

CHARACTERIZATION AND CERTIFICATION OF SMOKELESS TOBACCO REFERENCE PRODUCTS

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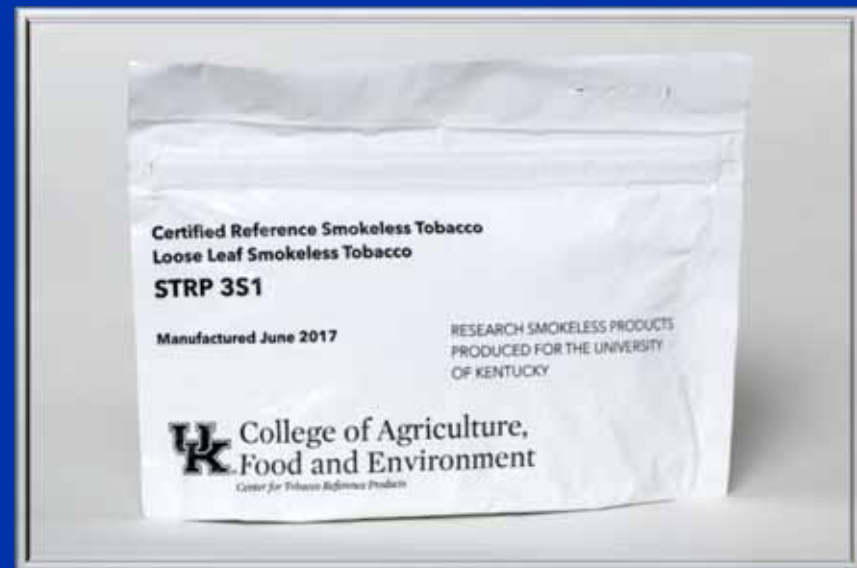


Smokeless Tobacco Reference Products

- Loose leaf chewing tobacco
- Moist snuff
- Snus
- Swedish Style Snus

Loose Leaf Tobacco

- Production
 - Completed 06/2017
 - 16,560 units
 - Unit is 85g foil pouch
 - Case = 12 cartons of 12 units
- Sampling for CoA
 - 35 samples
 - 4 cartons per sample



Moist Snuff

- Production
 - Completed 06/2018
 - 24,930 units
 - Unit is plastic can of 34 g
 - Case = 18 sleeves of 5 units
- Sampling for CoA
 - 11 samples
 - 1 case per sample



Snus

- Production
 - Completed 06/2017
 - 23,040 units
 - Unit is plastic container with 12 pouches
 - Case = 18 sleeves of 5 units
- Sampling for CoA
 - 18 samples
 - 2 cases per sample



Swedish Style Snus

- Production
 - Completed 06/2017
 - 23,040 units
 - Unit is plastic container with 24 1-g pouches
 - Case = 240 units
- Sampling for CoA
 - 18 samples
 - 1 case per sample



Analytical Strategy

- Same method as used to certify the 1R6F reference cigarette.
 - Follows ISO Guides 34:2009, 25:2009, & 17034:2016 and JCGM 100:2008.
 - Uses a hybrid of ISO-recommended approaches.
- Model for characterization
 - $$U_{CRM} = k \sqrt{u_{char}^2 + u_{bb}^2 + u_{lts}^2 + u_{sts}^2}$$
 - Since the last 2 uncertainties were initially considered to be null, the working model becomes $U_{CRM} = k \sqrt{u_{char}^2 + u_{bb}^2}$ where u_{bb}^2 assesses the between lab variability and u_{char}^2 is the within laboratory variability.
 - The coverage factor, k , is based on the t-distribution with calculated effective degrees of freedom.

Certificate of Analysis

- 14 Analytes
 - Total nicotine
 - Free nicotine
 - NNK
 - NNN
 - NAT
 - NAB
 - Acetaldehyde
 - Crotonaldehyde
 - Formaldehyde
 - Benzo[α]pyrene
 - Cadmium
 - Arsenic
 - Moisture
 - pH
- 4 Labs
- Convenience sampling plan
- Nesting for characterization: Replicate \rightarrow Sample \rightarrow Lab

Results

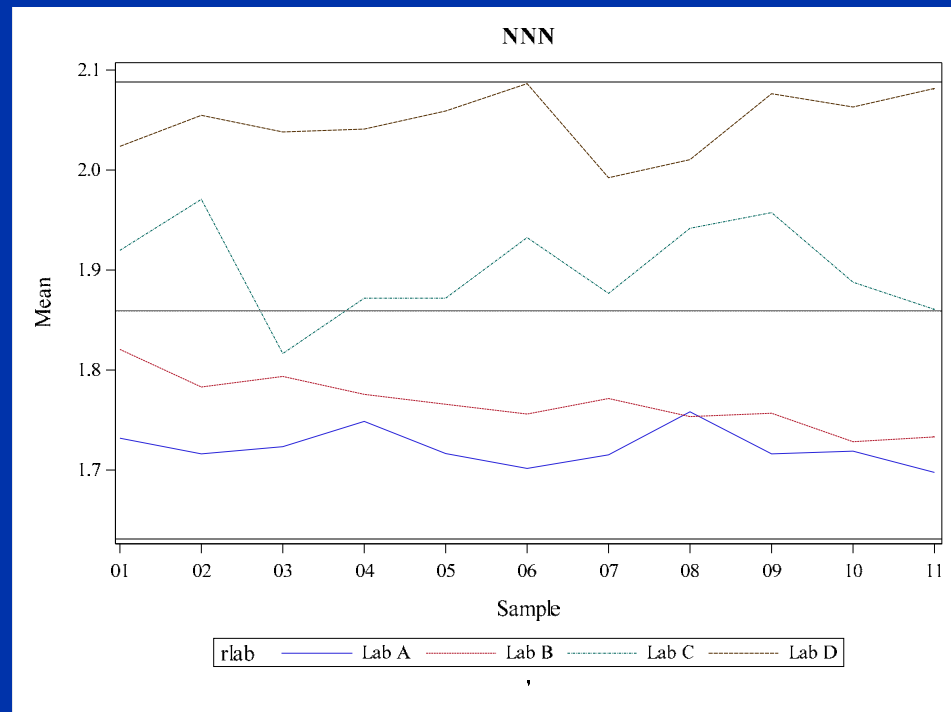
- Initial analyses ran in March 2018 for loose leaf chewing tobacco, snus & Swedish style snus.
- Significant variability within and between labs
- Quality assurance measures suggested
 - Re-analyzation of samples
 - Verification of calculations and data submissions
 - Review of internal quality control data
- All Crotonaldehyde were below the detection limit

Results

- Almost two-thirds of the uncertainties were as expected after reanalysis.
- Lingering concerns
 - Drifts in data (Chew Tob – Acetaldehyde)
 - Analysis issues (Snus – Arsenic)
 - Splits in data (All – Formaldehyde)

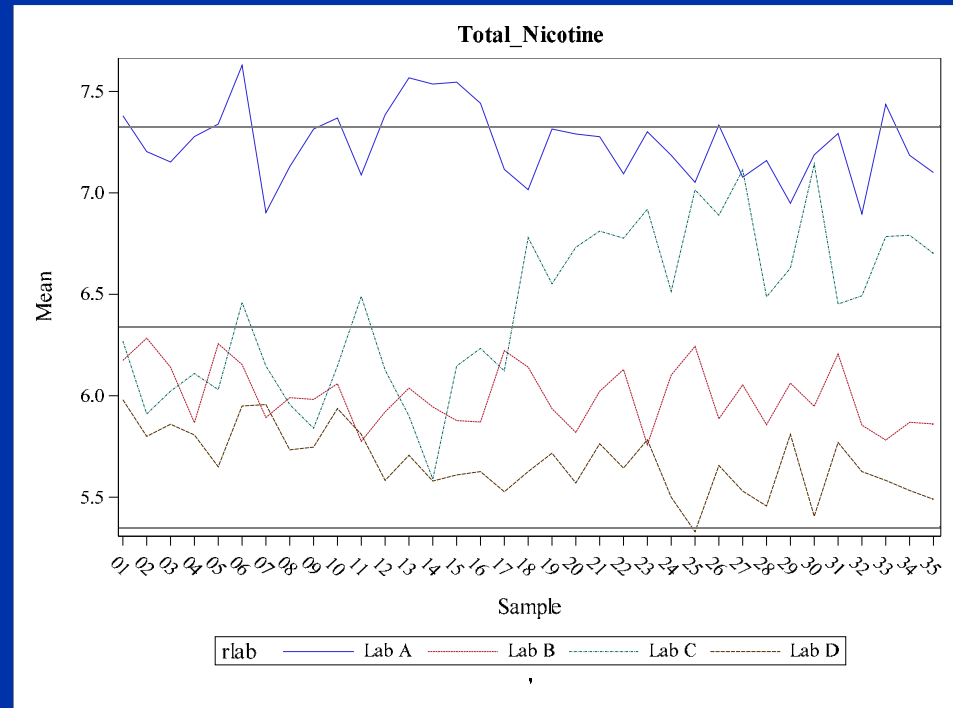
Moist Snuff - NNN

- Certified Value = 1.859 $\mu\text{g/g}$
- Certified Uncertainty = 0.229 $\mu\text{g/g}$
- Coverage factor, $k = 3.08$
- 100% coverage
- Observations:
 - Very little within lab variability



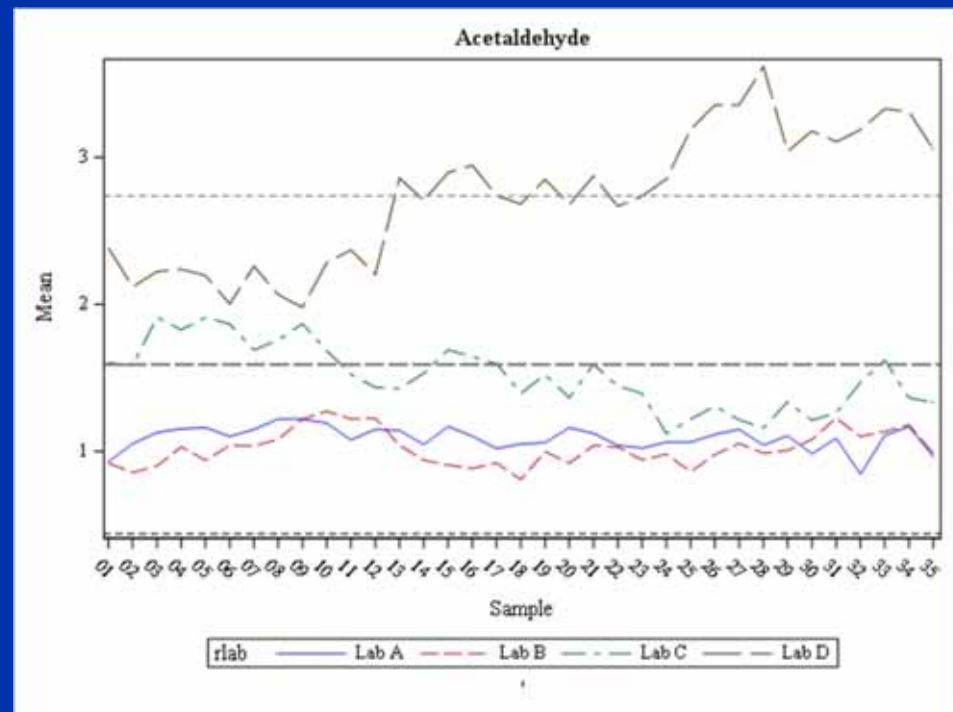
Chewing Tobacco – Total Nicotine

- Certified Value = 6.338 $\mu\text{g/g}$
- Certified Uncertainty = 0.987 $\mu\text{g/g}$
- Coverage factor, $k = 2.42$
- 91% coverage
- Observations:
 - Some variability, but no concerning patterns.



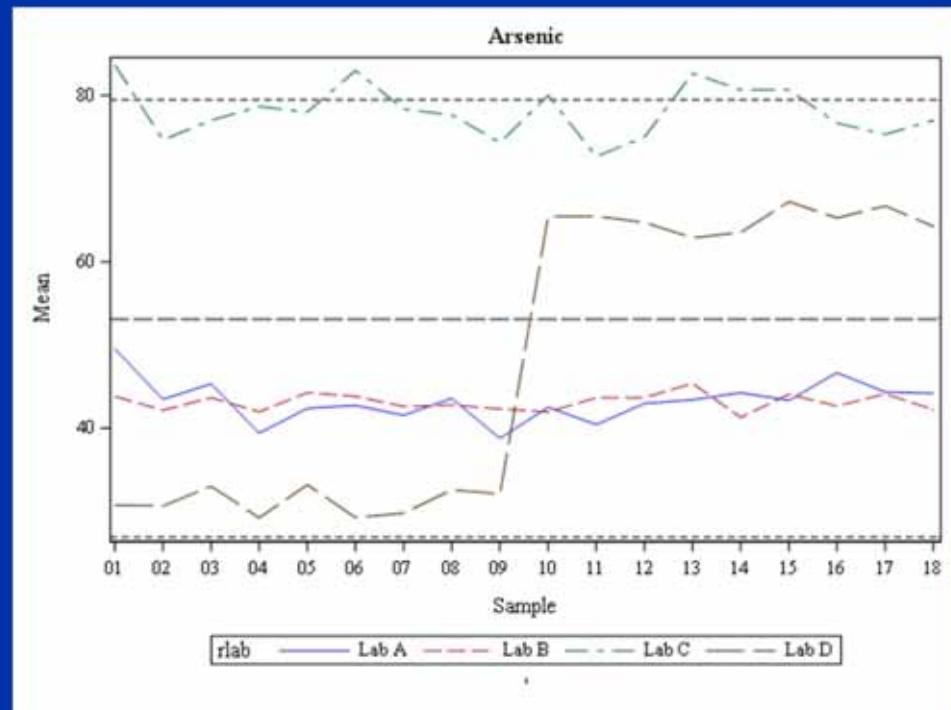
Chewing Tobacco - Acetaldehyde

- Certified Value = 1.589 $\mu\text{g/g}$
- Certified Uncertainty = 1.150 $\mu\text{g/g}$
- Coverage factor, $k = 2.45$
- 87% coverage
- Observations:
 - Possible upward drift in Lab D
 - Lab C has slight downward drift.



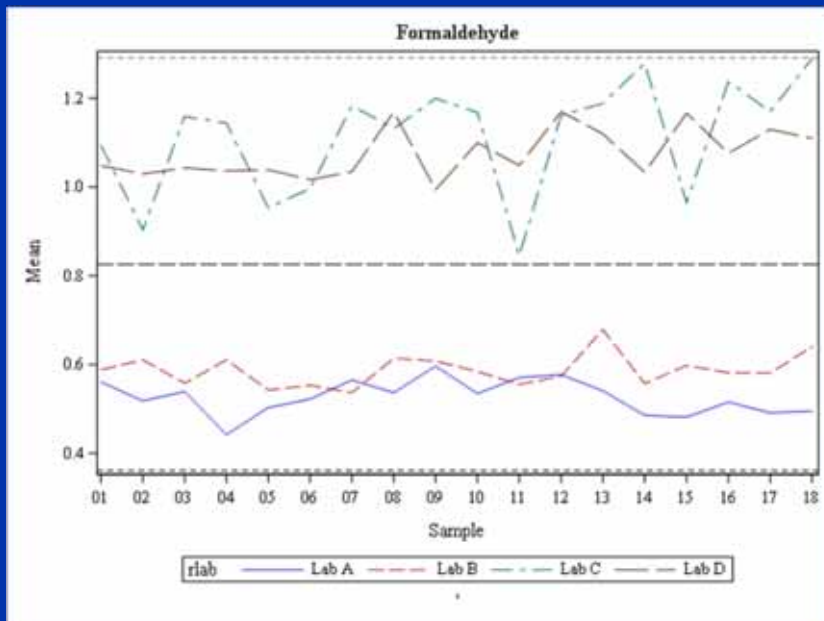
Snus - Arsenic

- Certified Value = 53.17 ng/g
- Certified Uncertainty = 26.27 ng/g
- Coverage factor, $k = 2.16$
- 92% coverage
- Observations:
 - Lab D split between samples 9 and 10.
 - Smaller but similar pattern for Arsenic in Sw Snus.

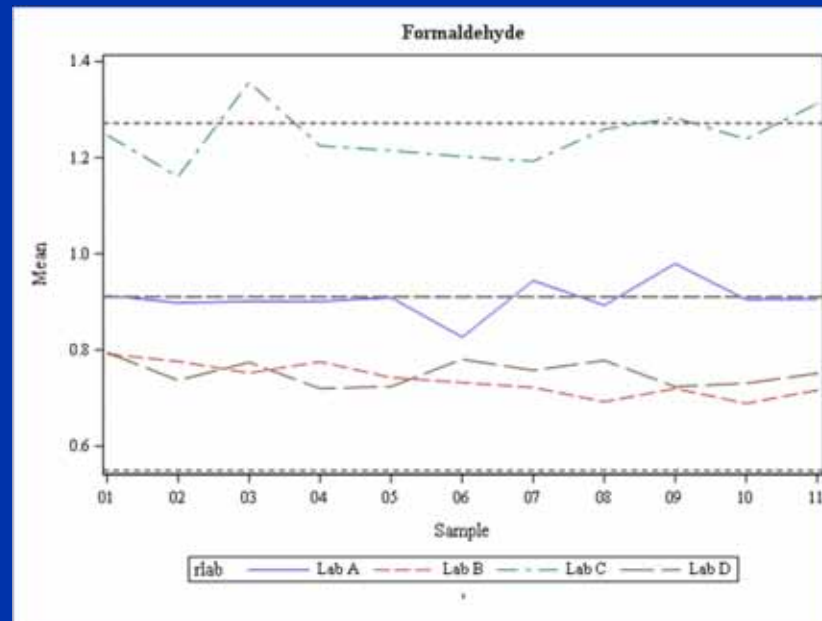


Formaldehyde

Swedish style Snus



Moist Snuff



Conclusions

- Most of the analytes had expected levels of uncertainties.
- Data must be reviewed carefully.
- Standardization of analysis methods should be considered.

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