



Tobacco and Tobacco Products Analytes Sub-Group (TTPA) Annual Report

CORESTA Congress – Kunming, China

October 24, 2018



- ❖ **Coordinator and Scientific Commission Liaison:**
 - Karl Wagner, Ph.D. (Altria Client Services LLC, United States)
- ❖ **Secretary:**
 - Johan Lindholm, Ph.D. (Swedish Match, Sweden)
- ❖ **Established in November 2008**
- ❖ **Typically two meetings per year:**
 - ~ 50 attendees
 - ~ 40 companies represented
- ❖ **Meetings since 2017 CORESTA Congress, Kitzbühel**
 - 22nd meeting - Guildford, UK (April 2018)
 - 23rd meeting - Kunming, China (October 2018)



Revised Objectives

- 1) To propose and maintain CORESTA Recommended Methods (CRMs) and related documents for the analysis of tobacco and unburned tobacco products.**
- 2) To organise interlaboratory testing related to Objective 1.**
- 3) To organise the manufacture of and maintain smokeless tobacco reference products.**



CORESTA Reference Products

❖ Smokeless Tobacco Reference Products (CRPs)

➤ Coordinated production in 2009 and 2016

- CRP 1.1: Swedish style snus pouch
- CRP 2.1: American-style loose moist snuff
- CRP 3.1: American-style loose dry snuff powder
- CRP 4.1: American-style (chopped) loose-leaf chewing tobacco

➤ Distributed by North Carolina State University



Supply of 2009 CRPs

	Dec 2009	Dec 2010	Dec 2011	Dec 2012	Dec 2013	Dec 2014	Dec 2015	Dec 2016	Dec 2017	Sept 2018
CRP 1	7000	6135	5443	4895	3975	3242	1992	1195	1160	1134
CRP 2	7020	6363	6093	5082	4453	3680	1723	620	592	576
CRP 3	7040	6653	6207	5520	5113	4691	4191	2169	2063	2015
CRP 4	7000	6806	6567	6374	6236	6040	5686	2309	2278	2218



Supply of 2016 CRPs

	Apr 2016	Dec 2016	Dec 2017	Sept 2018
CRP 1.1	10008	9312	8873	8475
CRP 2.1	10000	9061	8285	7577
CRP 3.1	7000	6340	6040	5773
CRP 4.1	7200	6769	6563	6280



❖ Collaborative studies to assess stability:

- Nicotine
- TSNAs
- pH
- Moisture (oven volatiles)



Ground Tobacco Reference Products

University of Kentucky, Center for Tobacco Reference Products



Dark Fire Cured



Flue Cured



Dark Air Cured



Oriental



Burley



Flavored Cigar Filler



Unflavored Cigar Filler



Technical Reports Published (past 12 months)

- 1) Select Carbonyls in Tobacco and Tobacco Products - 2016/2017 Collaborative Study (Jan 2018)**
- 2) 2017 Collaborative Study on Ammonia and Benzo[a]pyrene in Tobacco Products (Mar 2018)**
- 3) Water Activity Proficiency Study (Aug 2018)**
- 4) Water and Moisture (OV) Collaborative Study (Aug 2018)**

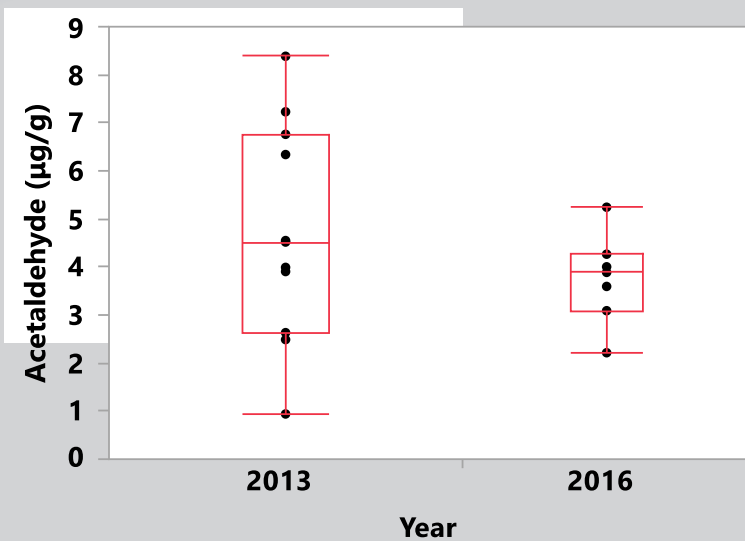


CORESTA Recommended Methods (past 12 months)

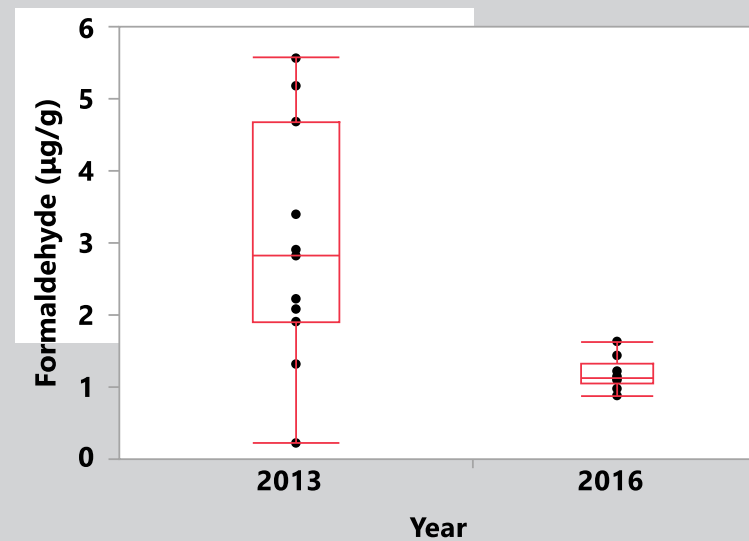
- 1) **CRM No. 56 - Determination of Water in Tobacco and Tobacco Products by Karl Fischer Method**
 - **Update to include cigars and CRPs**
- 2) **CRM No. 57 - Determination of Water in Tobacco and Tobacco Products by Gas Chromatographic Analysis**
 - **Update to include cigars and CRPs**
- 3) **CRM No. 79 - Determination of Ammonia in Tobacco and Tobacco Products by Ion Chromatographic Analysis**
 - **Update to include cigars and CRPs**
- 4) **CRM No. 82 - Determination of Benzo[a]pyrene in Tobacco Products by GC-MS**
 - **Update to include cigars and CRPs**
- 5) **CRM No. 86 - Determination of Select Carbonyls in Tobacco and Tobacco Products by UHPLC-MS/MS**

❖ Comparison of variability between initial and final studies

CRP 2 (Moist Snuff)

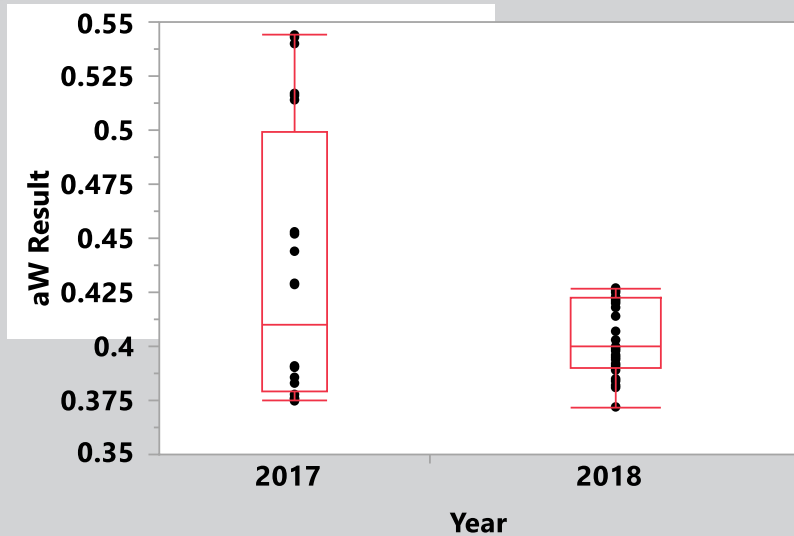


CRP 2 (Moist Snuff)

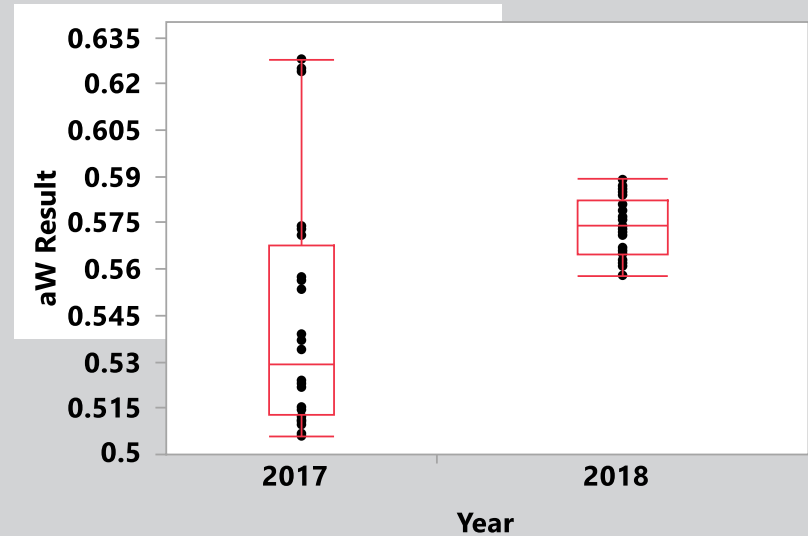


❖ Comparison of variability between initial and final studies

CRP 3.1 (Dry Snuff)



Mentholated Cigarette Filler





Ongoing Work Items

1) Expanded PAHs collaborative study

- Purpose: develop CRM
- Finalize technical report, and CRM

2) Water activity collaborative study

- Purpose: develop CRM
- Finalize technical report, and CRM

3) Metals collaborative study

- Purpose: develop CRM
- Finalize technical report, and CRM

4) Update the CORESTA Technical Guideline for sample handling of STPs

- Finalize guide



New Work Items

❖ Fall 2018

- 1) Modify CRM 62 (GC-FID) and CRM 87 (GC-MS) for very low nicotine containing products

❖ Spring 2019

- 1) Biennial 2016 CRP stability study (TSNA, nicotine, pH, and moisture)
- 2) 10-year stability study for 2009 CRPs (TSNA, nicotine, pH, and moisture)



Very Low Nicotine Collaborative Study Project 193

- ❖ **Purpose:** expand the scopes of CRM 62 (GC-FID) and CRM 87 (GC-MS) to include very low nicotine containing (VLNC) products
- ❖ **Timeline:** Nov 2018 to Feb 2019
- ❖ **Samples:** VLNC cigarette filler, VLNC MST, and traditional tobacco products (CRPs, reference cigarette filler, cigar filler)



Standards Development - ISO

- ❖ **ISO 21045: "Tobacco and tobacco products - Determination of ammonia - Method using ion chromatographic analysis"**
 - **Published August 2018**

- ❖ **ISO 21766: "Tobacco and tobacco products - Determination of tobacco-specific nitrosamines in tobacco products - Method using LC-MS/MS"**
 - **Published August 2018**



Constituents of Regulatory Concern

Unburned Tobacco Filler (cigarettes, cigars, RYO, etc.)

Constituent	CRM	ANVISA (Brazil)	Health Canada	China STMA	US FDA
Nitrate	36	F	F	F	--
Humectants	60, 61	F	F	F	--
Nicotine	62, 87	F	F	F	F
pH	69	F	F	F	--
TSNAs	72	F	F	F	F
Moisture	82	F	F	F	--
Ammonia	79	F	F	F	F
B[a]P	82	F	F	--	--
Metals	TBD	F	F	F	F



Constituents of Regulatory Concern

Smokeless Tobacco

Constituent	CRM	ANVISA (Brazil)	Health Canada	Swedish NFA	US FDA
Nitrate	36	ST	ST	--	--
Humectants	60, 61	ST	ST	ST	--
Nicotine	62, 87	ST	ST	--	ST
pH	69	ST	ST	--	ST
TSNAs	72	ST	ST	ST	ST
Moisture	82	ST	ST	--	--
Ammonia	79	ST	ST	--	ST
B[a]P	82	ST	ST	ST	ST
Carbonyls	86	--	--	--	ST
Metals	TBD	ST	ST	ST	ST



Benefits to the Scientific Community

- ❖ **Production and maintenance of CRPs**
- ❖ **Development of robust methods with defined repeatability and reproducibility**
- ❖ **Interlaboratory studies**
 - **Provide laboratory performance feedback**
 - **Support ISO 17025 accreditation**
- ❖ **Study results and methodology are a source of engagement with authorities and regulators**



Acknowledgements

- ❖ **Participating laboratories and their management's support**
- ❖ **John Bunch**
 - **WG4 Coordinator (reference products)**
- ❖ **Michael Morton**
 - **Statistical Support**