Arista Laboratories
*ISO 17025 Accredited*
*A2LA Certificate No. 1873.01*

PROJECT CODE: 11066

# TEST REPORT

# THE DETERMINATION OF SELECTED ANALYTES IN MAINSTREAM SMOKE UNDER ISO SMOKING CONDITIONS

**Prepared for:**

**Goodrich Tobacco Company**
**8201 Main Street**
**Suite 6**
**Williamsville, NY 14221**
**United States**

**May 19, 2011**

Arista Laboratories
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## I. STUDY OVERVIEW

The objective of this study was to determine the deliveries of selected smoke constituents in mainstream cigarette smoke under ISO smoking conditions.

Test cigarettes for this study consisted of one type, which is listed in Table I.

Selected analyte groups that were determined are listed in Table II.

The laboratory control cigarette for this project was the Kentucky Reference 3R4F cigarette.

A minimum of three observations per brand were determined for mainstream smoke and one observation for physicals, as specified by the client.

The results were electronically submitted to the client on May 19, 2011.

Analytical methods and results are included in the attached appendices.

## II. TESTING FACILITY

Activities were conducted at Arista Laboratories, 1941 Reymet Road Richmond, VA. 23237.

## III. TEST CIGARETTES

### A. Receipt, storage and conditioning of test cigarettes

- Three hundred and eighty cigarettes of each type were received on March 11, 2011.
- The brand code designations are given in Table I.
- Cigarettes were stored at ambient conditions until required for testing.
- Before smoking, cigarettes were conditioned according to Arista SOP FAC-035 Handling of Test Items.

**Table I. Brand Coding and Sampling**

| Client Sample I.D. | Arista Brand Code | Number of Cigarettes Received |
|---|---|---|
| Quest 3 Menthol Lights Box | E7791 | 380 |

### B. Constituent List

The analyses requested are listed in Table II. All applicable analytical methods have been fully validated. For lists of specific compounds in each analyte group, refer to the appendices.

**Table II. Constituent List**

| Analyte Group | Arista Analytical Methods | Validation Report |
|---|---|---|
| Nicotine, Menthol and Water in MS by GC | AM-001R v.6.0 | V-005 |
| Semivolatiles in MS & SS by GC/MS | AM-006 v.9.0 | V-004 |
| Carbon Monoxide in MS & SS by Non-Dispersive Infrared (NDIR) | AM-007R v.4.0 | V-005-2 |
| Physical Properties | AM-009 v.7.2 | N/A |
| Ammonia in MS & SS by HPLC | AM-011 v.7.2 | V-009 |
| Volatiles in MS & SS by GC/MS | AM-015 v.9.2 | V-007 |
| Tobacco Specific Nitrosamine (TSNA) in MS by LC/MS/MS | AM-020 v.8.1 | V-013 |
| Metals in MS by ICP/MS | AM-021 v.7.1 | V-014 |
| Phenols in MS by HPLC | AM-027 v.9.1 | V-017 |
| Nitric Oxide & Other Nitrogen Oxides (NO & NOx) in MS by Chemiluminescence | AM-029 v.6.1 | V-018 |
| Polycyclic Aromatic Amines (PAA) in MS by GC/MS | AM-030 v.11.0 | V-016 |
| Mercury in MS by Cold Vapor Atomic Absorption | AM-036 v.6.0 | V-021 |
| Polycyclic Aromatic Hydrocarbons (PAH's) in MS & SS by GC/MS | AM-044 v.4.2 | V-031 |
| Carbonyls in MS by UPLC | AM-076 v.3.1 | V-072 |
| Eugenol in MS & SS by HPLC | AM-087 v.2.2 | V-042 |
| pH in MS by pH Meter-Health Canada Method | AM-093 v.2.0 | N/A |
| Hydrogen Cyanide in MS & SS by CFA | AM-111 v.4.1 | V-063 |

### C. Sample Generation and Records

Smoking was performed under ISO smoking conditions. The project was completed with the electronic submission of the final data set.

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## IV. QUALITY STATEMENT\SIGNATURES

The analytical results in the attached appendices were reviewed for consistency and evaluated for outliers where deemed necessary. All outliers were noted and repeats conducted if cause was determined. All tests were conducted in accordance with Arista Laboratories quality and technical procedures.

QA Review: [redacted] (b) (6) [redacted] Date _____

(b) (6) QA Manager

Management Approval: (b) (6) [redacted] Date _____

(b) (6), PhD, Laboratory Director

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# APPENDIX A

## ANALYSIS OF MAINSTREAM CIGARETTE SMOKE FOR TAR, NICOTINE AND CARBON MONOXIDE

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# DETERMINATION OF TAR, NICOTINE AND CARBON MONOXIDE IN MAINSTREAM SMOKE

## I. PRINCIPLE OF METHOD

The methods used for this study are detailed in Arista Analytical Method AM-001, Determination of Nicotine and Water in Smoke Condensates – Gas Chromatographic Method, and Arista Analytical Method T-007, Determination of Carbon Monoxide in Vapor Phase Cigarette Smoke.

This method applies to the measurements of nicotine and water in total particulate matter (TPM) from cigarette smoke collected on a 44mm Cambridge filter pad. The smoke condensate collected on the pad is dissolved in iso-propanol. Subsequently, an aliquot of the sample solution is analyzed by gas chromatography (GC), and the nicotine and water content of the smoke condensate is determined. Using the nicotine and water values, and the measured wet total particulate matter (WTPM), the tar content is then calculated. The resulting data is reported in milligrams per cigarette (mg/cig) for each smoke sample.

For carbon monoxide determination, the smoke is collected in gas sampling bags attached to the smoking machine. A non-dispersive infrared absorption method (NDIR) is used to measure the CO concentration in the vapor phase in percent by volume (%vol). Using the number of cigarettes, puff counts, puff volume and ambient conditions, the %CO is converted to milligrams per cigarette (mg/cig).

**Mainstream TNC Test Report for:**
**Goodrich Tobacco Company**
**8201 Main Street**
**Suite 6**
**Williamsville, NY 14221**
[redacted]

**Project Code:** 11066
**Project Start Date:** 11-Mar-11
**Arista SOP #:** AM-001
**Smoking Protocol:** ISO
**Authorized By:** [redacted]
**Title:** QA

*For questions or concerns, please see the customer feedback form at http://www.aristalabs.com*

| Client Code | Run # | Port | Arista Code | Puffs (/cigt) | MS TPM (mg/cigt) | CO (mg/cigt) | Water (mg/cigt) | Nicotine (mg/cigt) | Tar (mg/cigt) |
|---|---|---|---|---|---|---|---|---|---|
| Quest 3 Menthol Lights Box | 500546 | 1 | E7791 | 5.8 | 13.5 | 13.5 | 2.48 | 0.043 | 11.0 |
| Quest 3 Menthol Lights Box | 500546 | 2 | E7791 | 5.5 | 13.4 | 15.5 | 2.16 | 0.045 | 11.2 |
| Quest 3 Menthol Lights Box | 500546 | 3 | E7791 | 5.4 | 12.6 | 16.4 | 1.70 | 0.042 | 10.8 |
| | | | **Average** | **5.6** | **13.1** | **15.1** | **2.11** | **0.043** | **11.0** |
| | | | **sd** | **0.2** | **0.512** | **1.48** | **0.388** | **0.001** | **0.183** |
| | | | **%RSD** | **3.5** | **3.89** | **9.78** | **18.4** | **3.23** | **1.66** |
| Lab Control | 500546 | 4 | 3R4F | 8.3 | 9.76 | 11.1 | 0.712 | 0.730 | 8.32 |
| Lab Control | 500546 | 9 | 3R4F | 8.3 | 10.1 | 10.6 | 0.869 | 0.753 | 8.50 |
| | | | **Average** | **8.3** | **9.94** | **10.8** | **0.790** | **0.742** | **8.41** |
| | | | **sd** | **0.0** | **0.255** | **0.375** | **0.111** | **0.017** | **0.127** |
| | | | **%RSD** | **0.0** | **2.56** | **3.46** | **14.1** | **2.24** | **1.51** |

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# APPENDIX B

## ANALYSIS OF MAINSTREAM CIGARETTE SMOKE FOR PYRIDINE, 3-VINYLPYRIDINE AND QUINOLINE

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# DETERMINATION OF PYRIDINE, 3-VINYLPYRIDINE AND QUINOLINE (SEMIVOLATILES) IN MAINSTREAM SMOKE

## I. PRINCIPLE OF METHOD

The method used for this study is detailed in Arista Analytical Method AM-006 Determination of Pyridine, 3-Vinylpyridine and Quinoline in Mainstream Smoke.

Mainstream (MS) smoke is passed through a 44-mm Cambridge filter and collected in one impinger containing 20 mL MeOH/TEA that is immersed in a dry ice / isopropyl alcohol bath. After smoking, the MS filter is weighed and transferred to an Erlenmeyer flask. The contents of the impinger are added, the internal standard (ISTD) is added, and the sample is shaken for 30 min at 200 rpm. The extract is filtered into an ALS vial and subsequently analyzed by GC/MSD.

Individual analyte concentrations are determined by the internal standard method using gas-chromatography with mass-selective detection. The concentrations of the analytes determined by the GC/MSD are reported in units of mass-to-volume (i.e., µg/mL). The measured concentration, the number of cigarettes smoked, and the sample solution volume(s) are used to calculate the total analyte mass on a per cigarette basis.

**Mainstream Semivolatiles Test Report for:**
**Goodrich Tobacco Company**
**8201 Main Street**
**Suite 6**
**Williamsville, NY 14221**
(b) (4)

**Project Code:** 11066
**Project Start Date:** 11-Mar-11
**Arista SOP #:** AM-006
**Smoking Protocol:** ISO
**Authorized By:** (b) (6)
**Title:** QA

*For questions or concerns, please see the customer feedback form at http://www.aristalabs.com*

| Client Code | Run # | Port | Arista Code | Puffs (/cigt) | MS TPM (mg/cigt) | Pyridine (µg/cigt) | Quinoline (µg/cigt) |
|---|---|---|---|---|---|---|---|
| Quest 3 Menthol Lights Box | 600578 | 1 | E7791 | 5.4 | 15.4 | 11.8 | 0.378 |
| Quest 3 Menthol Lights Box | 600578 | 2 | E7791 | 5.5 | 17.1 | 12.4 | 0.394 |
| Quest 3 Menthol Lights Box | 600578 | 3 | E7791 | 5.5 | 15.6 | 10.9 | 0.406 |
| | | | **Average** | **5.5** | **16.0** | **11.7** | **0.393** |
| | | | **sd** | **0.1** | **0.9** | **0.8** | **0.014** |
| | | | **% RSD** | **0.9** | **5.5** | **6.5** | **3.6** |
| Lab Control | 600578 | 4 | 3R4F | 8.3 | 11.6 | 4.70 | 0.217 |


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# APPENDIX C

## MEASUREMENT OF PHYSICAL PROPERTIES

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## CIGARETTE WEIGHT

### PRINCIPLE OF METHOD

The method used for this study is detailed in Arista Analytical Method AM-009 – Physical Properties.

This measurement can either be performed automatically on the QTM stack or manually. The QTM automatically feeds cigarettes from a hopper and analyzes for weight, circumference, pressure drop and ventilation (VO & VC). In the manual method, conditioned cigarettes are weighed individually on a calibrated analytical balance and recorded in grams to the nearest 0.0001g. The average, standard deviation and relative standard deviation are reported for multiple observations.

## CIRCUMFERENCE

### PRINCIPLE OF METHOD

The method used for this study is detailed in Arista Analytical Method AM-009 – Physical Properties.
Ten cigarettes are placed into the hopper of a QTM Stack with Module QTM 3. The Module should be calibrated before use. Circumference is reported in millimeters.

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## TIP VENTILATION

### PRINCIPLE OF METHOD

The method used for this study is detailed in Arista Analytical Method AM-009 – Physical Properties.
ISO conditioned test cigarettes are placed into the hopper of a Cerulean QTM Stack with module QTM 5U installed, with filters pointing in the direction of the hopper diagram. The cigarette measurement head contains three distensible latex-grip seals that isolate the tipping paper region and the cigarette paper region of the cigarette. Tip ventilation is defined as the air entering the cigarette through the perforated tipping as a proportion of the total flow through the cigarette. Tip ventilation is expressed as percentage i.e.:

$$\%\,\mathrm{TV} = \frac{\mathrm{QT}}{\mathrm{Q}} \times 100$$

Where: %TV = percent tip ventilation
QT = airflow through ventilation holes
Q = total airflow through the cigarette

## DRAW RESISTANCE

### PRINCIPLE OF METHOD

Ten previously conditioned cigarettes are placed into the hopper of a QTM Stack with module QTM 5U with filters pointing to the direction of the hopper diagram.

The QTM 5U automatically calibrates pressure drop at 17.5 ml/s. A check standard is used to verify, 1.99 mmWg for cigarettes and 803 mmWg for filter rods. Total RTD is defined as the pressure differential developed by a cigarette when air is pulled through it at the rate of 1050 mL/min.

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# PLUG RESISTANCE

## PRINCIPLE OF METHOD

The filter is cut and removed from ten conditioned cigarettes with a razor or knife and dropped into the slot at the top of the QTM 6 unit. Plug RTD is defined as the pressure differential developed by the filter portion of a cigarette when air is pulled through it at the rate of 1050 mL/min. Values are recorded in mm H2O.

**Physical Properties Test Report for:**
**Goodrich Tobacco Company**
**8201 Main Street**
**Suite 6**
**Williamsville, NY 14221**
[redacted]

**Project Code:** 11066
**Project Start Date:** 11-Mar-11
**Analytical Method #:** AM-009
**Authorized By:** [redacted]
**Title:** QA

*For questions or concerns, please see the customer feedback form at http://www.aristalabs.com*

| Client Code | Arista Code | QTM Cigarette Weight (g) | Circumference (mm) | Pressure Drop "open" | Pressure Drop "closed" | Tip Ventilation | PPM 100 |
|---|---|---|---|---|---|---|---|
| Quest 3 Menthol Lights Box | E7791 | 0.887 | 25.0 | 118 | 118 | 0.30 | 66.2 |
| Quest 3 Menthol Lights Box | E7791 | 0.853 | 24.9 | 121 | 121 | 0.30 | 68.3 |
| Quest 3 Menthol Lights Box | E7791 | 0.861 | 25.0 | 114 | 113 | 0.30 | 61.7 |
| Quest 3 Menthol Lights Box | E7791 | 0.878 | 24.9 | 122 | 122 | 0.50 | 50.6 |
| Quest 3 Menthol Lights Box | E7791 | 0.827 | 25.0 | 118 | 117 | 0.30 | 48.6 |
| Quest 3 Menthol Lights Box | E7791 | 0.820 | 24.9 | 114 | 113 | 0.30 | n/a |
| Quest 3 Menthol Lights Box | E7791 | 0.862 | 25.0 | 126 | 125 | 0.20 | n/a |
| Quest 3 Menthol Lights Box | E7791 | 0.866 | 25.0 | 112 | 113 | 2.00 | n/a |
| Quest 3 Menthol Lights Box | E7791 | 0.872 | 25.0 | 124 | 123 | 0.40 | n/a |
| Quest 3 Menthol Lights Box | E7791 | 0.862 | 24.9 | 124 | 124 | 0.90 | n/a |
| Quest 3 Menthol Lights Box | E7791 | 0.851 | 24.9 | 119 | 118 | 0.70 | n/a |
| Quest 3 Menthol Lights Box | E7791 | 0.821 | 24.9 | 108 | 108 | 0.20 | n/a |
| Quest 3 Menthol Lights Box | E7791 | 0.837 | 25.0 | 110 | 109 | 0.20 | n/a |
| Quest 3 Menthol Lights Box | E7791 | 0.831 | 24.9 | 113 | 113 | 0.40 | n/a |
| Quest 3 Menthol Lights Box | E7791 | 0.842 | 24.9 | 109 | 109 | 0.20 | n/a |
| Quest 3 Menthol Lights Box | E7791 | 0.836 | 25.0 | 112 | 112 | 0.40 | n/a |
| Quest 3 Menthol Lights Box | E7791 | 0.828 | 25.0 | 112 | 113 | 3.70 | n/a |
| Quest 3 Menthol Lights Box | E7791 | 0.838 | 25.0 | 114 | 113 | 0.20 | n/a |
| Quest 3 Menthol Lights Box | E7791 | 0.841 | 24.9 | 107 | 106 | 0.30 | n/a |
| Quest 3 Menthol Lights Box | E7791 | 0.868 | 25.0 | 123 | 122 | 0.30 | n/a |
| Totals | **Average** | **0.849** | **25.0** | **116** | **115.6** | **0.605** | **59.1** |
| | **SD** | **0.020** | **0.036** | **5.87** | **5.75** | **0.834** | **9.00** |
| | **%RSD** | **2.3** | **0.1** | **5.1** | **5.0** | **138** | **15.2** |
| | **Count** | **20** | **20** | **20** | **20** | **20** | **5** |

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# APPENDIX D

## ANALYSIS OF MAINSTREAM CIGARETTE SMOKE FOR AMMONIA

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# DETERMINATION OF AMMONIA IN MAINSTREAM SMOKE

## I. PRINCIPLE OF METHOD

The method used for this study is detailed in Arista Analytical method AM-011 Determination of Ammonia in Mainstream Smoke.

For mainstream smoke collection, cigarette smoke is passed through a 44-mm Cambridge filter and collected in 2 impingers each containing 30 ml of 10 mmol Methanesulfonic acid (MSA). Immediately after smoking, the filter is transferred to a 125 ml Polymethylpentene Erlenmeyer flask along with the contents of both impingers. The flask is placed on the shaker for 30 min at 200rpm and the extract is subsequently analyzed by High Performance Liquid Chromatography, (HPLC), with conductivity detection.

The concentration of ammonia determined by ion chromatography is reported in units of mass-to-volume (i.e., µg/mL). The measured ammonia concentration, the number of cigarettes smoked, and the sample solution volume(s) are also used to calculate the total amount of ammonia on a per cigarette basis.

**Mainstream Ammonia Test Report for:**
**Goodrich Tobacco Company**
**8201 Main Street**
**Suite 6**
**Williamsville, NY 14221**
(b) (4)

**Project Code:** 11066
**Project Start Date:** 11-Mar-11
**Arista SOP #:** AM-011
**Smoking Protocol:** ISO
**Authorized By:** (b) (6)
**Title:** QA

*For questions or concerns, please see the customer feedback form at http://www.aristalabs.com*

| Client Code | Run # | Port | Arista Code | Puffs (/cigt) | MS TPM (mg/cigt) | Ammonia ($\mu g\ NH_4^+$/cigt) |
|---|---|---|---|---|---|---|
| Quest 3 Menthol Lights Box | 600556 | 1 | E7791 | 5.8 | 13.3 | 37.8 |
| Quest 3 Menthol Lights Box | 600556 | 2 | E7791 | 5.4 | 12.4 | 32.6 |
| Quest 3 Menthol Lights Box | 600556 | 3 | E7791 | 5.3 | 12.5 | 32.9 |
| | | | **Average** | **5.5** | **12.7** | **34.4** |
| | | | **SD** | **0.3** | **0.49** | **2.89** |
| | | | **%RSD** | **4.7** | **3.8** | **8.4** |
| Lab Control | 600556 | 4 | 3R4F | 8.3 | 10.6 | 7.79 |

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# APPENDIX E

## ANALYSIS OF MAINSTREAM CIGARETTE SMOKE FOR VOLATILES

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# DETERMINATION OF VOLATILES IN MAINSTREAM SMOKE

## I. PRINCIPLE OF METHOD

The method used for this study is detailed in Arista Analytical Method AM-015 Determination of Volatiles in Mainstream Smoke. The term Volatiles refers to 1,3-butadiene, isoprene, acrylonitrile, benzene, toluene, and styrene.

Mainstream smoke from test cigarettes is passed through a 44-mm Cambridge filter and collected in one fritted impinger containing 20mL methanol that is immersed in a dry-ice/isopropanol bath. After smoking, the filter is weighed and transferred to the impinger. Internal standard is added, the impinger is vortexed briefly and the extract subsequently analyzed using gas chromatography with a mass selective detector (GC/MSD).

Individual volatile concentrations are determined by the internal standard method. The concentrations of the volatiles determined by the GC/MSD are reported in units of mass-to-volume (i.e., µg/mL). The measured concentration, the number of cigarettes smoked, and the sample solution volume(s) are used to calculate the total analyte mass on a per cigarette basis.

**Mainstream Volatiles Test Report for:**
**Goodrich Tobacco Company**
**8201 Main Street**
**Suite 6**
**Williamsville, NY 14221**
[redacted]

**Project Code:** 11066
**Project Start Date:** 11-Mar-11
**Arista SOP #:** AM-015
**Smoking Protocol:** ISO
**Authorized By:** [redacted]
**Title:** QA

*For questions or concerns, please see the customer feedback form at http://www.aristalabs.com*

| Client Code | Arista Code | Run # | Port | Puffs (/cigt) | MS TPM (mg/cigt) | 1,3-butadiene (µg/cigt) | Isoprene (µg/cigt) | Acrylonitrile (µg/cigt) | Benzene (µg/cigt) | Toluene (µg/cigt) | Styrene (µg/cigt) |
|---|---|---|---|---|---|---|---|---|---|---|---|
| Quest 3 Menthol Lights Box | E7791 | 600569 | 1 | 6.00 | 17.8 | 50.8 | 413 | 22.6 | 55.8 | 102 | 12.9 |
| Quest 3 Menthol Lights Box | E7791 | 600569 | 2 | 5.88 | 15.9 | 50.0 | 419 | 21.5 | 54.3 | 98.6 | 12.8 |
| Quest 3 Menthol Lights Box | E7791 | 600569 | 3 | 6.00 | 15.3 | 46.0 | 371 | 20.1 | 51.8 | 91.8 | 11.4 |
| | | | **Average** | **5.96** | **16.3** | **48.9** | **401** | **21.4** | **54.0** | **97.5** | **12.4** |
| | | | **SD** | **0.0693** | **1.33** | **2.55** | **25.7** | **1.28** | **2.02** | **5.23** | **0.799** |
| | | | **%RSD** | **1.16** | **8.12** | **5.21** | **6.41** | **5.99** | **3.75** | **5.37** | **6.46** |
| Lab Control | 3R4F | 600569 | 4 | 8.46 | 11.9 | 42.1 | 361 | 9.88 | 44.9 | 79.5 | 6.28 |

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# APPENDIX F

## ANALYSIS OF MAINSTREAM CIGARETTE SMOKE FOR TOBACCO SPECIFIC N-NITROSAMINES

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# DETERMINATION OF TOBACCO SPECIFIC N-NITROSAMINES IN MAINSTREAM SMOKE

## I. PRINCIPLE OF METHOD

The method used for this study is detailed in Arista Analytical Method T-020 Determination of Tobacco Specific N-Nitrosamines in Mainstream Smoke.

The tobacco specific n-nitrosamines (TSNAs) are quantitated from mainstream smoke collected on a 44 mm Cambridge filter as the collection media. LC-MS/MS (liquid chromatography coupled to a triple quadrupole mass spectrometer) allows for the determination of the targeted nitrosamines with minimum interference from other components in mainstream smoke. The LC-MS/MS method uses a high performance liquid chromatograph (HPLC) equipped with a C18 reversed phase column to resolve TSNAs from potential interference. Further selectivity is accomplished using a triple quadrupole mass spectrometer with electrospray ionization (ESI) and selective reaction monitoring of characteristic daughter ions. Deuterated internal standards (d-NNN, and d-NNK) are added to the buffer extraction solution and are used to correct for recovery of nitrosamines.

The concentrations of the TSNAs determined by this method are reported in units of mass-to-volume (i.e., ng/mL). The measured concentration, the number of cigarettes smoked, and the sample solution volume(s) are also used to calculate the total analyte mass on a per cigarette basis.

**MS Smoke TSNAs Test Report for:**
**Goodrich Tobacco Company**
**8201 Main Street**
**Suite 6**
**Williamsville, NY 14221**
(b) (4)

**Project Code:** 11066
**Project Start Date:** 11-Mar-11
**Arista Method:** AM-020
**Smoking Regime:** ISO
**Authorized By:** (b) (6)
**Title:** QA

*For questions or concerns, please see the customer feedback form at http://www.aristalabs.com*

| Client Code | Arista Code | Run No. | Port | Puffs (/cig.) | MS TPM (mg/cig.) | NAB (ng/cig.) | NAT (ng/cig.) | NNK (ng/cig.) | NNN (ng/cig.) |
|---|---|---|---|---|---|---|---|---|---|
| Quest 3 Menthol Lights Box | E7791 | 500545 | 2 | 5.52 | 13.2 | 1.22 | 7.84 | 13.4 | 85.4 |
| Quest 3 Menthol Lights Box | E7791 | 500545 | 3 | 5.82 | 13.3 | <LOQ | 7.77 | 12.7 | 78.5 |
| Quest 3 Menthol Lights Box | E7791 | 500545 | 4 | 5.56 | 13.9 | 1.43 | 7.16 | 10.2 | 83.8 |
| | | | **Average** | **5.63** | **13.4** | **1.33** | **7.59** | **12.1** | **82.6** |
| | | | **SD** | **0.16** | **0.39** | **0.15** | **0.37** | **1.69** | **3.65** |
| | | | **%RSD** | **2.89** | **2.90** | **11.3** | **4.92** | **13.9** | **4.42** |
| Lab Control | 3R4F | 500545 | 5 | 8.22 | 10.1 | 15.4 | 120 | 99.6 | 111 |
| ***Limit of Quantitation (ng/mL)*** | | | | | | ***0.195*** | | | |
| ***Approximate Limit of Quantitation (ng/cig.)*** | | | | | | ***0.780*** | | | |



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Richmond, VA 23237, USA

Arista Laboratories
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Certificate No. 1873.01

PROJECT CODE: 11066

# APPENDIX G

# ANALYSIS OF MAINSTREAM CIGARETTE SMOKE FOR SELECTED TRACE ELEMENTS

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ISO 17025 Accredited
Certificate No. 1873.01

# THE DETERMINATION OF SELECTED METALS IN MAINSTREAM SMOKE

## I. PRINCIPLE OF METHOD

The methods used for this study is detailed in Arista Analytical Method AM-021 - The Determination of Selected Metals in Mainstream Cigarette Smoke and Arista Analytical Method AM-036 - The Determination of Mercury in Mainstream Cigarette Smoke.

**The Determination of Arsenic, Cadmium, Chromium, Lead, Nickel and Selenium in Mainstream Cigarette Smoke.**

Smoking is performed on a Borgwaldt RM 20 CSR analytical smoking machine equipped with an electrostatic precipitation (EP) unit. A quartz EP tube is used in the EP unit for smoke collection. The smoke condensate is rinsed out of the EP tube, using methanol. Next, the methanol is evaporated and the sample is digested using nitric acid, hydrogen peroxide and heat. The digested samples are subsequently analyzed by ICP-MS. The ICP-MS used in the method is a Perkin Elmer ICP-MS equipped with a dynamic reaction cell that allows for the determination of elements, including Chromium and Selenium, which normally suffer from severe polyatomic interferences. Ammonia and methane are used as the reaction cell gas for chromium and selenium, respectively. Samples are introduced by nebulization into a radio frequency plasma where energy transfer processes cause desolvation, atomization, and ionization. The ions are extracted from the plasma through a differentially pumped vacuum interface and detected on the basis of their mass to charge ratio by a quadrupole mass spectrometer. The ions transmitted through the quadrupole are detected by an electron multiplier and the ELAN software processes the ion information. Instrumental drift is corrected for by the use of indium and gallium as internal standards, which are added online.

The metals are reported in units of mass-to-volume (ng/mL). The measured concentration, the number of cigarettes smoked, and the sample solution volume(s) are also used to calculate the total analyte mass on a per cigarette basis. The measured concentration of the metals and the number of cigarettes smoked are used to calculate the concentration of each element in units of nanograms per cigarette (ng/cigt).

**The Determination of Mercury in Mainstream Cigarette Smoke.**

Smoking is performed on a Borgwaldt RM 20 CSR. Particulate phase and gas phase mercury in mainstream smoke is trapped using two impingers connected in-series, each containing an acidic potassium permanganate solution. The impinger solutions containing the smoke are subjected to a microwave digestion in which mercury compounds (organic and inorganic) are oxidized to $Hg^{+2}$. Mercury ions ($Hg^{+2}$) in the digestate are reduced by stannous chloride to elemental mercury and analyzed by cold vapor atomic absorption spectrometry.

Mercury is reported in units of mass-to-volume (ng/mL). The measured concentration, the number of cigarettes smoked, and the sample solution volume(s) are used to calculate the total analyte mass in units of nanograms per cigarette (ng/cigt).

**Mainstream Metals Test Report for:**
**Goodrich Tobacco Company**
**8201 Main Street**
**Suite 6**
**Williamsville, NY 14221**
[redacted]

| | |
|---|---|
| **Project Code:** | **11066** |
| **Project Start Date:** | **11-Mar-11** |
| **Arista SOP #:** | **AM-021** |
| **Smoking Protocol:** | **ISO** |
| **Authorized By:** | [redacted] |
| **Title:** | **QA** |

*For questions or concerns, please see the customer feedback form at http://www.aristalabs.com*

| Client Code | Run Number | Ports | Arista Code | Puffs (/cigt) | MS TPM (mg/cigt) | Arsenic (ng/cigt) | Cadmium (ng/cigt) | Chromium (ng/cigt) | Lead (ng/cigt) | Nickel (ng/cigt) | Selenium (ng/cigt) |
|---|---|---|---|---|---|---|---|---|---|---|---|
| Quest 3 Menthol Lights Box | 2011D0004 | C | E7791 | 5.8 | 11.8 | 3.28 | 76.6 | 0.514 | 19.2 | 1.31 | 5.63 |
| Quest 3 Menthol Lights Box | 2011D0004 | D | E7791 | 5.9 | 11.6 | 3.16 | 77.1 | 0.787 | 17.6 | 1.47 | 5.29 |
| Quest 3 Menthol Lights Box | 2011D0004 | E | E7791 | 5.9 | 11.4 | 3.13 | 72.9 | 0.803 | 17.5 | 1.34 | 5.51 |
| | | | **Average** | **5.9** | **11.6** | **3.19** | **75.5** | **0.701** | **18.1** | **1.4** | **5.5** |
| | | | **sd** | **0.1** | **0.17** | **0.08** | **2.25** | **0.162** | **0.98** | **0.08** | **0.17** |
| | | | **%RSD** | **1.0** | **1.5** | **2.4** | **3.0** | **23.2** | **5.4** | **6.1** | **3.2** |
| Lab Control | 2011D0004 | B | 3R4F | 8.8 | 9.77 | 2.89 | 33.5 | <LOQ | 10.7 | 0.846 | 2.26 |
| Lab Control | 2011D0004 | F | 3R4F | 8.8 | 9.74 | 2.56 | 33.3 | <LOQ | 10.0 | 0.710 | 1.99 |
| | | | **Average** | **8.8** | **9.75** | **2.73** | **33.4** | **<LOQ** | **10.4** | **0.778** | **2.12** |
| | | | **sd** | **0.00** | **0.02** | **0.23** | **0.14** | **NA** | **0.53** | **0.10** | **0.19** |
| | | | **%RSD** | **0.0** | **0.2** | **8.6** | **0.4** | **NA** | **5.1** | **12.4** | **8.8** |
| ***Aproximate Limit of Quantitation (ng/cigt)*** | | | | | | ***0.51*** | ***0.49*** | ***0.51*** | ***0.58*** | ***0.52*** | ***0.50*** |

**Mainstream Mercury Test Report for:**
**Goodrich Tobacco Company**
**8201 Main Street**
**Suite 6**
**Williamsville, NY 14221**
(b) (4)

**Project Code:** 11066
**Project Start Date:** 11-Mar-11
**Arista SOP #:** AM-036
**Smoking Protocol:** ISO
**Authorized By:** (b) (6)
**Title:** QA

*For questions or concerns, please see the customer feedback form at http://www.aristalabs.com*

| Client Code | Run # | Port | Arista Code | Puffs (/cigt) | MS TPM | Mercury (ng/cigt) |
|---|---|---|---|---|---|---|
| Quest 3 Menthol Lights Box | 2011D0004 | C | E7791 | 5.8 | 11.8 | 3.3 |
| Quest 3 Menthol Lights Box | 2011D0004 | D | E7791 | 5.9 | 11.6 | 3.0 |
| Quest 3 Menthol Lights Box | 2011D0004 | E | E7791 | 5.9 | 11.4 | 3.1 |
| | | | **Average** | **5.9** | **11.60** | **3.12** |
| | | | **SD** | **0.1** | **0.17** | **0.17** |
| | | | **%RSD** | **1.0** | **1.5** | **5.4** |
| Lab Control | 2011D0004 | B | 3R4F | 8.8 | 9.8 | 2.2 |
| Lab Control | 2011D0004 | F | 3R4F | 8.8 | 9.7 | 2.0 |
| | | | **Average** | **8.8** | **9.75** | **2.08** |
| | | | **SD** | **0.0** | **0.02** | **0.14** |
| | | | **%RSD** | **0.0** | **0.2** | **6.5** |

Arista Laboratories

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*Certificate No. 1873.01*

PROJECT CODE: 11066

# APPENDIX H

## ANALYSIS OF MAINSTREAM CIGARETTE SMOKE FOR PHENOLICS

Arista Laboratories

*ISO 17025 Accredited*
*Certificate No. 1873.01*

# DETERMINATION OF PHENOLIC COMPOUNDS IN MAINSTREAM SMOKE

## I. PRINCIPLE OF METHOD

The method used for this study is detailed in Arista Analytical Method AM-027, Determination of Phenolic Compounds in Mainstream Smoke. Phenolics refers to hydroquinone, resorcinol, catechol, phenol, o-cresol and m+p-cresol.

Phenolics in mainstream tobacco smoke are collected on a 44 mm Cambridge filter pad. The pad is extracted with 20 mL of aqueous acetic acid, and the solution is diluted to a known volume. Before analysis by High Performance Liquid Chromatography (HPLC), the solution is filtered. An HPLC equipped with a reversed-phase column and a fluorescence detector is used for the analysis. This system utilizes an increasing methanol fraction in a methanol:water gradient to elute potentially interfering compounds, and the analytes from the column, at different times. All phenolics are well separated except m-cresol and p-cresol, which coelute. Therefore, only one value is reported for m+p-cresol. The eluted compounds are detected with a fluorescence detector. Hydroquinone is detected with an excitation wavelength of 304 nm and emission wavelength of 338 nm. The other compounds are detected with an excitation wavelength of 274 nm and emission wavelength of 298 nm.

The concentrations of phenolic compounds determined by HPLC are reported in units of mass-to-volume (i.e., µg/mL). The measured concentration, the number of cigarettes smoked, and the sample solution volume(s) are also used to calculate the total mass of phenolics on a per cigarette basis.

**MS Smoke Phenols Test Report for:**
**Goodrich Tobacco Company**
**8201 Main Street**
**Suite 6**
**Williamsville, NY 14221**
[redacted]

**Project Code:** 11066
**Project Start Date:** 11-Mar-11
**Arista Method:** AM-027
**Smoking Regime:** ISO
**Authorized By:** [redacted]
**Title:** QA

*For questions or concerns, please see the customer feedback form at http://www.aristalabs.com*

| Client Code | Arista Code | Run No. | Port | Puffs (/cig.) | MS TPM (mg/cig.) | Hydroquinone (µg/cig.) | Resorcinol (µg/cig.) | Catechol (µg/cig.) | Phenol (µg/cig.) | m- + p-Cresol (µg/cig.) | o-Cresol (µg/cig.) |
|---|---|---|---|---|---|---|---|---|---|---|---|
| Quest 3 Menthol Lights Box | E7791 | 600561 | 2.00 | 5.38 | 13.3 | 22.6 | 0.67 | 26.9 | 16.5 | 14.7 | 4.54 |
| Quest 3 Menthol Lights Box | E7791 | 600561 | 3.00 | 5.36 | 11.4 | 21.3 | 0.58 | 23.7 | 13.8 | 12.4 | 3.81 |
| Quest 3 Menthol Lights Box | E7791 | 600561 | 4.00 | 5.26 | 11.6 | 21.0 | 0.59 | 24.3 | 14.3 | 12.6 | 3.98 |
| | | | **Average** | **5.33** | **12.1** | **21.6** | **0.61** | **25.0** | **14.9** | **13.2** | **4.11** |
| | | | **SD** | **0.06** | **1.04** | **0.83** | **0.05** | **1.73** | **1.44** | **1.27** | **0.38** |
| | | | **%RSD** | **1.21** | **8.60** | **3.84** | **8.13** | **6.93** | **9.71** | **9.57** | **9.24** |
| Lab Control | 3R4F | 600561 | 5.00 | 8.18 | 10.2 | 31.7 | 0.69 | 36.8 | 7.26 | 6.27 | 2.20 |
| Lab Control | 3R4F | 600561 | 12.0 | 8.24 | 10.4 | 33.1 | 0.71 | 38.0 | 7.74 | 6.52 | 2.36 |
| | | | **Average** | **8.21** | **10.3** | **32.4** | **0.70** | **37.4** | **7.50** | **6.40** | **2.28** |
| | | | **SD** | **0.04** | **0.16** | **0.96** | **0.02** | **0.83** | **0.34** | **0.18** | **0.11** |
| | | | **%RSD** | **0.52** | **1.51** | **2.97** | **2.66** | **2.21** | **4.52** | **2.75** | **4.89** |



**Arista Laboratories, Inc.**
1941 Reymet Road
Richmond, VA 23237, USA

Arista Laboratories
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Certificate No. 1873.01

PROJECT CODE: 11066

# APPENDIX I

## ANALYSIS OF MAINSTREAM CIGARETTE SMOKE FOR NITRIC OXIDE **(NO)**

Arista Laboratories
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Certificate No. 1873.01

# THE DETERMINATION OF NITRIC OXIDE IN MAINSTREAM SMOKE

## I. PRINCIPLE OF METHOD

The method used for this study is detailed in Arista Analytical Method AM-029 - The Determination of Nitric Oxide in Mainstream Cigarette Smoke.

The mainstream smoke from one cigarette is collected puff by puff into a mixing chamber attached to an analytical smoking machine. Once the smoke is collected in the mixing chamber a valve is switched to allow the sample to enter the analyzer. A highly sensitive and selective chemiluminescent gas analyzer measures the chemiluminescence of the smoke sample after the completion of each puff. The concentration of NO is proportional to the intensity of the chemiluminescent reaction. Results are generated from puff by puff sample measurements and reported as parts per million and then corrected to STP (standard temperature and pressure) and finally converted to μg NO per cigarette.

**Mainstream Nitric Oxide Test Report for:**
**Goodrich Tobacco Company**
**8201 Main Street**
**Suite 6**
**Williamsville, NY 14221**
(b) (4)

**Project Code:** 11066
**Project Start Date:** 11-Mar-11
**Arista SOP #:** AM-029
**Smoking Protocol:** MS
**Authorized By:** (b) (6)
**Title:** QA

*For questions or concerns, please see the customer feedback form at http://www.aristalabs.com*

| Client Code | Arista Code | Set # | Run # | Puffs (/cigt) | MS TPM (mg/cigt) | NO (μg/cigt) | NOx (μg/cigt) |
|---|---|---|---|---|---|---|---|
| Quest 3 Menthol Lights Box | E7791 | 500548 | 3 | 5.70 | 15.7 | 312 | 539 |
| Quest 3 Menthol Lights Box | E7791 | 500548 | 4 | 6.00 | 14.6 | 315 | 601 |
| Quest 3 Menthol Lights Box | E7791 | 500548 | 5 | 6.00 | 11.7 | 276 | 558 |
| | | | **Average** | **5.90** | **14.0** | **301** | **566** |
| | | | **SD** | **0.173** | **2.07** | **22.0** | **31.9** |
| | | | **%RSD** | **2.94** | **14.8** | **7.29** | **5.64** |
| Lab Control | 3R4F | 500548 | 2 | 9.00 | 11.9 | 247 | 330 |
| Lab Control | 3R4F | 500548 | 10 | 8.80 | 10.8 | 232 | 361 |
| | | | **Average** | **8.90** | **11.4** | **239** | **346** |
| | | | **SD** | **0.141** | **0.778** | **10.2** | **22.0** |
| | | | **%RSD** | **1.59** | **6.85** | **4.27** | **6.37** |

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# APPENDIX J

## ANALYSIS OF MAINSTREAM CIGARETTE SMOKE FOR POLYCYCLIC AROMATIC AMINES (PAA)

Arista Laboratories
*ISO 17025 Accredited*
*Certificate No. 1873.01*

# THE DETERMINATION OF 1-NAPHTHYLAMINE, 2-NAPHTHYLAMINE, 3-AMINOBIPHENYL AND 4-AMINOBIPHENYL (PAAS) IN MAINSTREAM SMOKE

## I. PRINCIPLE OF METHOD

The method used for this study is detailed in Arista Analytical Method AM-030 Determination of 1-naphthylamine, 2-naphthylamine, 3-aminobiphenyl and 4-aminobiphenyl (PAAs) in mainstream smoke.

Mainstream (MS) smoke from test cigarettes is passed through a 44-mm Cambridge filter. After smoking, the filter is weighed and transferred to a Polymethylpentene Erlenmeyer flask containing 25 mL of 5% HCl. Internal standard is added and the sample is shaken for 30 min on a wrist action shaker. The extract is filtered into a separatory funnel and extracted with dichloromethane. The dichloromethane is disposed of as chlorinated waste and sodium hydroxide (50% solution in water) is added to the aqueous phase to make the sample basic. The contents are extracted three times with dichloromethane. The combined dichloromethane extract is derivatized with pentafluoropropionic acid anhydride (PFPA), passed through a Florisil Cartridge clean-up step and concentrated to approximately 2 mL.

Individual analyte concentrations are determined by the internal standard method using gas-chromatography followed by negative chemical ionization with Selected Ion Monitoring (SIM) detection. The concentrations of the analytes, determined by the GC/MSD are reported in units of mass-to-volume (i.e., ng/mL). The measured concentration, the number of cigarettes smoked, and the sample solution volume(s) are used to calculate the total analyte mass on a per cigarette basis.

**Mainstream Aromatic Amines Test Report for:**
**Goodrich Tobacco Company**
**8201 Main Street**
**Suite 6**
**Williamsville, NY 14221**
(b) (4)

**Project Code:** 11066
**Project Start Date:** 11-Mar-11
**Arista SOP #:** AM-030
**Smoking Protocol:** ISO
**Authorized By:** (b) (6)
**Title:** QA

*For questions or concerns, please see the customer feedback form at http://www.aristalabs.com*

| Client Code | Arista Code | Run # | Port | Puffs (/cigt) | MS TPM (mg/cigt) | 1-Naphthyl Amine (ng/cigt) | 2-Naphthyl Amine (ng/cigt) | 3-Amino Biphenyl (ng/cigt) | 4-Amino Biphenyl (ng/cigt) |
|---|---|---|---|---|---|---|---|---|---|
| Quest 3 Menthol Lights Box | E7791 | 500544 | 2 | 5.64 | 13.8 | 11.6 | 6.87 | 2.53 | 1.70 |
| Quest 3 Menthol Lights Box | E7791 | 500544 | 3 | 5.32 | 11.3 | 12.8 | 7.94 | 2.95 | 1.99 |
| Quest 3 Menthol Lights Box | E7791 | 500544 | 4 | 5.12 | 12.3 | 12.6 | 7.49 | 2.62 | 1.86 |
| | | | **Average** | **5.36** | **12.5** | **12.3** | **7.43** | **2.70** | **1.85** |
| | | | **SD** | **0.262** | **1.23** | **0.675** | **0.541** | **0.222** | **0.143** |
| | | | **% RSD** | **4.89** | **9.84** | **5.48** | **7.27** | **8.22** | **7.75** |
| Lab Control | 3R4F | 500544 | 5 | 8.00 | 10.1 | 9.49 | 6.29 | 1.68 | 1.10 |

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PROJECT CODE: 11066

# APPENDIX K

## ANALYSIS OF MAINSTREAM CIGARETTE SMOKE FOR BENZO**[a]**PYRENE **(BaP)**

Arista Laboratories
*ISO 17025 Accredited*
*Certificate No. 1873.01*

# DETERMINATION OF BENZO[a]PYRENE IN MAINSTREAM SMOKE

## PRINCIPLE OF METHOD

The method used for this study is detailed in Arista Analytical Method AM-044 Determination of Selected Polynuclear Aromatic Hydrocarbons (PAHs) in Mainstream Smoke.

Mainstream (MS) smoke from test cigarettes is passed through a 44-mm Cambridge filter. After smoking, the MS filter is weighed and transferred to an amber bottle, then 20 mL of MEOH and internal standard are added and the sample is shaken for 30min. Type-1 water is added and the solution is passed through a C-18 SPE cartridge that has been rinsed with 30% MeOH in Type 1 water. The eluent is discarded and toluene is added to the SPE cartridge for extraction of the B[a]P fraction using vacuum. The toluene extract from the C-18 cartridge is then poured through a Silica Si-1 SPE cartridge and collected in a disposable 15mL culture tube. The sample is transferred to an autosampler vial and diluted 1:1 with acetone for analysis by GC/MSD.

Individual analyte concentrations are determined by the internal standard method using gas chromatography with a DB-17ms column for chromatographic separation of the analytes and selected ion monitoring (SIM) after electron impact ionization. The concentrations of the analytes, determined by the GC/MSD are reported in units of mass-to-volume (i.e., ng/mL). The measured concentration, the number of cigarettes smoked, and the sample solution volume(s) are used to calculate the total analyte mass on a per cigarette basis.

**Mainstream B[a]P Test Report for:**
**Goodrich Tobacco Company**
**8201 Main Street**
**Suite 6**
**Williamsville, NY 14221**
(b) (4)

**Project Code:** 11066
**Project Start Date:** 11-Mar-11
**Arista SOP #:** AM-044
**Smoking Protocol:** ISO
**Authorized By:** (b) (6)
**Title:** QA

*For questions or concerns, please see the customer feedback form at http://www.aristalabs.com*

| Client Code | Arista Code | Run # | Port | Puffs (/cigt) | MS TPM (mg/cigt) | B[a]P (ng/cigt) |
|---|---|---|---|---|---|---|
| Quest 3 Menthol Lights Box | E7791 | 600549 | 2 | 5.98 | 14.0 | 5.31 |
| Quest 3 Menthol Lights Box | E7791 | 600549 | 3 | 5.80 | 13.4 | 5.76 |
| Quest 3 Menthol Lights Box | E7791 | 600549 | 4 | 5.72 | 13.1 | 5.77 |
| | | | **Average** | **5.83** | **13.5** | **5.61** |
| | | | **SD** | **0.133** | **0.458** | **0.263** |
| | | | **%RSD** | **2.28** | **3.39** | **4.69** |
| Lab Control | 3R4F | 600549 | 5 | 8.42 | 10.6 | 5.58 |

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PROJECT CODE: 11066

# APPENDIX L

# DETERMINATION OF CARBONYLS IN MAINSTREAM CIGARETTE SMOKE BY UHPLC USING UV DETECTION

Arista Laboratories

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# DETERMINATION OF CARBONYLS IN MAINSTREAM SMOKE BY UHPLC

## I. PRINCIPLE OF METHOD

The method used for this study is detailed in Arista Analytical Method AM-076 Determination of Carbonyls and Aldehydes in Mainstream Smoke.

Mainstream smoke is collected in two impingers containing acidified 2,4-dinitrophenylhydrazine (DNPH) solution. After smoking, the combined solution from the impingers containing the derivatized carbonyls is stabilized by adding pyridine and subsequently analyzed by UHPLC. Individual carbonyl concentrations are determined by an external standard method using UV detection. Note that for this method, the term carbonyls refers to the following compounds: formaldehyde, acetaldehyde, acrolein, acetone, propionaldehyde, crotonaldehyde, butyraldehyde & methylethylketone.

A UHPLC (Ultra High Pressure Liquid Chromatograph) equipped with a reversed phase column and a fixed wavelength UV detector is used for the analysis of the samples. A gradient is used to separate interfering compounds from the analytes of interest. The eluting compounds are detected on the basis of their absorbance at 355 nm. The detector's responses of the hydrazone derivatives of the carbonyls are calibrated against standard solutions of known concentration to form a linear calibration curve.

The concentrations of the carbonyls determined by UHPLC are reported in units of mass-to-volume (i.e., µg/mL). The measured concentration, the number of cigarettes smoked, and the sample solution volume(s) are also used to calculate the total analyte mass on a per cigarette basis.

**MS Smoke Carbonyls Test Report for:**
**Goodrich Tobacco Company**
**8201 Main Street**
**Suite 6**
**Williamsville, NY 14221**
[redacted]

**Project Code:** 11066
**Project Start Date:** 11-Mar-11
**Arista Method:** AM-076
**Smoking Regime:** ISO
**Authorized By:** [redacted]
**Title:** QA

*For questions or concerns, please see the customer feedback form at http://www.aristalabs.com*

| Client Code | Arista Code | Run No. | Port | Puffs (/cig.) | Formaldehyde (µg/cig.) | Acetaldehyde (µg/cig.) | Acetone (µg/cig.) | Acrolein (µg/cig.) | Propionaldehyde (µg/cig.) | Crotonaldehyde (µg/cig.) | Methylethylketone (µg/cig.) | Butyraldehyde (µg/cig.) |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Quest 3 Menthol Lights Box | E7791 | 600548 | 1 | 6.00 | 11.8 | 763 | 353 | 51.5 | 57.1 | 18.8 | 90.9 | 34.4 |
| Quest 3 Menthol Lights Box | E7791 | 600548 | 2 | 6.20 | 11.9 | 798 | 377 | 52.8 | 58.8 | 19.1 | 95.0 | 34.1 |
| Quest 3 Menthol Lights Box | E7791 | 600548 | 3 | 6.00 | 11.5 | 796 | 369 | 55.5 | 59.1 | 18.9 | 93.3 | 34.7 |
| | | | **Average** | **6.07** | **11.8** | **786** | **366** | **53.3** | **58.3** | **18.9** | **93.1** | **34.4** |
| | | | **SD** | **0.12** | **0.24** | **19.5** | **12.3** | **2.08** | **1.06** | **0.14** | **2.04** | **0.30** |
| | | | **%RSD** | **1.90** | **2.01** | **2.48** | **3.35** | **3.90** | **1.82** | **0.72** | **2.19** | **0.87** |
| Lab Control | 3R4F | 600548 | 4 | 8.80 | 14.7 | 758 | 340 | 55.6 | 59.2 | 14.0 | 95.7 | 31.0 |



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1941 Reymet Road
Richmond, VA 23237, USA

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# APPENDIX M

## DETERMINATION OF EUGENOL IN MAINSTREAM AND SIDESTREAM SMOKE

Arista Laboratories

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*A2LA Certificate No. 1873.01*

# EUGENOL IN MAINSTREAM AND SIDESTREAM SMOKE

## PRINCIPLE OF METHOD

The method used for this study is detailed in Arista Analytical Method AM-087 Determination of Eugenol in Mainstream and Sidestream Smoke.

Eugenol is quantitated in mainstream and sidestream smoke from unconditioned cigarettes.

The mainstream smoke is collected on a 44mm Cambridge filter pad. After smoking, the pad is placed into an Erlenmeyer flask and extracted with 50mL of extraction solution. The flask is shaken for 30 minutes using an orbital shaker set at ~200 rpm. The extract is then filtered through a 0.45-µm filter directly into an ALS vial and then analyzed by HPLC with UV detection. The eugenol concentration in mainstream smoke is determined by an external standard calibration and reported in µg/cigarette.

The sidestream smoke is collected on a 44mm Cambridge filter, which is followed by an impinger filled with 30mL of the trapping solution to collect the analytes not retained by the filter pad. The 44-mm Cambridge filter pad in a holder is connected directly to an out-port of a fishtail chimney. The impinger is placed in an ice-bath in which the temperature is maintained between 0-3 °C. A flow rate of 3.0 liters per minute is maintained during smoking. After smoking, the pad and the trapping solution from the impinger are combined into an Erlenmeyer flask. The fishtail chimney is rinsed with 50mL of the extraction solution and the rinse is added into the flask. The flask is shaken for 30 minutes using an orbital shaker set at ~200 rpm. The extract is then filtered through a 0.45-µm filter directly into an ALS vial and then analyzed by HPLC with UV detection. The eugenol concentration in sidestream smoke is determined by an external standard calibration and reported in µg/cigarette.

**MS Smoke Eugenol Test Report for:**
**Goodrich Tobacco Company**
**8201 Main Street**
**Suite 6**
**Williamsville, NY 14221**
(b) (4)

**Project Code:** 11066
**Project Start Date:** 11-Mar-11
**Arista Method:** AM-087
**Smoking Regime:** ISO
**Authorized By:** (b) (6)
**Title:** QA

*For questions or concerns, please see the customer feedback form at http://www.aristalabs.com*

| Client Code | Arista Code | Run No. | Port | Puffs (/cig.) | MS TPM (mg/cig.) | Eugenol (µg/cig.) |
|---|---|---|---|---|---|---|
| Quest 3 Menthol Lights Box | E7791 | 301970 | 2 | 4.83 | 12.7 | <LOQ |
| Quest 3 Menthol Lights Box | E7791 | 301970 | 3 | 5.40 | 12.5 | <LOQ |
| Quest 3 Menthol Lights Box | E7791 | 301970 | 4 | 5.07 | 12.8 | <LOQ |
| | | | **Average** | **5.10** | **12.6** | **NA** |
| | | | **SD** | **0.28** | **0.16** | **<LOQ** |
| | | | **%RSD** | **5.58** | **1.25** | **<LOQ** |
| | | | | | | **% Recovery** |
| Lab Control | 3R4F Recovery | 301970 | 5 | 7.40 | 9.37 | 102 |
| ***Limit of Quantitation (µg/mL)*** | | | | | | ***6.17*** |
| ***Approximate Limit of Quantitation (µg/cig)*** | | | | | | ***103*** |


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Arista Laboratories

*ISO 17025 Accredited*
*Certificate No. 1873.01*

# APPENDIX N

## DETERMINATION OF pH IN MAINSTREAM CIGARETTE SMOKE BY THE HEALTH CANADA OFFICIAL

Arista Laboratories

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# DETERMINATION OF PH OF MAINSTREAM SMOKE

## PRINCIPLE OF METHOD

The method used for this study is detailed in Arista Analytical Method AM-093 - The Determination of pH in Mainstream Smoke – Health Canada Official Method T-113.

The mainstream smoke from cigarettes is collected on a puff-by-puff basis. Using a modified pH electrode, the acid-base effect of both the condensate and gas phase is monitored and recorded. Data is directly transferred from the pH meter to an Excel spreadsheet at a fixed print interval. An average pH of the smoke is determined by averaging all of the data points transferred.

**Mainstream pH Test Report for:**
**Goodrich Tobacco Company**
**8201 Main Street**
**Suite 6**
**Williamsville, NY 14221**
(b) (4)

**Project Code:** 11066
**Project Start Date:** 11-Mar-11
**Arista SOP #:** AM-093
**Smoking Protocol:** MS
**Authorized By:** (b) (6)
**Title:** QA

*For questions or concerns, please see the customer feedback form at http://www.aristalabs.com*

| Client Code | Arista Code | Set # | Run # | pH Meter | Puffs/Cigt | pH |
|---|---|---|---|---|---|---|
| Quest 3 Menthol Lights Box | E7791 | 502091 | 3 | 1 | 6.00 | 6.11 |
| Quest 3 Menthol Lights Box | E7791 | 502091 | 4 | 1 | 6.00 | 6.05 |
| Quest 3 Menthol Lights Box | E7791 | 502091 | 5 | 1 | 6.00 | 6.01 |
| | | | | **Average** | **6.00** | **6.06** |
| | | | | **SD** | **0.00** | **0.0466** |
| | | | | **%RSD** | **0.00** | **0.770** |
| 3R4F | 3R4F | 502091 | 2 | 1 | 8.00 | 6.11 |
| 3R4F | 3R4F | 502091 | 6 | 1 | 8.00 | 6.08 |
| | | | | **Average** | **8.00** | **6.10** |
| | | | | **SD** | **0.00** | **0.0232** |
| | | | | **%RSD** | **0.00** | **0.381** |

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PROJECT CODE: 11066

# APPENDIX O

## ANALYSIS OF MAINSTREAM CIGARETTE SMOKE FOR HYDROGEN CYANIDE (HCN)

Arista Laboratories
*ISO 17025 Accredited*
*Certificate No. 1873.01*

# DETERMINATION OF HYDROGEN CYANIDE IN MAINSTREAM SMOKE

## I. PRINCIPLE OF METHOD

The a method used for this study is detailed in Arista Analytical Method AM-111 Hydrogen Cyanide in Mainstream and Sidestream Smoke by Continuous Flow Analyzer.

HCN is determined from mainstream smoke collected on a Cambridge filter followed by one non-fritted impinger containing 0.1M aqueous sodium hydroxide as the collection media. The Cambridge filter pad is extracted into 0.1M aqueous sodium hydroxide and analyzed separately from the impinger solution. A continuous flow analyzer is used to add the required reagents to a sample of the pad extract and the impinger trapping solutions. Hydrogen cyanide in the sample is converted to cyanogen chloride by the addition of an aqueous solution of chloramine-T. The cyanogen chloride then reacts with pyridine and barbituric acid to produce a reddish-violet color complex that is measured photometrically at 570nm. The Beer-Lambert Law linearly relates formation of the colored complex to concentration.

The concentration of HCN is reported in units of mass-to-volume (i.e., µg/mL). The measured HCN concentration, the number of cigarettes smoked, and the sample solution volumes are used to calculate the total mass of HCN on a per cigarette basis.

**Mainstream HCN Test Report for:**
**Goodrich Tobacco Company**
**8201 Main Street**
**Suite 6**
**Williamsville, NY 14221**
(b) (4)

**Project Code:** 11066
**Project Start Date:** 11-Mar-11
**Arista SOP #:** AM-111
**Smoking Protocol:** ISO
**Authorized By:** (b) (6)
**Title:** QA

*For questions or concerns, please see the customer feedback form at http://www.aristalabs.com*

| Client Code | Arista Code | Run # | Port | Puffs (/cigt) | MS TPM (mg/cigt) | Impinger HCN (µg/cigt) | Pad HCN (µg/cigt) | Total HCN (µg/cigt) |
|---|---|---|---|---|---|---|---|---|
| Quest 3 Menthol Lights Box | E7791 | 600550 | 1 | 5.53 | 13.3 | 202 | 86.7 | 289 |
| Quest 3 Menthol Lights Box | E7791 | 600550 | 2 | 5.77 | 16.0 | 211 | 102.3 | 313 |
| Quest 3 Menthol Lights Box | E7791 | 600550 | 3 | 5.67 | 14.6 | 221 | 98.5 | 320 |
| | | | **Average** | **5.66** | **14.6** | **211** | **95.8** | **307** |
| | | | **SD** | **0.117** | **1.32** | **9.53** | **8.12** | **16.1** |
| | | | **%RSD** | **2.07** | **9.01** | **4.51** | **8.47** | **5.26** |
| Lab Control | 3R4F | 600550 | 4 | 8.67 | 11.0 | 73.1 | 46.8 | 120 |