Special Analytes Sub-Group Report Berlin 2016

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Overview

- Terms of Reference
- Sub-Group Meetings
- Achievements and Status of Projects
- Joint Experiments on Aromatic Amines
- New Collaborative Study on Aromatic Amines
- Outlook on future NWIP

Terms of Reference

- To propose practical and robust recommended methods for smoke analytes
- To organise and conduct periodically collaborative/proficiency testing of smoke analytes other than TNCO

Special Analytes Sub-Group Meetings

- Lausanne on 28th April 2015 was hosted by PMI; 31 Participants
- Berlin on 8th October hosted by CORESTA; 34 Participants
- Next Meeting will take place in Sept/Oct 2017

SPA SG Achievements and Activities

❖ Achievements

Project No.	Activity	Leader	Time
46	CRM83 Ammonia in mainstream smoke recently published	M. Intorp / J.Ticha	August 2016

❖ On-Going

Project No.	Activity	Leader	Time
48	Prestudies on Aromatic Amines 4 methods being evaluated (GC-MS and LC-MS/MS)	M. Intorp / J.Ticha	Oct 2016

Status of ISO Projects

Reviewed standards

- ***WG14 BaP in cigarette smoke***
  - ✓ **Established to undertake collaborative study with alternative extraction solvent (cyclohexane)**
  - ✓ **CD 22634-2 approved with minor comments, amended, and to be published as DIS.**

New Standards

- **ISO/DIS 19290 TSNAs in mainstream cigarette smoke by LC-MS/MS; Based on CRM75 - DIS approved Nov 2015, IS prepared, close to publication**
- **ISO/NP 21160 Carbonyls in mainstream cigarette smoke; Based on CRM74 – Approved as WD**
- **ISO/NP 21330 VOCs in mainstream cigarette smoke; Based on CRM70 – Approved as WD**

Joint Experiments
Comparison of various Aromatic Amines Methods

- **Performance check of GC-MS method provided by JTI/Ökolab**
- **Comparison of two GC/MS(NCI) methods – Altria and BAT**
- **Performance of LC-MS/MS method provided by CNTC**
  - *Analytes: o-toluidine, 2,6-dimethylanilin, o-anisidine, 1-aminonaphthalene, 2-aminonaphthalene, 3-aminobiphenyl and 4-aminobiphenyl*
  - *Sample: 3R4F*
  - *Smoking regimes: ISO and Health Canada Intense T-115 (HCI)*
  - *Five replicates per method and per smoking regime.*

Aromatic amines
Summary of activities

JE	Method	From	Activity	Observations
1	GC/MS	JTI/Ökolab	Familiarisation study	Challenging to adopt widely – issues with sample throughput
2	2 GC/MS methods	Altria BAT	Familiarisation study Comparison of EI and NCI Selection of the method	NCI better than EI BAT method
3	GC/MS	BAT	Derivatisation experiments Solvent comparison Increase of no. of IS (from 4 to 6)	DCM and DCE seem comparable Derivatisation conditions seem comparable
3	LC-MS/MS	CNTC	Familiarisation study Off-line SPE clean up evaluation	Not enough results available for any reliable evaluation

Aromatic Amines – Data Comparison

ANALYTE	REGIME	CIG	Mean [ng/cig]	r [ng/cig]	R [ng/cig]	Remarks
1 - AN	ISO	KR 3R4F	12.19	*4.34**	*6.44**	JE 2016 GC/MS
			10.88	*2.57**	*9.18**	JE 2016 LC-MS/MS
			11.79	3.72	15.93	JE 2015 all methods
			9.26	12.46	12.54	Altria Method (NCI)
			13.1	10.55	10.7	BAT Brazil Method (NCI)
	HCI		25.08	9.44	10.64	JE 2016 GC/MS
			26.59	4.33	9.23	JE 2016 LC-MS/MS
			22.94	4.42	11.76	JE 2015 all methods
			17.89	39.5	39,83	Altria Method (NCI)
			24.52	21.61	22.13	BAT Brazil Method (NCI)

Limited data set - indicative value only

Aromatic Amines – Data Comparison

ANALYTE	REGIME	CIG	Mean [ng/cig]	r [ng/cig]	R [ng/cig]	Remarks
4 - ABP	ISO	KR 3R4F	1.41	*1.09**	*1.32**	JE 2016 GC/MS
			0.72	*0.22**	*0.27**	JE 2016 LC-MS/MS
			1.33	0.48	2.45	JE 2015 all methods
			1.14	1.04	1.11	Altria Method (NCI)
			1.44	1.92	1.96	BAT Brazil Method (NCI)
	HCI		3.01	1.65	2.18	JE 2016 GC/MS
			1.71	0.32	0.41	JE 2016 LC-MS/MS
			2.85	1.22	2.91	JE 2015 all methods
			2.54	5,23	5,27	Altria Method (NCI)
			3.17	2.87	2.92	BAT Brazil Method (NCI)

Limited data set - indicative value only

Aromatic amines
Next steps

- **GC/MS method**
  - Preparation of Collaborative Study (Oct/Nov16)
  - Familiarisation with method by all participants (Nov16/Feb17)
  - Shipment of samples (Dec16/Feb17)
  - Laboratories to generate data and report results (Mar/July17)
  - Statistical evaluation (Aug17)

- **LC-MS/MS method**
  - Smaller group to work on weaknesses/improvements of circulated method and report progress to SG

Outlook

New project proposals

- Proficiency testing (PT) on existing in-house methods, ISO, CRMs, SOPs should include B[a]P (1), Carbonyls (8), Selected Volatiles (5), TSNA (4), Phenols (7), Ammonia (1)
  - Majority of labs participate in PT offered by University of Kentucky
  - SG member from University of Kentucky agreed to clarify by end 2016 with PT team to possibly extend scope of current/future studies on:
    - All compounds included in CRMs
    - More condensate levels (1R6F)
- For further discussion on NWIP information presented on combined methods (VOC/Carbonyls/SV) and TobLabNet SOP VOC/Carbonyls will be provided

Acknowledgements

- We usually have at least 20 laboratories participating in current collaborative studies

- Thanks to all the current and previous participants for their lively discussion and openness - without whom CORESTA would not be able to deliver such robust and reliable data

Thank you for your attention!