Project Code: 0318-112
Quote: (b) (4) (b) (4)

The Determination of Selected Analytes in Cigarettes

Prepared for:

22nd Century Group, Inc.
9530 Main Street
Clarence, NY 14031

Report Issued: 4 June 2019
Report Revision 1.0 Issued: 12 June 2019

ENTHALPY ANALYTICAL

www.enthalpy.com

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I. REPORT REVISION HISTORY

Revision Type (Initial/Revision)	Version Number	Report Date	Reason for Revision
Initial	0.0	04Jun2019	Initial release
Revision	1.0	12Jun2019	Changed view of TNC Data File

II. OVERVIEW

The objective of this study was to determine the concentration of selected analytes in cigarettes.

The client provided two samples, which are listed in Table I. Twenty replicates per sample were determined to be used for testing of TNCO, three replicates per sample were determined to be used for Oven Volatiles testing, five replicates per sample were used for Water Activity testing, and seven replicates per sample were used for Nicotine testing.

The analyses requested for this study are listed in Table II. Analytical method summaries and results are included in the attached appendices. The laboratory control type used for acceptance is included on each final table for a given test method, if applicable.

Any method deviation(s) that occurred during the course of this study are included in Section IV, C: Quality Control and Reporting Notes.

III. TESTING FACILITY

Activities were conducted at:

Enthalpy Analytical, LLC
800-1 Capitola Drive
Durham, NC 27713-4385
Phone: (919) 850 - 4392
Fax: (919) 850 - 9012

Enthalpy Analytical, LLC
1470 East Parham Road
Richmond, VA. 23228-2300
Phone: (804) 887 - 2100
Fax: (804) 271 – 5594

**Please refer to Table II for the specific site location where each analysis was conducted.*

IV. TEST SAMPLES

A. Receipt, Storage and Conditioning of Test Samples

- Enthalpy Analytical's Richmond, VA facility received the samples on 7 May 2019 after being relinquished by 22nd Century Group. The samples were received at ambient temperature and in good condition and assigned project code: 0318-112. A portion of the samples were sent to Enthalpy's Durham, NC facility under project code: 0519-503/0318-112. Prior to, during, and after analysis, the samples were kept under lock with access only to authorized personnel by Enthalpy Analytical, LLC.
- The sample designations are given in Table I.

- Storage was maintained at 25 ·C/60 %RH test for 0,3,6, 9, and 12 months for standard stability evaluation and 40 ·C/75 %RH test for 1, 3, 6 months for accelerated stability evaluation.

Table I. Sample Coding

Client Sample ID	Enthalpy Sample ID	Sample Type	Quantity Received
(b) (4) PARE Regular King Box - 12 month 25C/60% RH	A	cigarettes	480
(b) (4) PARE Menthol King Box - 12 month 25C/60% RH	B	cigarettes	480

B. Sample Analysis and Constituents List

All applicable analytical methods have been fully validated (unless otherwise specified). For lists of specific compounds of interest identified in each analyte group to be reported, refer to Table II.

All sample analyses performed were conducted on site from the time the samples were received (7 May 2019) to the date the final report was issued (4 June 2019).

For combustible smoke analyses, smoking of the products was carried out using SM450 20-port linear analytical smoking machines. Each product was placed into a holder and smoked according to the applicable analytical method(s) and collected on Cambridge filter pads or impingers. Each product was smoked in replicates of twenty via the ISO smoking regime.

- ISO Smoking Regime: five products smoked using a 35mL two second puff every 60 seconds using a bell curve. Ventilation holes are not covered.

Refer to method summaries for more information.

Table II. Constituents List

Analyses	Analyte(s)	Analytical Method Reference and Version #	Validation Report #	*Site Location
Nicotine, Menthol, and Water in Mainstream Smoke by GC Carbon Monoxide in Mainstream and Sidestream Smoke by Non-Dispersive Infrared Analyzer	Tar, Nicotine, Water, Carbon Monoxide	AM-001 v10.0 AM-007 v4.2	VR-001	Richmond
Water Activity in Smokeless Tobacco	Water Activity	AM-233	VR-233	Richmond
Analysis of Total Moisture in Tobacco	Oven Volatiles	ENT046 v1.0	CDC Protocol	Durham
GC/MS Analysis of Nicotine and Alkaloids in Tobacco	Nicotine	ENT246 V2.0	TPT027	Durham

C. Quality Control and Reporting Notes

All quality control samples were within limits unless otherwise noted. The results presented in this report are representative of the samples as provided to the laboratory.

D. Sample Generation and Records

All analytical results generated in this report were conducted per protocol-specific parameters and conditions.

Records will be stored by Enthalpy for a period of ten years. Following this period, all records will be destroyed. Electronic records are maintained indefinitely.

Sample and calibration curve chromatograms are available upon request.

E. Sample and LOD/LOQ Concentration Calculations

The analyte concentration is determined by the internal standard calibration method using the regression equation derived from the calibration curve. Results are then converted and reported on a per weight, per device/cigarette, or per puff basis. Example calculations are provided below for varying analysis types. Please note in the example calculations, nanograms (ng) are shown, however the results may be expressed in other units (mg, µg, pg). Dilution factors may also be used in some methods depending on the sample preparation procedure.

Please note that approximate LOD and LOQ values are calculated using averages for each assay and are not specific to individual samples.

E-Liquid and Tobacco:

E-liquid and tobacco analysis results may be reported on a mass/mass basis, catch weight basis (ng) or mass/volume (ng/mL). The calculation is as follows:

$$\text{Analyte Concentration (ng/g)} = \frac{\text{Concentration (ng/mL)} \times \text{Dilution Factor} \times \text{Extraction Volume (mL)}}{\text{E-Liquid or Tobacco Weight (g)}}$$

In instances where results are required to be reported on a dry weight basis (DWB), the moisture content of a sample (expressed as moisture or oven volatiles) can be determined by any one of several different analytical methods. The following calculation is used to convert an “as is” result to a “dry weight basis” result:

$$\text{Analyte Concentration (ng/g DWB)} = \frac{\text{Analyte Concentration (ng/g)} \times 100}{(100 - \%\ \text{Moisture})}$$

Aerosol and Smoke:

The final results for aerosol or smoke analysis may be reported as catch weight basis (ng), ng/puff, ng/cig, ng/device, or ng/g ACM (aerosol collected mass). The final sample concentrations are calculated using the following equations:

$$\text{Analyte Amount (ng/puff)} = \frac{\text{Concentration (ng/mL)} \times \text{Dilution Factor} \times \text{Extraction Volume (mL)}}{\text{Total \# of Puffs}}$$

$$\text{Analyte Amount (ng/cig or ng/device)} = \frac{\text{Conc. (ng/mL)} \times \text{Dilution Factor} \times \text{Extraction Volume (mL)}}{\text{Total \# of Cigar(ette)s or Devices}}$$

$$\text{Analyte Amount (ng/g ACM)} = \frac{\text{Concentration (ng/mL)} \times \text{Dilution Factor} \times \text{Extraction Volume (mL)}}{\text{ACM (g)}}$$

Limits of Detection and Quantitation:

The Limit of Detection (LOD) and Limit of Quantitation (LOQ) values are calculated using the same formulas above. The LOD/LOQ value in mass/mL is converted to the final unit by multiplying by the extraction volume and dividing by the preferred unit (grams, puffs, device, etc.). An example calculation is shown below:

$$\text{Limit of Detection (ng/g)} = \frac{\text{LOD (ng/mL) x Dilution Factor x Extraction Volume (mL)}}{\text{E-Liquid or Tobacco Weight (g)}}$$

V. QUALITY STATEMENT\SIGNATURES

I certify to the best of my knowledge, all analytical data presented in this report summary:

- Has been reviewed for consistency and completeness.
- Is accurate, error-free, and legible.
- Has been conducted in accordance with the ISO/IEC 17025 standard; in instances where no regulatory guidance exists or is not applicable, the study was conducted in accordance with Enthalpy Analytical quality and technical procedures, and/or approved protocol.

Outliers were evaluated, where deemed necessary. If any were discovered and an assignable cause was determined, they were noted and repeats conducted.

This report is considered to describe accurately the procedures used in this study and the results obtained.

QA Reviewed By: Electronically signed by: (b) (6) (ARISTA\mjohnso2)
Date: Wednesday, June 12, 2019 7:17:13 AM (GMT -04:00)
Reason: QA Reviewed

General Reporting Notes

Acronym	Name	Explanation
J ***<LOQ***	*J-Flag* *Less than LOQ*	Indicates detection of the analyte, but at a value less than the LOQ. The laboratory can positively identify the presence of the analyte of interest, but it cannot be reliably quantitated.
DF	*Dilution Factor*	This number represents a dilution of the sample during the preparation and/or analysis process. The analytical result taken from a laboratory instrument is multiplied by the DF to determine the final, undiluted sample result.
E	*E-Flag*	Indicates an analytical result exceeding the highest calibration point. The associated value should be considered an estimate.
PCS or PCM	*Process Control Sample or Process Control Monitor*	Clean matrix or a reference matrix that is prepared and analyzed using the same reagents, procedures and spiking standards (if applicable) used for the client samples. Used to assess the control of the laboratory's analytical system. Examples: LCS, 3R4F, CM7
LOQ	*Limit of Quantitation*	(aka: *Lowest Standard Value* or *Lower Curve Limit*). The laboratory cannot reliably quantitate analytes of interest below this value within method criteria. The result is considered an estimate.
LOD	*Limit of Detection*	The LOD is how low the instrument can consistently detect analytes in the presence of the sample matrix.
MS	*Matrix Spike*	An aliquot of an actual sample spiked with a known amount of analyte to determine possible percent recovery. The MS indicates what effect the sample matrix may have on the target analyte.
ND	*Non-Detect*	Indicates an analytical result below the LOD.
ACM	*Aerosol Collection Mass*	The amount of particulate captured on the pad during aerosol collection.
TPM	*Total Particulate Matter*	The amount of particulate captured on the pad during smoking.
WtLoss	*Device Weight Loss*	Weight loss is the amount of e-liquid mass used during smoking. The device is weighed before and after smoking.

- **Significant Figures**: Three (3) significant figures are reported unless the reported value is much greater than unity (1.00) in the units expressed, the number is rounded to a whole number. For example, a value of 1,456.45 µg/mL is rounded to 1,456 µg/mL. There are four significant digits displayed, but no confidence should be placed on more than two significant digits.

- **Manual Integration**: The data systems used for processing will flag manually integrated peaks with an "M." Several reasons a peak may be manually integrated (listed below) will be identified by two-letter designations on sample chromatograms, if provided in the report. These codes will accompany the analyst's manual integration stamp placed next to the compound name on the chromatogram.

    - ***NI***: The peak was *not integrated* by the software
    - ***II***: The peak was *integrated incorrectly* by the software
    - ***WP***: The *wrong peak* was integrated by the software

APPENDIX A

TNCO METHOD SUMMARY AND RESULTS

NICOTINE, MENTHOL, AND WATER IN MAINSTREAM SMOKE BY GC

ENTHALPY ANALYTICAL METHOD AM-001, VERSION 10.0

Data was generated by Enthalpy Analytical (EA) (Richmond, Virginia, USA), LLC in accordance with EA Method AM-001. Smoke particulate is collected on a Cambridge filter pad (CFP), and dissolved in extraction solution (isopropanol with internal standards). An aliquot of the sample solution is analyzed by gas chromatography (GC) and the water, nicotine, and menthol content of the smoke condensate is determined. Since water is a measured and significant constituent for this analytical method, environmental water content should be uniform throughout sample collection and analysis. Method blanks are collected by extracting conditioned, unused CFPs. The method blanks are used to determine the concentration of water inherent in the CFPs and extraction solution. The average water content of the method blanks is subtracted from the water content of the sample extracts to obtain the final, corrected water content for each smoke sample. Nicotine and menthol are separated from other constituents using a packed Carbowax column connected to a Flame Ionization Detector (FID). A packed Porapak QS column and Thermal Conductivity Detector (TCD) are used for the determination of water. The tar (nicotine free dry particulate matter or NFDPM) is calculated by subtracting nicotine and water from the TPM. Results are typically reported in milligrams per cigarette (mg/cig) for each smoke sample.

The method was included on the laboratory's A2LA scope of accreditation at the time of testing and internal quality control procedures were followed. Enthalpy Analytical, LLC – Richmond is accredited by the American Association for Laboratory Accreditation (Certificate number 1873.01).

CARBON MONOXIDE IN MAINSTREAM AND SIDESTREAM SMOKE BY NON-DISPERSIVE INFRARED ANALYZER

ENTHALPY ANALYTICAL METHOD AM-007, VERSION 4.2

Carbon Monoxide (CO) data was generated by Enthalpy Analytical (EA) (Richmond, Virginia, USA), LLC in accordance with EA Method AM-007. Cigar(ette)s are smoked using an analytical smoking machine following a pre-determined smoking procedure (ISO, Canadian Intense, CORESTA, or protocol specific). The vapor phase of the smoke is collected in gas sampling bags attached to the smoking machine configured to the requested puffing parameters. The sidestream smoke is pulled by vacuum pumps through a glass fishtail chimney which sit over the burning cigar(ette). The vapor phase of the smoke is collected in gas sampling bags attached to the exhaust port of the vacuum pumps. 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 cigar(ette)s, the cigar(ette) puff count, the puff volume, and ambient conditions, the % CO is converted to milligrams per cigar(ette) (mg/cig).

The method was included on the laboratory's A2LA scope of accreditation at the time of testing and internal quality control procedures were followed. Enthalpy Analytical, LLC – Richmond is accredited by the American Association for Laboratory Accreditation (Certificate number 1873.01).

Enthalpy Analytical

0318-112 AM-001 TNC ISO

22nd Century Group, Inc.

Analyst: (b) (6)

Approximate Limit Of Quantitation (mg/cig):	0.00482	3.17	0.201
Approximate Limit of Detection(mg/cig)):	0.00161	1.06	0.0670

Summary

Sample Name	Sample Code	Device Code	Run No	Port	Cigs	Puffs/Cig	TPM mg/cig	Tar NFDPM mg/cig	Nicotine mg/cig	CO mg/cig	Water mg/cig
Laboratory Controls	AM001 R1I P4 0318-112 3R4F		1	4	5	7.82	9.24	7.90	0.713	10.5	0.624
	AM001 R2I P6 0318-112 3R4F		2	6	5	7.82	8.56	7.34	0.671	9.84	0.551
	AM001 R3I P13 0318-112 3R4F		3	13	5	8.12	8.80	7.62	0.714	10.4	0.466
	Average					7.92	8.87	7.62	0.699	10.2	0.547
	StdDev					0.17	0.34	0.28	0.025	0.4	0.079
	RSD					2.2%	3.9%	3.7%	3.5%	3.4%	14.5%
(b) (4) PARE Regular King Box - 12 month 25C/60% RH	AM001 R1I P1 0318-112 A		1	1	5	6.00	7.08	6.64	0.0245	11.9	0.413
	AM001 R1I P8 0318-112 A		1	8	5	5.60	6.58	6.23	0.0246	10.6	0.327
	AM001 R1I P9 0318-112 A		1	9	5	5.86	7.14	6.68	0.0252	11.9	0.432
	AM001 R1I P11 0318-112 A		1	11	5	5.90	7.24	6.68	0.0269	12.5	0.538
	AM001 R1I P12 0318-112 A		1	12	5	5.70	7.38	6.96	0.0268	12.7	0.397
	AM001 R1I P13 0318-112 A		1	13	5	5.78	6.86	6.44	0.0273	11.9	0.394
	AM001 R1I P15 0318-112 A		1	15	5	5.52	7.40	6.87	0.0250	11.8	0.507
	AM001 R2I P4 0318-112 A		2	4	5	5.60	7.08	6.59	0.0263	11.8	0.467
	AM001 R2I P5 0318-112 A		2	5	5	6.06	7.28	6.79	0.0252	12.8	0.462
	AM001 R2I P8 0318-112 A		2	8	5	5.92	8.04	7.32	0.0278	11.9	0.688
	AM001 R2I P9 0318-112 A		2	9	5	6.00	7.36	6.72	0.0296	12.4	0.607
	AM001 R2I P10 0318-112 A		2	10	5	6.04	7.88	7.19	0.0274	13.4	0.660
	AM001 R2I P11 0318-112 A		2	11	5	5.54	7.32	6.72	0.0251	12.1	0.577
	AM001 R2I P15 0318-112 A		2	15	5	6.00	7.58	6.96	0.0302	12.2	0.592
	AM001 R3I P2 0318-112 A		3	2	5	5.96	8.26	7.71	0.0287	13.6	0.517
	AM001 R3I P4 0318-112 A		3	4	5	5.70	7.18	6.77	0.0248	12.4	0.382
	AM001 R3I P5 0318-112 A		3	5	5	5.84	7.52	7.06	0.0277	12.5	0.434
	AM001 R3I P9 0318-112 A		3	9	5	5.96	6.96	6.42	0.0243	11.8	0.513
	AM001 R3I P10 0318-112 A		3	10	5	5.26	6.82	6.50	0.0232	11.8	0.295
	AM001 R3I P11 0318-112 A		3	11	5	5.90	7.44	6.97	0.0270	12.8	0.445
	Average					5.81	7.32	6.81	0.0264	12.2	0.482
	StdDev					0.21	0.41	0.34	0.0019	0.6	0.106
	RSD					3.7%	5.6%	5.0%	7.1%	5.3%	21.9%

Enthalpy Analytical

0318-112 AM-001 TNC ISO

22nd Century Group, Inc.

Analyst: (b) (6)

Approximate Limit Of Quantitation (mg/cig):	0.00482	3.17	0.201
Approximate Limit of Detection(mg/cig)):	0.00161	1.06	0.0670

Summary

Sample Name	Sample Code	Device Code	Run No	Port	Cigs	Puffs/Cig	TPM mg/cig	Tar NFDPM mg/cig	Nicotine mg/cig	CO mg/cig	Water mg/cig
(b) (4) PARE Menthol King Box - 12 month 25C/60% RH	AM001 R1I P2 0318-112 B		1	2	5	5.18	6.48	6.15	0.0213	10.6	0.311
	AM001 R1I P3 0318-112 B		1	3	5	5.40	7.36	6.91	0.0261	11.5	0.424
	AM001 R1I P5 0318-112 B		1	5	5	5.22	6.86	6.54	0.0251	11.1	0.297
	AM001 R1I P6 0318-112 B		1	6	5	5.36	6.12	5.87	0.0219	10.3	0.228
	AM001 R1I P10 0318-112 B		1	10	5	5.44	6.92	6.42	0.0259	11.9	0.475
	AM001 R1I P14 0318-112 B		1	14	5	5.22	6.50	6.13	0.0230	10.6	0.347
	AM001 R1I P16 0318-112 B		1	16	5	5.48	6.78	6.40	0.0212	10.2	0.354
	AM001 R2I P1 0318-112 B		2	1	5	5.72	8.20	7.49	0.0256	12.0	0.683
	AM001 R2I P2 0318-112 B		2	2	5	5.54	7.26	6.83	0.0254	11.5	0.407
	AM001 R2I P3 0318-112 B		2	3	5	5.46	6.76	6.34	0.0247	10.8	0.394
	AM001 R2I P7 0318-112 B		2	7	5	5.52	6.64	6.28	0.0230	11.5	0.339
	AM001 R2I P12 0318-112 B		2	12	5	5.82	7.56	7.01	0.0255	11.6	0.524
	AM001 R2I P14 0318-112 B		2	14	5	5.20	7.02	6.52	0.0238	11.9	0.474
	AM001 R3I P1 0318-112 B		3	1	5	5.88	7.96	7.44	0.0250	12.6	0.499
	AM001 R3I P3 0318-112 B		3	3	5	5.30	6.34	5.68	0.0226	10.8	0.639
	AM001 R3I P6 0318-112 B		3	6	5	5.46	6.08	5.83	0.0218	10.4	0.233
	AM001 R3I P7 0318-112 B		3	7	5	5.12	6.34	6.08	0.0239	11.1	0.236
	AM001 R3I P8 0318-112 B		3	8	5	5.46	5.90	5.65	0.0214	9.70	0.225
	AM001 R3I P12 0318-112 B		3	12	5	5.24	6.58	6.28	0.0232	11.0	0.282
	AM001 R3I P14 0318-112 B		3	14	5	5.32	6.62	6.29	0.0239	11.0	0.306
	Average					5.42	6.81	6.41	0.0237	11.1	0.384
	StdDev					0.21	0.61	0.52	0.0016	0.71	0.133
	RSD					3.9%	8.9%	8.1%	6.9%	6.4%	34.5%
Collection Blanks	AM001 R1I P7 0318-112 blank		1	7	5	8.00	ND	ND	ND	0.119	0.00281
	AM001 R2I P13 0318-112 blank		2	13	5	8.00	ND	ND	ND	0.239	ND
	AM001 R3I P15 0318-112 blank		3	15	5	8.20	ND	ND	ND	0.122	ND
	Average					8.07	-0.393	-0.394	ND	0.160	0.000937
	StdDev					0.12	0.070	0.072	NA	0.068	0.001623
	RSD					1.4%	17.9%	18.2%	NA	42.6%	173.2%

APPENDIX B

WATER ACTIVITY METHOD SUMMARY AND RESULTS

WATER ACTIVITY IN SMOKELESS TOBACCO

ENTHALPY ANALYTICAL METHOD AM-233, VERSION 6.0

Water activity data was generated by Enthalpy Analytical, Inc. (Richmond, VA), LLC in accordance with Enthalpy SOP AM-233. Instrument verification and/or calibration are performed prior to sample analysis. A portion of tobacco is taken and placed into a water activity sample cup. The sample cup is then placed into the water activity meter and the sample is analyzed using a laser sensor.

The method was included on the laboratory's A2LA scope of accreditation at the time of testing and internal quality control procedures were followed. Enthalpy Analytical, LLC – Richmond is accredited by the American Association for Laboratory Accreditation (Certificate number 1873.01).

Enthalpy Analytical

0318-112 ((b) (4)) AM-233 Water Activity
22nd Century Group
Analyst: (b) (6)

Summary

Client Code	Enthalpy ID	Replicate	Water Activity
(b) (4) PARE Regular King Box - 12 month 25C/60%	A	01	0.566
PARE Regular King Box - 12 month 25C/60%	A	02	0.565
PARE Regular King Box - 12 month 25C/60%	A	03	0.565
PARE Regular King Box - 12 month 25C/60%	A	04	0.568
PARE Regular King Box - 12 month 25C/60%	A	05	0.572
		Average	**0.567**
		SD	**0.003**
		%RSD	**0.5**
(b) (4) PARE Menthol King Box - 12 month 25C/60%	B	01	0.598
PARE Menthol King Box - 12 month 25C/60%	B	02	0.599
PARE Menthol King Box - 12 month 25C/60%	B	03	0.596
PARE Menthol King Box - 12 month 25C/60%	B	04	0.600
PARE Menthol King Box - 12 month 25C/60%	B	05	0.598
		Average	**0.598**
		SD	**0.001**
		%RSD	**0.2**
Laboratory Control	CRP2.1	01	0.839

APPENDIX C

OVEN VOLATILES METHOD SUMMARY AND RESULTS

ANALYSIS OF TOTAL MOISTURE IN TOBACCO
SOP No. ENT046_Rev 1.0

Tobacco tested by this procedure was ground and supplied by the client. Five gram aliquots of each sample were removed and weighed. The aliquot samples were then placed in a forced-air oven and heated at an average temperature of 99°C (±1.0°C) for three hours. The samples were allowed to cool in a desiccator and re-weighed. The percent loss in weight is reported as total moisture. Control sample aliquots used in this project were 5 grams.

The method was included on the laboratory's A2LA scope of accreditation at the time of testing and internal quality control procedures were followed. Enthalpy Analytical, LLC – Durham is accredited by the American Association for Laboratory Accreditation (Certificate Number: 3198.01).

Report for: 22nd Century Group
Client Project: Tobacco & Smoke Stability Study
Analysis Method: CDC Protocol (ENT046)

Enthalpy Project #: 0519-503
Project Start Date: 05/06/2019
Type: Cigarettes

Enthalpy Code		Client Code	% Solids	% Moisture	Balance	Weight
0519-503-A-1	(b) (4)	PARE Regular King Box - 12 month 25C/60% RH	88.3	11.7	# 19	~5g
0519-503-A-2	(b) (4)	PARE Regular King Box - 12 month 25C/60% RH	88.2	11.8	# 19	~5g
0519-503-A-3	(b) (4)	PARE Regular King Box - 12 month 25C/60% RH	88.2	11.8	# 19	~5g
0519-503-B-1	(b) (4)	PARE Menthol King Box - 12 month 25C/60% RH	87.7	12.3	# 19	~5g
0519-503-B-2	(b) (4)	PARE Menthol King Box - 12 month 25C/60% RH	87.7	12.3	# 19	~5g
0519-503-B-3	(b) (4)	PARE Menthol King Box - 12 month 25C/60% RH	87.8	12.2	# 19	~5g
CRP2.1-20190509-1		Laboratory Control Sample (CRP2.1)	48.6	51.4	# 19	~5g
CRP2.1-20190509-1		Laboratory Control Sample (CRP2.1)	48.7	51.3	# 19	~5g

APPENDIX D

NICOTINE METHOD SUMMARY AND RESULTS

GC/MS ANALYSIS OF NICOTINE AND ALKALOIDS IN TOBACCO
SOP No. ENT264_Rev 2.0

Tobacco samples are weighed and alkalinized to increase solubility. Samples are then extracted with methanol (MEOH) that has been previously spiked with internal standards. After extraction, an aliquot of the MEOH extract is removed for analysis via GC/MS for nicotine and minor alkaloids (nornicotine, myosmine, anatabine and anabasine).

The method was included on the laboratory's A2LA scope of accreditation at the time of testing and internal quality control procedures were followed. Enthalpy Analytical, LLC – Durham is accredited by the American Association for Laboratory Accreditation (Certificate Number: 3198.01).

Enthalpy Analytical

Job No.: 0519-503 line 2 ENT264: GC-MS Analysis - Tobacco

22nd Century Group, Inc.

Analyst: (b) (6)

Approximate Limit of Quantitation (mg/g)	*0.00815*
Approximate Limit of Detection (mg/g)	*0.000863*

Summary (As Is Basis)

Sample Name		Enthalpy ID	Nicotine mg/g
(b) (4)	PARE Regular King Box - 12 month 25C/60% RH	0519-503-A-4	0.448
		0519-503-A-5	0.519
		0519-503-A-6	0.519
		0519-503-A-7	0.511
		0519-503-A-8	0.628
		0519-503-A-9	0.582
		0519-503-A-10	0.537
		Average	0.535
		Std Dev	0.0572
		RSD	10.7%
(b) (4)	PARE Menthol King Box - 12 month 25C/60% RH	0519-503-B-4	0.469
		0519-503-B-5	0.527
		0519-503-B-6	0.469
		0519-503-B-7	0.479
		0519-503-B-8	0.488
		0519-503-B-9	0.483
		0519-503-B-10	0.521
		Average	0.491
		Std Dev	0.0238
		RSD	4.8%
Laboratory Control Sample (3R4F)		3R4F Ref-01 57-37	17.4

Enthalpy Analytical

Job No.: 0519-503 line 2 ENT264: GC-MS Analysis - Tobacco

22nd Century Group, Inc.

Analyst: (b) (6)

Approximate Limit of Quantitation (mg/g)	*0.00927*
Approximate Limit of Detection (mg/g)	*0.000981*

Summary (Dry Weight Basis)

Sample Name	Enthalpy ID	Nicotine mg/g	OV %
(b) (4) PARE Regular King Box - 12 month 25C/60% RH	0519-503-A-4	0.508	11.7
	0519-503-A-5	0.587	11.7
	0519-503-A-6	0.588	11.7
	0519-503-A-7	0.579	11.7
	0519-503-A-8	0.712	11.7
	0519-503-A-9	0.660	11.7
	0519-503-A-10	0.608	11.7
	Average	0.606	11.7
	Std Dev	0.0648	0.00
	RSD	10.7%	0.0%
(b) (4) PARE Menthol King Box - 12 month 25C/60% RH	0519-503-B-4	0.535	12.3
	0519-503-B-5	0.601	12.3
	0519-503-B-6	0.534	12.3
	0519-503-B-7	0.546	12.3
	0519-503-B-8	0.557	12.3
	0519-503-B-9	0.551	12.3
	0519-503-B-10	0.594	12.3
	Average	0.560	12.3
	Std Dev	0.0271	0.00
	RSD	4.8%	0.0%

APPENDIX E

CHAIN OF CUSTODY

(b) (4)

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(b) (6) 06 May 2019

CHAIN OF CUSTODY
Removal

Site: Wilson, NC

Page 25 of 27

Project Code: 0318-112 (b) (4)
Quote: (b) (4)

Client: Enthalpy Analytical, LLC | Date: 06 May 2019 | Time: 08:20AM | Form Page 1 of 2

Reason for Removal: ☒ Scheduled Time Point ☐ Non-Scheduled Request ☐ End of Study ☐ Cancellation by Client | Data Logger No.: N/A

Time Point: 12 month | Removal Documentation (Protocol with version, submitted schedule, email request, etc.): Attachment 1: Sample Submission dated 05/02/2018

Removed By: (Sign | Date) (b) (6) 06 May 2019 | Reviewed By: (Sign | Date) (b) (6) 06 May 2019

	Sample Description | PSS Sample Tracking No.	Lot | Batch No.	Chamber ID & Condition	Quantity Removed	Sample Orientation	Reviewer Initial | Date
1.	Class A Cigarettes | ENT-050318-03	0318-112 A	I | 25C/60%Rh	12	Upright	(b) (6) 06 may 19
2.	Class A Cigarettes | ENT-050318-03	0318-112 A Freeze	I | 25C/60%Rh	12	Upright	06 may 19
3.	Class A Cigarettes | ENT-050318-03	0318-112 B	I | 25C/60%Rh	12	Upright	06 may 19
4.	Class A Cigarettes | ENT-050318-03	0318-112 B Freeze	I | 25C/60%Rh	12	Upright	06 may 19
5.						
6.						
7.						
8.						
9.						
10.						
11.						
12.						
13.						
14.						
15.						

N/A (b) (6) 06 May 2019

Notes: N/A (b) (6) 06 May 2019

Relinquished By: Sign | Date: (b) (6) 06 May 2019 | Company: (b) (4)

Relinquished To: *If shipping, insert tracking number on signature line.* Sign | Date: (b) (6) 5-6-19 | Company: Enthalpy Analytical, LLC (Transporting to Richmond)

Project Code: 0318-112
Quote: (b) (4) (b) (4)

Description/Label	Condition	Testing Site	Quantity (Packs)	Ship to Address	Fedex #	Pull Date	Ship Date
0318-101 A	40/75	RVA	12	(b) (4)		3-Jun-18	29-May-18
0318-101 A DUR	40/75	DUR	3			3-Jun-18	29-May-18
0318-101 A Freeze	40/75	RVA	12			3-Jun-18	29-May-18
0318-101 B	40/75	RVA	12			3-Jun-18	29-May-18
0318-101 B DUR	40/75	DUR	3			3-Jun-18	29-May-18
0318-101 B Freeze	40/75	RVA	12			3-Jun-18	29-May-18
0318-103 A	40/75	RVA	12			3-Aug-18	30-Jul-18
0318-103 A DUR	40/75	DUR	3			3-Aug-18	30-Jul-18
0318-103 A Freeze	40/75	RVA	12			3-Aug-18	30-Jul-18
0318-103 A	25/60	RVA	12			3-Aug-18	30-Jul-18
0318-103 A DUR	25/60	DUR	3			3-Aug-18	30-Jul-18
0318-103 A Freeze	25/60	RVA	12			3-Aug-18	30-Jul-18
0318-103 B	40/75	RVA	12			3-Aug-18	30-Jul-18
0318-103 B DUR	40/75	DUR	3			3-Aug-18	30-Jul-18
0318-103 B Freeze	40/75	RVA	12			3-Aug-18	30-Jul-18
0318-103 B	25/60	RVA	12			3-Aug-18	30-Jul-18
0318-103 B DUR	25/60	DUR	3			3-Aug-18	30-Jul-18
0318-103 B Freeze	25/60	RVA	12			3-Aug-18	30-Jul-18
0318-106 A	40/75	RVA	12			3-Nov-18	29-Oct-18
0318-106 A DUR	40/75	DUR	3			3-Nov-18	29-Oct-18
0318-106 A Freeze	40/75	RVA	12			3-Nov-18	29-Oct-18
0318-106 A	25/60	RVA	12			3-Nov-18	29-Oct-18
0318-106 A DUR	25/60	DUR	3			3-Nov-18	29-Oct-18
0318-106 A Freeze	25/60	RVA	12			3-Nov-18	29-Oct-18
0318-106 B	40/75	RVA	12			3-Nov-18	29-Oct-18
0318-106 B DUR	40/75	DUR	3			3-Nov-18	29-Oct-18
0318-106 B Freeze	40/75	RVA	12			3-Nov-18	29-Oct-18
0318-106 B	25/60	RVA	12			3-Nov-18	29-Oct-18
0318-106 B DUR	25/60	DUR	3			3-Nov-18	29-Oct-18
0318-106 B Freeze	25/60	RVA	12			3-Nov-18	29-Oct-18
0318-109 A	25/60	RVA	12			3-Feb-19	29-Jan-19
0318-109 A DUR	25/60	DUR	3			3-Feb-19	29-Jan-19
0318-109 A Freeze	25/60	RVA	12			3-Feb-19	29-Jan-19
0318-109 B	25/60	RVA	12			3-Feb-19	29-Jan-19
0318-109 B DUR	25/60	DUR	3			3-Feb-19	29-Jan-19
0318-109 B Freeze	25/60	RVA	12			3-Feb-19	29-Jan-19
0318-112 A	25/60	RVA	12			3-May-19	29-Apr-19
0318-112 A DUR	25/60	DUR	3			3-May-19	29-Apr-19
0318-112 A Freeze	25/60	RVA	12			3-May-19	29-Apr-19
0318-112 B	25/60	RVA	12			3-May-19	29-Apr-19
0318-112 B DUR	25/60	DUR	3			3-May-19	29-Apr-19
0318-112 B Freeze	25/60	RVA	12			3-May-19	29-Apr-19

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ENT - 060318 - 06
ENT - 050318 - 02
ENT - 050318 - 03

(b) (6)

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