigarette smoke (based on CRM74)**
- ❖ **ISO/NP 21330 VOCs in mainstream cigarette smoke (based on CRM70)**

  Both approved as ISO/DIS

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❖ **Collaborative Study by GC/MS**

❖ **Pre-study by LC-MS/MS**

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Aromatic Amines by GC/MS Collaborative Study

❖ **Overview**

- **7 Aromatic Amines (FDA HPHC list): o-Toluidine, 2,6-Dimethylaniline, o-Anisidine, 1-Aminonaphthaline, 2-Aminonaphthaline, 3-Aminobiphenyl and 4-Aminobiphenyl**

- **Samples: 4 commercial products and 3 reference cigarettes**

- **Smoking regimes: ISO and Health Canada Intense**

- **5 replicates per sample and per smoking regime**

- **Randomised sample generation design**

- **GC-MS (NCI) method**

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❖ Products

Sample	Product characterisation	ISO tar yield (mg/cig)	Distributed by
Sample 1	Dark air-cured product	8-10	Imperial Tobacco
Sample 2	American blended product	4-6	Altria Client Services
Sample 3	Virginia blended product	4-7	PMI
Sample 4	Charcoal filtered / blended product	1-2	Japan Tobacco
3R4F	Kentucky Reference 3R4F	8	Participating laboratories sourced the product by themselves.
1R6F	Kentucky Reference 1R6F	8	
CM8	CM8 Test Piece	12-14	

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❖ 11 participating laboratories

Altria Client Services, USA
BAT, Brazil
Enthalpy Analytical, USA
Global Laboratory Services, USA
Japan Tobacco, Japan
KT&G, South Korea
Labstat, Canada
Liggett Group, USA
PMI, Brazil
PMI, Sampoerna
R.J. Reynolds, USA

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Statistical Analysis

❖ **Outlier testing**

❖ **r & R**

- **Repeatability between 15-64% of mean**

- **Reproducibility 32-193% of mean**

❖ **Smoking machines comparison**

- **No difference between linear and rotary smoking machines**

- **Consistent number of cigarettes smoked**

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1-aminonaphthalene

ISO smoking regime

Analyte=1-aminonaphthalene Regime=ISO
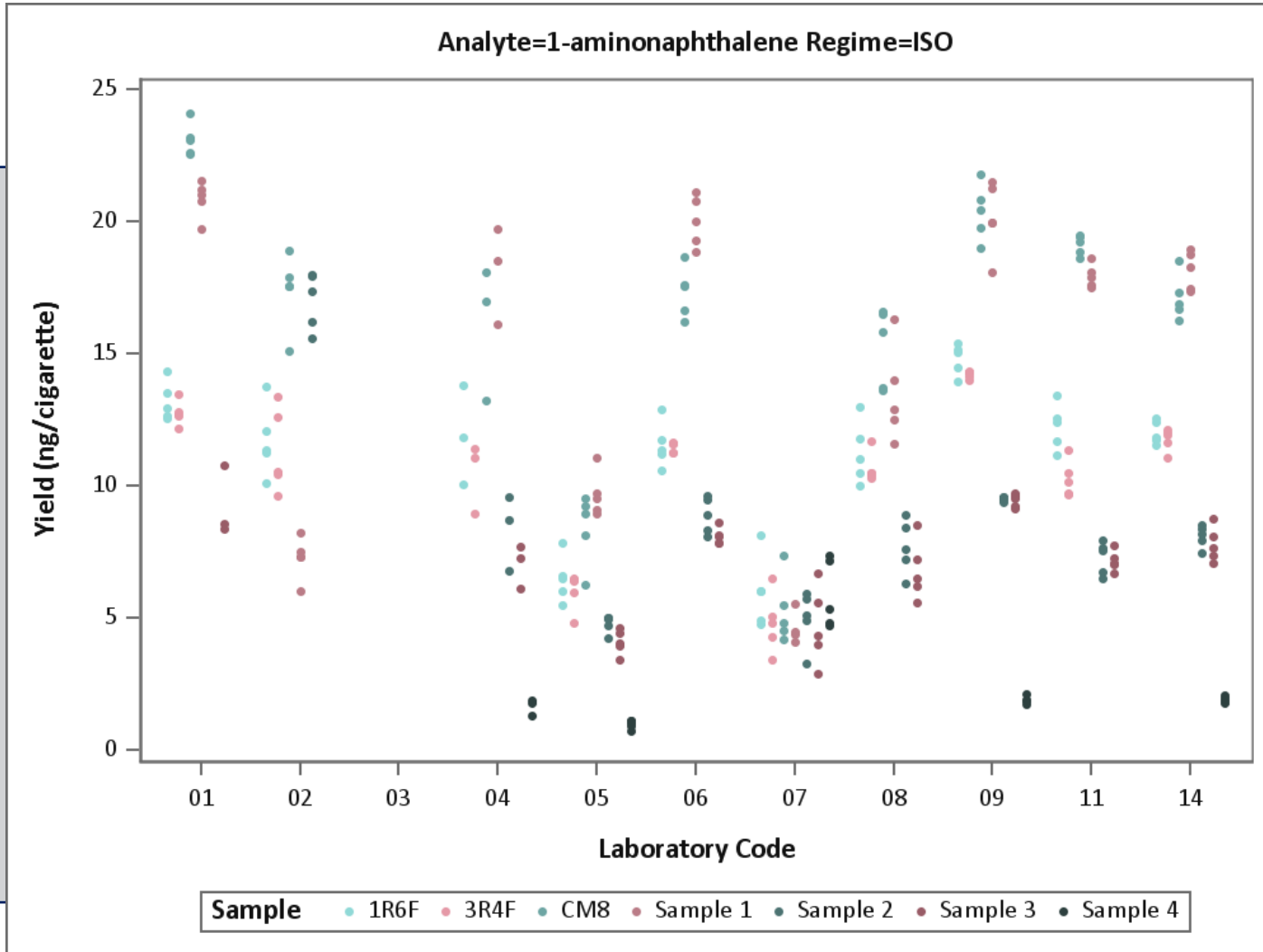
1-aminonaphthalene

3R4F

ISO smoking regime

Analyte=1-aminonaphthalene Sample=3R4F Regime=ISO

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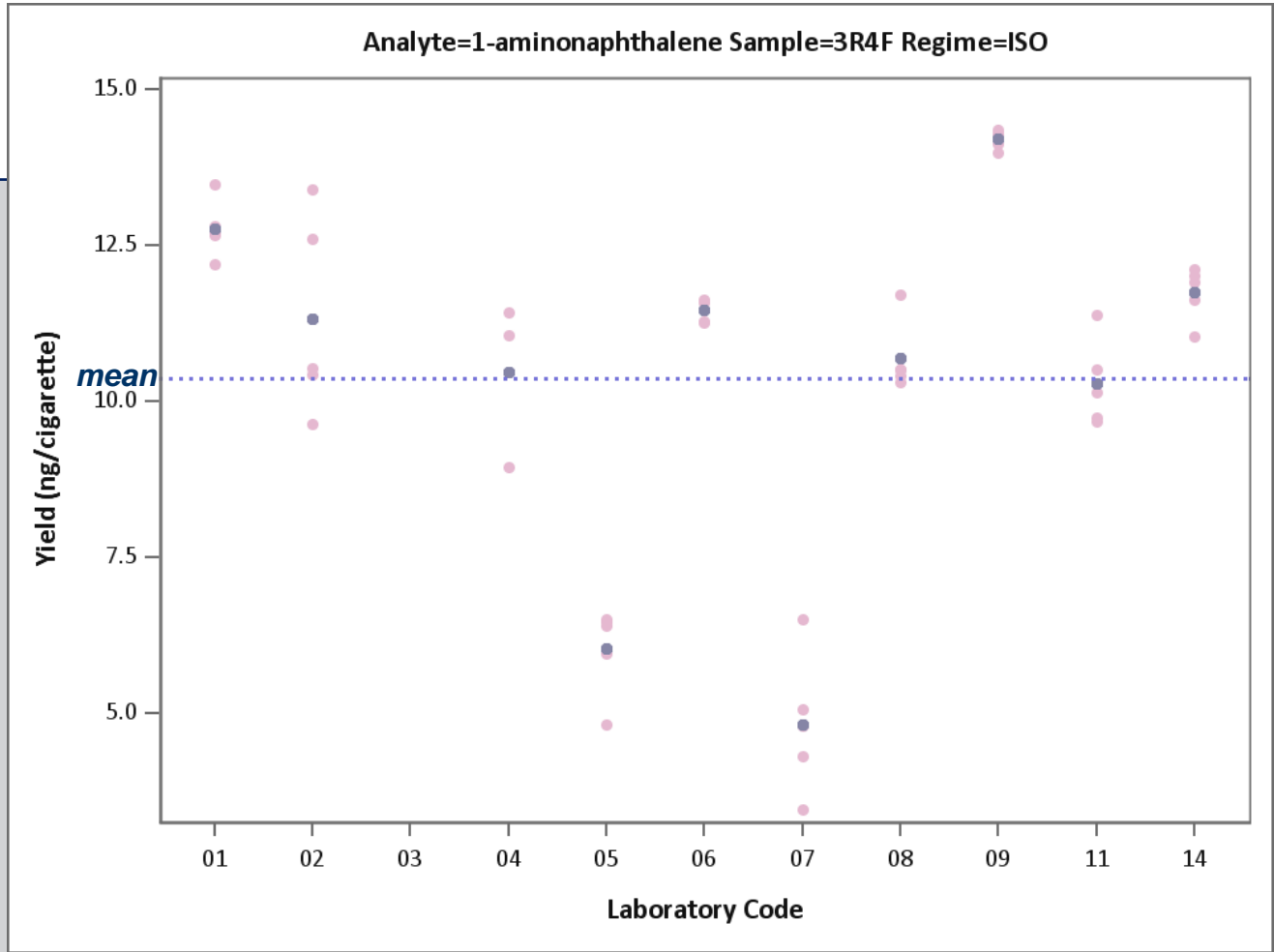
❖ **Effect of acidic environment for the determination of aromatic amines when using mass labelled (deuterated) internal standards (IS)**

▪ **Comparison of two sets of working standards prepared in (i) 0.6M HCl and (ii) deionised water during simulation of the analytical method**

▪ **Statistical analysis ⇒ no difference**

❖ **Storage stability of standard solutions**

▪ **Working standards – 1 week, refrigerator**

▪ **Internal standards solution – 1 month, freezer**

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❖ **GC/MS method**

- ▪ **Technical report from Collaborative Study. Timeline - Q1 2018**

- ▪ **Project extension. Small focus group to investigate robustness of derivatisation. Continue with all 7 analytes. Timeline - share results with the SG at 2018 spring meeting**

❖ **LC-MS/MS method**

- ▪ **Technical report from the work conducted so far. Timeline - Q1 2018**

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Ongoing Activities

Area	Plan	Status
Documents	Ammonia in Mainstream Smoke (based on CRM 83)	Started
	Selected Phenolic Compounds in MCS (based on CRM 78), project 162	Project launched
	CRMs promotion to ISO standards ISO comparison study	In progress according to ISO schedule
	Periodic CRMs for review	Projects launched
Strategy	Develop 2-3 year plan	Completed

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Technical Reports

❖ **Ammonia in Mainstream Cigarette Smoke by Ion Chromatography**
- **CRM 83 published in July 2016**
- **In progress**
- **Completion date end 2017**

❖ **Selected Phenolic Compounds in Mainstream Cigarette Smoke by HPLC-FLD**
- **CRM 78 published in July 2014**
- **Project 162 launched September 2017**
- **Completion date Q2 2018**

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Periodic CRMs review

CRM	Title	Issued	Review due
CRM 58	Benzo[a]Pyrene by GC/MS	07/2014	2017
CRM 63	TSNAs by GC/TEA	06/2005	
CRM 70	Selected VOCs by GC/MS	03/2013	
CRM 74	Selected Carbonyls by HPLC	03/2013	
CRM 75	TSNAs by LC-MS/MS	06/2012	2020
CRM 78	Phenols by HPLC/FLD	08/2014	2018
CRM 83	Ammonia by IC	05/2017	2019

- ❖ **Project 153 (CRM 74) - in progress**
- ❖ **Project 163 (CRM 58, CRM 63 and CRM 70) - launched**
- ❖ **Timeline 11/2017**

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Future studies

- ❖ **Cigars**

- ❖ **Cigarettes – methods and analytes**

- ❖ **Proficiency testing**

- ❖ **Documents**

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❖ **Smoke methods for cigars**

- **Introduction to cigars – insights from Bob van Mierlo (Cigar Smoking Methods SG, CSM)**
  - **Discussion around CSM TNCO Collaborative**
- **Small scale proficiency study for B[*a*]P and TSNAs on 2-3 products**
  - **NWIP**

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❖ **Cigarettes – methods and analytes**

- ▪ **Comparison of TobLabNet methods and CRMs (review).Timeline – 2017**
- ▪ **Discussion about further candidate analytes/analyte groups for future SG work**

❖ **Proficiency testing on existing in-house methods**

- ▪ **University of Kentucky PT programme update (Huihua Ji)**
- ▪ **Opportunities for extending of the PT programme scope**
- ▪ **PT organized within SMA SG – proposal including timelines for further discussion in spring 2018 meeting**

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Action list

Area	Item	Plan
Projects	Cigarettes – AA GC/MS Study	NWIP 4Q17 Complete 2Q18
	Cigar – MMC BaP/TSNA PT	NWIP 4Q17
PT	Develop Templates & Proposed Calendar	2Q 18
Documents	Project 163 - 2017 CRM Review (B[a]P, TSNAs, VOCs)	Complete 4Q17
	TR Ammonia 2015 Collaborative	Complete 4Q17
	Project 162 - TR Phenols 2014 Collaborative	Complete 2Q18
	ISO Promotion for Smoke Ammonia, Phenols	NWIP 3Q 2018
	Project 48 TRs Aromatic Amines GC/MS and LC-MS/MS	Complete 2Q18
	2018 CRM Review	Complete 2Q18

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Acknowledgements

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- ❖ **To all laboratories who volunteer with distribution of products for our studies**

- ❖ **To all laboratories participating in SMA projects**

- ❖ **To statistical support for Aromatic Amines project – Eleni Mavropoulou**

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Thank you for your attention

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Aromatic Amines Data Comparison

ANALYTE	REGIME	CIG	Mean [ng/cig]	r [ng/cig]	R [ng/cig]	Remarks
1 - AN	ISO	KR 3R4F	10.2	1.9	9.2	CS 2017 GC/MS
			10.9	*2.6**	*9.2**	JE 2016 LC-MS/MS
			11.8	3.7	15.9	JE 2015 all methods
	HCl		23.9	*6.2*	*23.4*	CS 2017 GC/MS
			26.6	4.3	9.2	JE 2016 LC-MS/MS
			22.9	4.4	11.8	JE 2015 all methods

ANALYTE	REGIME	CIG	Mean [ng/cig]	r [ng/cig]	R [ng/cig]	Remarks
4 - ABP	ISO	KR 3R4F	1.0	0.2	1.3	CS 2017 GC/MS
			0.7	0.2*	0.3*	JE 2016 LC-MS/MS
			1.3	0.5	2.5	JE 2015 all methods
	HCl		2.7	0.7	3.2	CS 2017 GC/MS
			1.7	0.3	0.4	JE 2016 LC-MS/MS
			2.9	1.2	2.9	JE 2015 all methods

Extremely limited data set - indicative value only

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❖ **Example on 3R4F**

- ▪ **7 laboratories linear SM**
- ▪ **4 laboratories rotary SM**

Comparison of Linear VS Rotary Machines			
Analyte	**Regime**	**Effect**	**P-value**
1-aminonaphthalene	HCI		0.80
	ISO		0.61
2,6-dimethylanilin	HCI		0.84
	ISO		**0.00**
2-aminonaphthalene	HCI		0.97
	ISO		0.85
3-aminobiphenyl	HCI		0.39
	ISO	Smoking Machine	0.33
4-aminobiphenyl	HCI		0.95
	ISO		0.92
o-anisidine	HCI		0.97
	ISO		0.85
o-toluidine	HCI		**0.02**
	ISO		0.74

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