Special Analytes Sub-Group Report Jeju 2015

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Secretary: Jana Ticha

Agenda

❖ **Terms of Reference**

❖ **Previous work Summary**
  - ➢ **Status on CRMs with regard to upcoming regulations**
  - ➢ **ISO issues**

❖ **Sub-Group Meetings**

❖ **Collaborative study on ammonia**
  - ➢ **Study results & Status of CRM**

❖ **Joint experiment on aromatic amines**
  - ➢ **Results & Discussion**
  - ➢ **Next steps**

❖ **Future Work Items**

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❖**To propose practical and robust recommended methods for smoke analytes**

➢ Develop CRMs for ammonia and aromatic amines as agreed by the Scientific Commission.

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Special Analytes Sub-Group Meetings

❖ **Hangzhou Meeting on 30th April 2015 was hosted by China Tobacco Zhejiang Industrial Co. ; 31 Participants**

❖ **Jeju Island on 4th October was hosted by KT&G; 30 Participants**

❖ **Next Meeting will take place end of April 2016**

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Previous Work Summary - ISO

❖ **WG14 Benzo[a]pyrene in cigarette mainstream smoke**

➢ **Revision of ISO 22634-**

   Collaborative study was conducted with 11 laboratories
      =>Results are equivalent between both methods (ISO 22634 and ISO 22634-2)
   Next is to prepare final report and committee draft (CD) for ISO voting
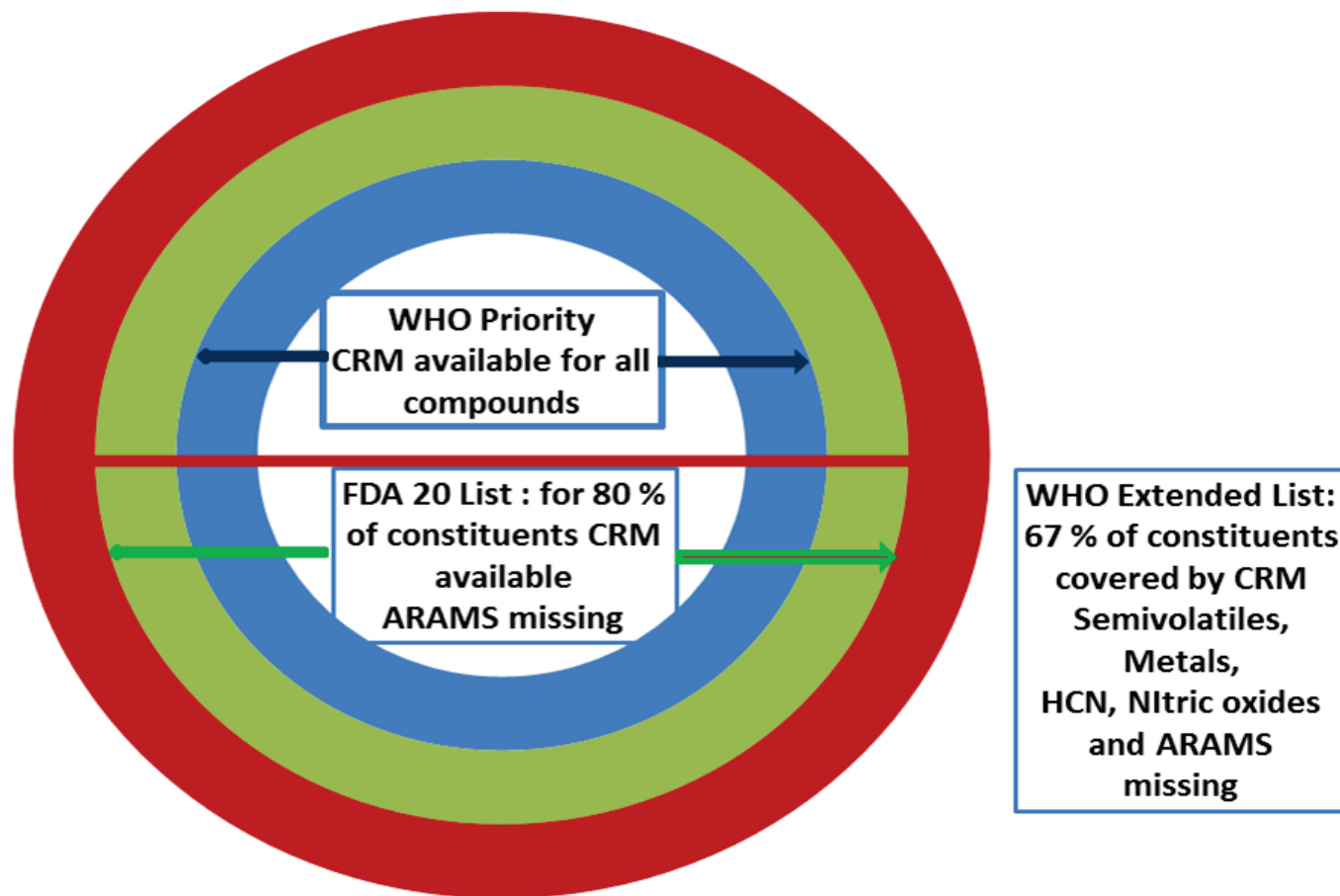
❖ **DIS Status TSNA Method**

   ➢ **ISO/DIS 19290: Cigarettes – Determination of TSNA in MS cigarette smoke – method using LC-MS/MS**

❖ **New Work Item Proposals:**

   ➢ **CRM 70 and 74 methods were submitted to ISO secretariat after removing HCI related parts from the method drafts**

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WHO Priority CRM available for all compounds

FDA 20 List : for 80 % of constituents CRM available ARAMS missing

WHO Extended List: 67 % of constituents covered by CRM Semivolatiles, Metals, HCN, NItric oxides and ARAMS missing

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Ammonia – Collaborative Study

❖ **Results Overview**

❖ **Presentation of statistics**

❖ **Next Steps**

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Ammonia – Learnings from various studies

- ➢ **Cooling of samples in auto sampler highly recommended as well as injections within 24 h after extraction of CFP**

- ➢ **Extraction with lower acid concentration may also limit artificial generation of ammonia**

- ➢ **Investigated trapping systems showed no differences in yield**

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Collaborative Study Ammonia - Participating Laboratories

Data received from 17 laboratories

- ❖ Altria, USA
- ❖ Arista, USA
- ❖ BAT, Brazil
- ❖ CNTC, Anhui, China
- ❖ CNTC, Beijing, China
- ❖ CNTC, Quality, China
- ❖ CNTC, ZTRI, China
- ❖ Enthalpy, USA
- ❖ Essentra, UK,

- ❖ Global, USA
- ❖ ITG, France
- ❖ JT, Japan
- ❖ JTI, Austria
- ❖ KT&G, Korea
- ❖ Labstat, Canada
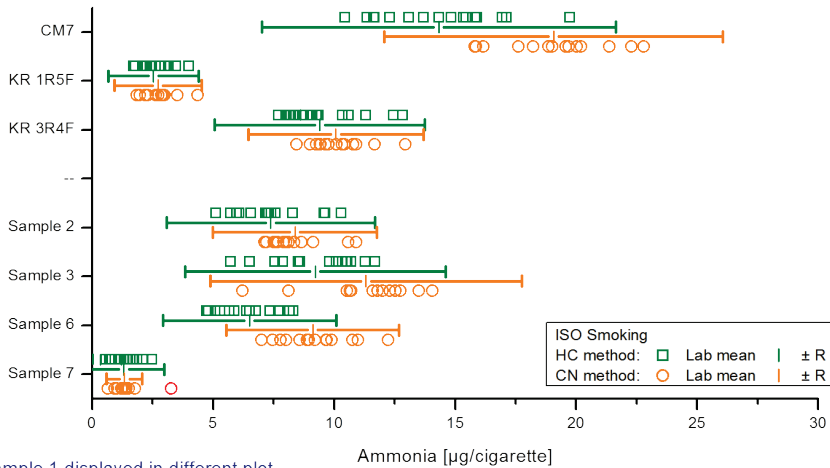- ❖ PMI, Brazil
- ❖ RJReynolds, USA

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Sample list overview

Sample	Product description	Tar yield (mg)	Manufacturer
Sample 1	Dark air-cured product	8.2 mg	ITG Altadis
Sample 2	American blended product	8.0 mg	BAT Germany
Sample 3	American blended product	6.5 mg	PMI Neuchatel
Sample 6	Virginia blended product	9.4 mg	CNTC, China
Sample 7	Charcoal filtered / blended product	1.7 mg	JT, Japan
Control 1	Kentucky Reference 3R4F	8.2 mg	University of Kentucky
Control 2	Kentucky Reference 1R5F	1.7 mg	University of Kentucky
Control 3	CM7 Test Piece	14.3 mg	Borgwaldt

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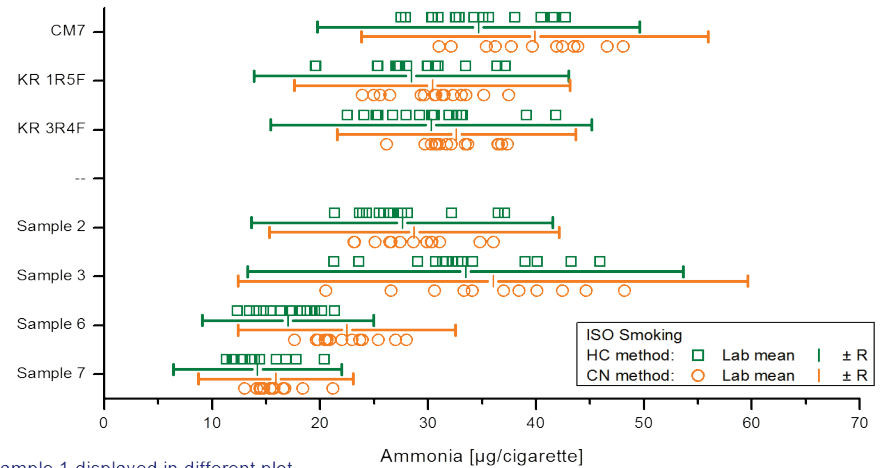Cooperation Centre for Scientific Research Relative to Tobacco

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Ammonia - mean yield plots
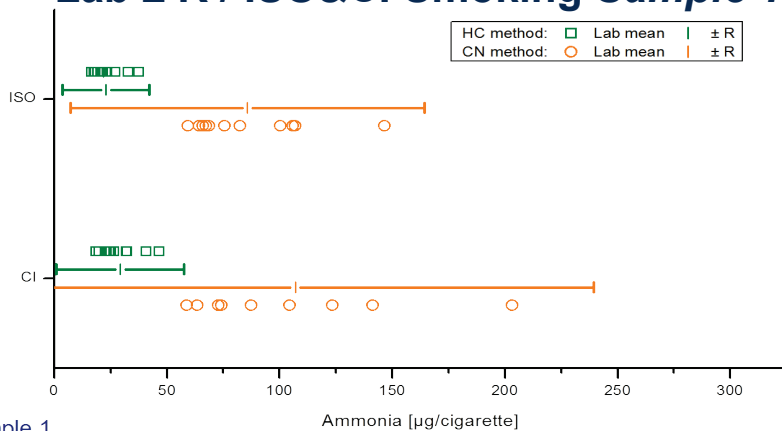
Lab ± R / ISO Smoking

Sample 1 displayed in different plot

Lab ± R / CI Smoking

Sample 1 displayed in different plot

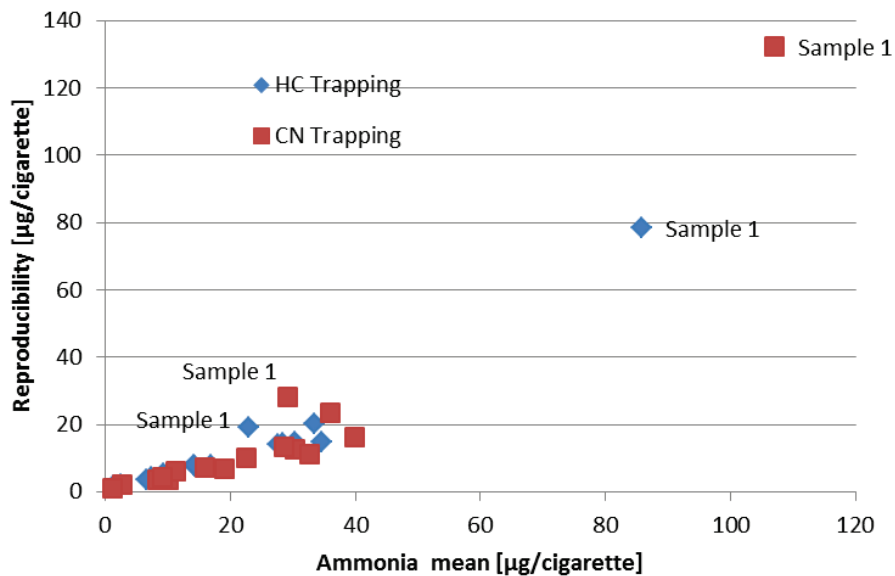## Lab ± R / ISO&CI Smoking *Sample 1*

Sample 1

➢ANOVA analysis indicated no significant differences between both trapping systems.
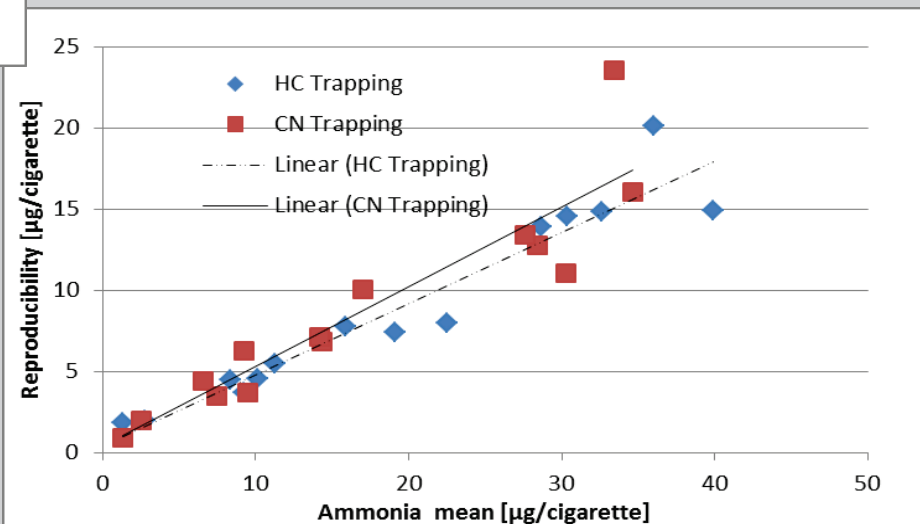
Ammonia - Comparison of R across labs

➢ **The reproducibility and repeatability data observed for Sample 1 indicated a significantly higher variability for the CN trapping system.**

➢ **When removing Sample 1 on average, the CN method delivers r & R values comparable to the HC method .**

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Ammonia Recommended Method – Next steps

➢ **Amended method draft to be circulated within SG to ask for comments and corrections.**

➢ **Final version will be distributed for approval to SG, SC and CORESTA Board.**

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Aromatic Amines
2015 Joint Experiment

❖ **Study Protocol – Joint Experiment**

➢ 2 Reference cigarettes (Ky 1R5F, Ky 3R4F)

➢ 7 Aromatic Amines (FDA List):

▪ o-Toluidine, 2,6-Dimethylaniline, o-Anisidine, 1-Aminonaphthaline, 2-Aminonaphthaline, 3-Aminobiphenyl and 4-Aminobiphenyl

➢ LC-MS/MS method

➢ GC-MS method

❖ **Statistical evaluation of the Joint Experiment results**

➢ Majority of results for all analytes within 2-fold z-scores

➢ Data outside 2-fold z-scores allocated to both methods

❖ **Mean values obtained for Ky 3R4F comparable with data from former Joint Experiments independent from applied method**

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❖ **Participating laboratories: 15**

	Total	GC/MS	LC/MS/MS
Data sets received	18	10	8
Proposed method	10	4	6
Own method	8	6	2

❖**Statistical evaluation of the Joint Experiment results**
  ➢Majority of results for all analytes within 2-fold z-scores
  ➢Data outside 2-fold z-scores allocated to both methods
❖**Mean values obtained for Ky 3R4F comparable with data from former studies and data mining independent from applied method**

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ANALYTE	REGIME	CIG	Mean [ng/cig]	r [ng/cig]	R [ng/cig]	Remarks
1 - AN	ISO	KR 3R4F	11.79	3.72	15.93	JE 2015 all methods
		KR 3R4F	11.97			Data Mining 2012
		KR 2R4F	11.90	1.51	8.56	JE 2007 - HFBA
1 - AN	HCl	KR 3R4F	22.94	4.42	11.76	JE 2015 all methods
		KR 3R4F	22.47			Data Mining 2012
2 - AN	ISO	KR 3R4F	6.88	1.04	5.66	JE 2015 all methods
		KR 3R4F	7.56			Data Mining 2012
		KR 2R4F	7.68			JE 2007 - HFBA
2 - AN	HCl	KR 3R4F	13.76	2.74	14.36	JE 2015 all methods
		KR 3R4F	14.28			Data Mining 2012

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ANALYTE	REGIME	CIG	Mean [ng/cig]	r [ng/cig]	R [ng/cig]	Remarks
3 - ABP	ISO	KR 3R4F	1.96	0.62	2.62	JE 2015 all methods
		KR 3R4F	1.96			Data Mining 2012
		KR 2R4F	2.21	0.23	1.70	JE 2007 - HFBA
3 - ABP	HCl	KR 3R4F	4.33	0.81	2.83	JE 2015 all methods
		KR 3R4F	4.4			Data Mining 2012
4 - ABP	ISO	KR 3R4F	1.33	0.48	2.45	JE 2015 all methods
		KR 3R4F	1.34			Data Mining 2012
		KR 2R4F	1.51	0.17	0.85	JE 2007 - HFBA
4 - ABP	HCl	KR 3R4F	2.85	1.22	2.91	JE 2015 all methods
		KR 3R4F	3.07			Data Mining 2012

Aromatic amines
Summary and next steps

GC-MS	LC-MS/MS
▪ Method throughput ▪ Requirement for adjustment of number of cigarettes smoked ▪ A single point calibration curve ▪ Only EI mode	▪ High method throughput ▪ Method not validated for 2,6-dimethylanilin ▪ Sensitivity?

Decision on the next steps

- **GC/MS: New candidate methods – less steps in clean-up**

  ➢ **Validation step for three compounds**

  ➢ **Working group volunteered to perform small scale study to test method**

- **LC-MS/MS: Adjusted to the compounds of interest**

  ➢ **Method will be improved and distributed**

  ➢ **Working group volunteered to perform small scale study to test method**

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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**

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Thank you for your attention!

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