Tobacco and Tobacco Products
Analytes Sub-Group (TTPA)
2017 Annual Report

CORESTA SSPT – Kitzbühel, Austria
October 11, 2017
Coordinator and Scientific Commission Liaison:
- Karl Wagner (Altria Client Services LLC, Virginia, United States)

Secretary:
- Johan Lindholm (Swedish Match, Stockholm, Sweden)

Sub-Group was established in November 2008

Typically two meetings per year:
- ~ 45 attendees
- ~ 30 companies represented

Meetings since 2016 CORESTA Congress, Berlin
- 2017: Charlottesville, Virginia (May) – 20th meeting
- 2017: Kitzbühel, Austria (October) – 21st meeting
Revised Sub-Group Name and Objectives

- Alignment of the Sub-Group’s name and objectives with the work performed
- Smokeless Tobacco and Tobacco Products Analytes Sub-Group (TTPA)

**Objectives:**

1. To propose and evaluate practical and robust Recommended Methods for the determination of analytes related to smokeless tobacco and tobacco products.
2. To periodically organise collaborative and/or proficiency testing.
3. To organise the manufacture and maintain smokeless tobacco reference products.
New Work Item Proposals

- Constituents of regulatory concern
  - Participating laboratories
  - Scientific Commission

Iterative, stepwise process to develop a CRM

1) Identify method(s) for evaluation
2) Initial small joint experiment
3) Method refinements
4) Large collaborative study

Robust CRM with defined repeatability and reproducibility
Smokeless Tobacco Reference Products (CRPs)

- Coordinated production in 2009 and 2016
- Maintained and distributed by North Carolina State University
  - Swedish style snus pouch
  - American-style loose moist snuff
  - American-style loose dry snuff powder
  - American-style (chopped) loose-leaf chewing tobacco
Supply of 2009 CRPs

<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>CRP 1</td>
<td>7000</td>
<td>6135</td>
<td>5443</td>
<td>4895</td>
<td>3975</td>
<td>3242</td>
<td>1992</td>
<td>1195</td>
<td>1160</td>
</tr>
<tr>
<td>CRP 2</td>
<td>7020</td>
<td>6363</td>
<td>6093</td>
<td>5082</td>
<td>4453</td>
<td>3680</td>
<td>1723</td>
<td>620</td>
<td>592</td>
</tr>
<tr>
<td>CRP 3</td>
<td>7040</td>
<td>6653</td>
<td>6207</td>
<td>5520</td>
<td>5113</td>
<td>4691</td>
<td>4191</td>
<td>2169</td>
<td>2116</td>
</tr>
<tr>
<td>CRP 4</td>
<td>7000</td>
<td>6806</td>
<td>6567</td>
<td>6374</td>
<td>6236</td>
<td>6040</td>
<td>5686</td>
<td>2309</td>
<td>2278</td>
</tr>
</tbody>
</table>
## Supply of 2016 CRPs

<table>
<thead>
<tr>
<th>CRP</th>
<th>Apr 2016</th>
<th>Sept 2016</th>
<th>Dec 2016</th>
<th>Sept 2017</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.1</td>
<td>10008</td>
<td>9384</td>
<td>9312</td>
<td>8908</td>
</tr>
<tr>
<td>2.1</td>
<td>10000</td>
<td>9132</td>
<td>9061</td>
<td>8380</td>
</tr>
<tr>
<td>3.1</td>
<td>7000</td>
<td>6426</td>
<td>6340</td>
<td>6210</td>
</tr>
<tr>
<td>4.1</td>
<td>7200</td>
<td>6855</td>
<td>6769</td>
<td>6598</td>
</tr>
</tbody>
</table>
Collaborative studies:
- Assess stability of the CORESTA Reference Products (CRPs)
- Support laboratory accreditation

Analytes
- Nicotine
- pH
- Tobacco-specific nitrosamines
- Moisture (oven volatiles)
- Ammonia
- Benzo[a]pyrene
- New CRMs

Even Years
Odd Years
Ground Tobacco Reference Products

- Prepared and distributed by the University of Kentucky, Center for Tobacco Reference Products

- Dark Fire Cured
- Flue Cured
- Dark Air Cured
- Oriental
- Burley
Technical Reports Completed (past 12 months)

1) CORESTA Reference Products (Smokeless Tobacco) - 2016 Analysis (Jan 2017)
2) 2016 Collaborative Study on Minor Alkaloids in Tobacco Products (Feb 2017)
3) 2016 Collaborative Study on Nicotine in Tobacco Products (Feb 2017)
4) 2017 Collaborative Study on TSNA, pH, and Moisture (OV) (July 2017)
5) 2017 Collaborative Study on Benzo[a]pyrene in Tobacco Products (July 2017)
6) Determination of Nitrite and Nitrate in Smokeless Tobacco Products by Ion Chromatography and Continuous Flow Analysis (July 2017)
7) 2017 Metals Proficiency Study (July 2017)
   ➢ Update to include ground tobacco, cigarette and cigar filler

2) CRM Nº 72: Determination of Tobacco Specific Nitrosamines in Tobacco and Tobacco Products by LC-MS/MS (4th ed., 2017)
   ➢ Update to include ground tobacco, cigarette and cigar filler

3) CRM Nº 76: Determination of Moisture Content (Oven Volatiles) of Tobacco and Tobacco Products (2nd ed., 2017)
   ➢ Update to include ground tobacco, cigarette and cigar filler

4) CRM Nº 82: Determination of Benzo[a]pyrene in Tobacco Products by GC-MS (2nd ed., 2017)
   ➢ Method update
1) Expanded PAHs
   - Purpose: Create CRM for analytes of regulatory concern
   - Receive data, draft technical report, and CRM

2) 2017 Ammonia, B[a]P Study
   - Purpose: Expand the scope of CRMs to include cigars
   - Receive remaining data sets, draft technical report and update CRMs

3) Carbonyls - Formaldehyde, acetaldehyde, crotonaldehyde
   - Purpose: Create a CRM for analytes of regulatory concern
   - Finalize technical report and CRM
New Work Items

❖ Spring 2018
1) Update CORESTA Guide No. 11 - *Technical Guideline for Sample Handling of Smokeless Tobacco and Smokeless Tobacco Products*
2) Water (GC/Karl Fischer) and moisture (OV) collaborative study
3) Development of CRM for select metals

❖ Fall 2018
1) TSNA, nicotine, pH, and moisture collaborative study
2) Continue development of CRM for metals
ISO/DIS 21045: "Tobacco and tobacco products - Determination of ammonia - Method using ion chromatographic analysis"
- The Draft International Standard (DIS) was approved on 2017-06-22

ISO/DIS 21766: “Tobacco and tobacco products - Determination of tobacco-specific nitrosamines in tobacco products - Method using LC-MS/MS”
- Approved for circulation as a DIS in April 2017
- DIS voting starts on 2017-10-11 and concludes 2018-01-03

Submit the B[a]P and Moisture CRMs to ISO as NWIPs
## Progress Against Objectives

### Smokeless Constituents of Regulatory Concern

<table>
<thead>
<tr>
<th>Constituent</th>
<th>Status</th>
<th>HPHC Abbreviated List</th>
<th>WHO STP Priority Toxicants</th>
<th>Swedish National Food Agency</th>
</tr>
</thead>
<tbody>
<tr>
<td>Nicotine (total and free)</td>
<td>CRM N°62</td>
<td>√</td>
<td></td>
<td></td>
</tr>
<tr>
<td>pH</td>
<td>CRM N°69</td>
<td>√</td>
<td></td>
<td></td>
</tr>
<tr>
<td>NNK, NNN</td>
<td>CRM N°72</td>
<td>√</td>
<td>√</td>
<td>√</td>
</tr>
<tr>
<td>Benzo[a]pyrene</td>
<td>CRM N°82</td>
<td>√</td>
<td>√</td>
<td>√</td>
</tr>
<tr>
<td>Acetaldehyde, Croton., Formaldehyde</td>
<td>Draft CRM</td>
<td>√</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Arsenic, Cadmium</td>
<td>PT</td>
<td>√</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Lead</td>
<td>PT</td>
<td></td>
<td></td>
<td>√</td>
</tr>
<tr>
<td>Aflatoxin B1</td>
<td></td>
<td></td>
<td></td>
<td>√</td>
</tr>
</tbody>
</table>
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
Participating laboratories and their management’s support

John Bunch
- WG4 Coordinator (reference products)

Michael Morton
- Statistical Support