Routine Analytical Chemistry Sub-Group

Technical Report

Menthol in Mainstream Total Particulate Matter 2009 Collaborative Study

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1 SUMMARY

Due to increased interest in menthol in mainstream smoke, in 2009, the Routine Analytical Chemistry (RAC) Sub-Group organised a collaborative study to measure the amount of menthol in mainstream total particulate matter (TPM). 22 laboratories took part in the study which involved 3 commercial mentholated cigarette brands. The study objective was to minimise the contribution of production to the variability of the analytical data thereby sampling and analysis were carried out within tight timescales.

Laboratories were instructed to use their own analytical procedures.

Basic statistical analysis was performed on TPM, puff number and menthol.

The reproducibility and repeatability for menthol in mainstream TPM using the above restraints and after the exclusion of outliers was calculated to be:-

Parameter	No of Labs	Mean	Repeatability		Reproducibility	
			r	% CV r	R	% CV R
Sample A TPM	25	7.57	0.55	7.3	0.95	12.5
Sample A Menthol	25	0.22	0.03	13.6	0.06	27.3
Sample A Puff Count	25	7.3	0.4	5.5	0.7	9.6
Sample B TPM	24	10.11	0.54	5.3	1.12	11.1
Sample B Menthol	23	1.25	0.10	8.0	0.24	19.2
Sample B Puff Count	24	6.8	0.2	2.9	0.6	8.8
Sample C TPM	25	16.97	0.86	5.1	2.11	12.4
Sample C Menthol	24	0.67	0.04	6.0	0.12	17.9
Sample C Puff Count	25	7.9	0.3	3.8	0.5	3.8

Table 1: Repeatability and Reproducibility Data for all Machines (After exclusion of outliers)

2 INTRODUCTION

During 2008, the WG11 working group was established by TC126 (ISO Committee for Tobacco and Tobacco Products). This working group was tasked with designing an ISO standard for the analysis of menthol in mainstream TPM.

Some members of this working group are members of the RAC. A proposal was put forward to the Scientific Commission for the RAC to organise a collaborative study for the analysis of menthol in mainstream TPM. The Scientific Commission approved this study which was carried out in 2009.

This study involved laboratories using their individual analytical methods. In addition, three commercial mentholated samples were provided by Philip Morris International (PMI) and the contribution to analytical variability due to sampling was minimised.

Because of the work being done by the WG11 working group, it was decided that an objective for this study would not be to draft a CORESTA Recommended Method (CRM), but to get an estimate of the R & r of menthol in mainstream TPM when laboratories use their own analytical methods.

3   ORGANISATION

3.1   Participants

The full list of the 22 participating laboratories is given is APPENDIX A.

14 linear and 11 rotary smoke engines were included, with three of the laboratories providing data from both linear and rotary engines.

To preserve anonymity in this report, the participating laboratories are coded 1 to 22. The smoke engines as 'L' (linear) or 'R' (rotary).

3.2   Protocol

The protocol is to be found in APPENDIX B.

A recommendation was made that laboratories followed ISO3308 and CRMs 21 and 25 but an instruction was given that, because of the volatility of menthol, samples should not be subjected to the normal ISO conditioning prior to smoking. The packs were kept sealed until immediately before the smoking.

Laboratories were instructed to use their own analytical methods for menthol in mainstream TPM.

The samples were coded as follows and the following butt lengths were used during the study:-

Coded A Marlboro Menthol (Switzerland)   : 35 mm
Coded B Marlboro Ice Mint   : 35 mm
Coded C Marlboro Menthol (Singapore)   : 29 mm

Because some Companies have local infestation policies which involve freezing on receipt and it is unknown whether this cycle may impact on the menthol yields, it was decided that all the samples were subjected to 48 hours freezing at -18 °C on arrival at each site. The instruction was then given to remove the samples from the freezer and store in the original bundles/packaging in conditioning rooms/cabinets under ISO conditions until smoking.

The smoking and analysis were carried out during weeks 37 and 38 in 2009.

For the linear machines, one smoke run (containing 4 ports of each of the samples) was carried out on each of three days using the same smoke machine and operator. For the rotary machines, two smoke runs (20 cigarettes per run) were carried out on each of three days for each brand.

Any deviations from the protocol are listed in APPENDIX C.

4   DATA - RAW

The raw data for all samples are to be found in the tables in APPENDIX D.

The following plots give the raw data for the each of the three measured components. All the outliers are included. (Please note the red dots are the individual values and the blue circles the mean values for each laboratory)

4.1   TPM (mg/cig)

Individual Value Plot of SPLE A TPM

Individual Value Plot of SPLE B TPM

Individual Value Plot of SPLE C TPM

4

4.2   Menthol (mg/cig)

Individual Value Plot of SPLE A MENTHOL

Individual Value Plot of SPLE B MENTHOL

Individual Value Plot of SPLE C MENTHOL

4.3 Puff Count

Individual Value Plot of SPLE A PUFF COUNT

Individual Value Plot of SPLE B PUFF COUNT
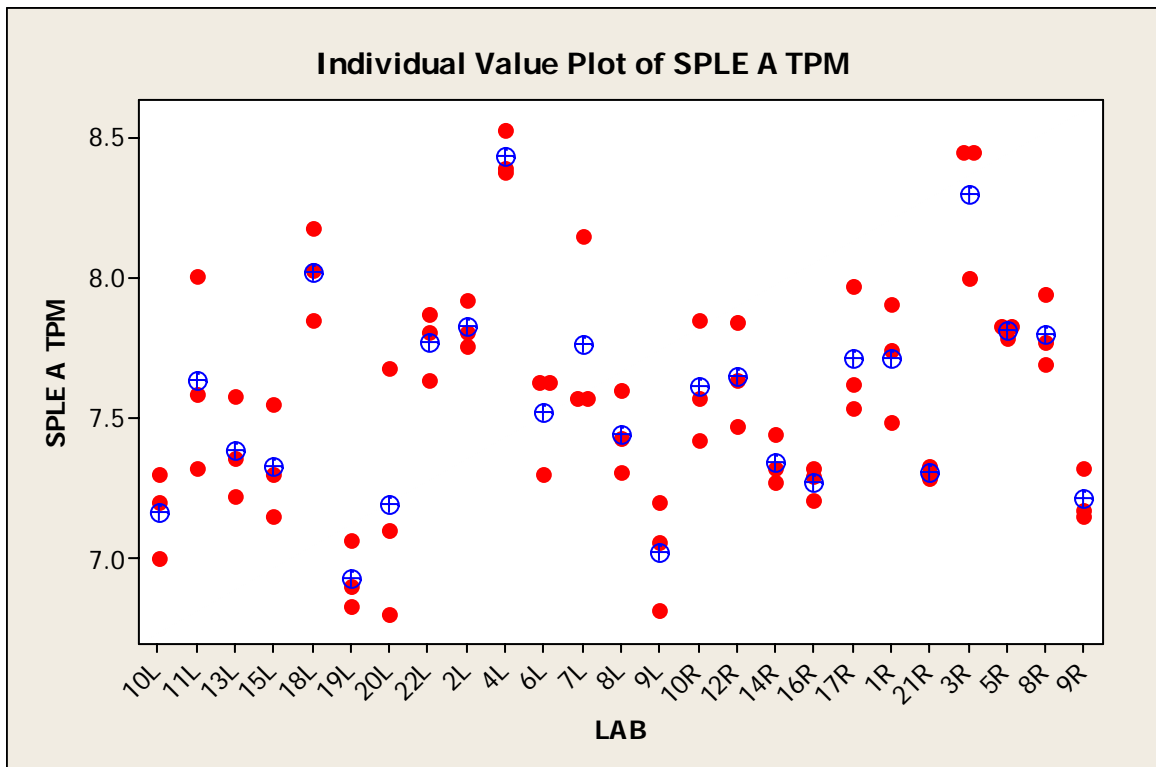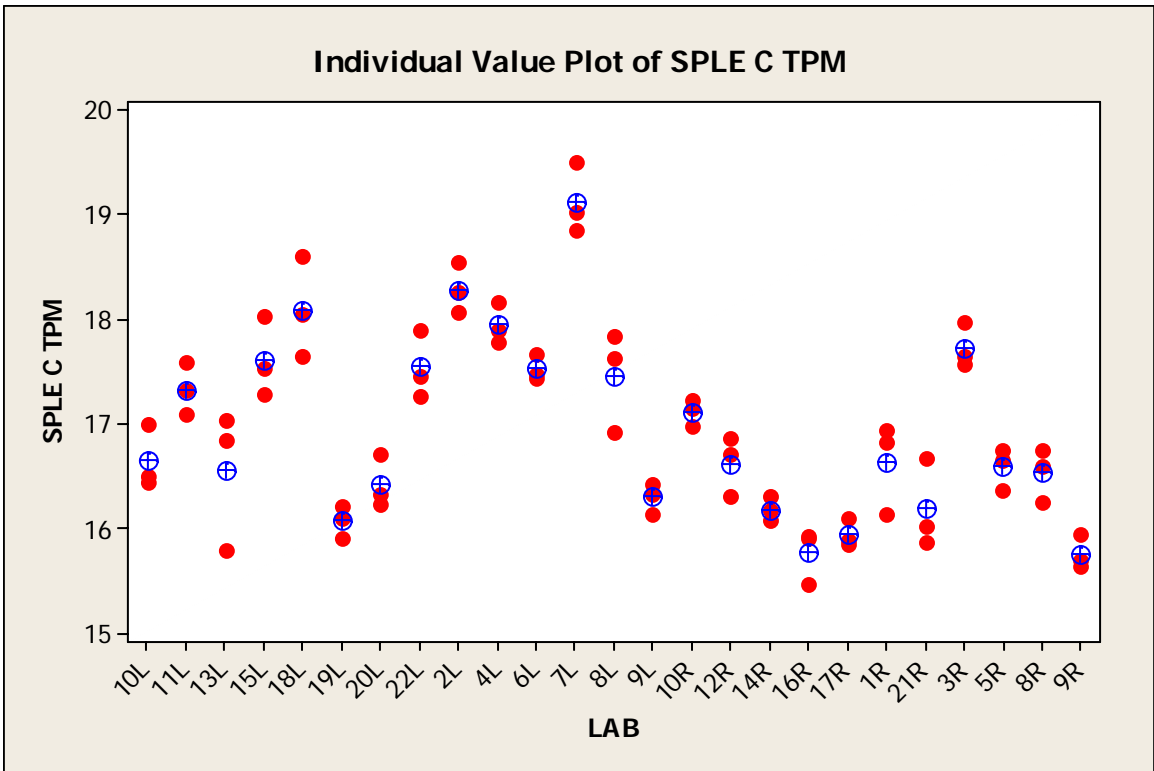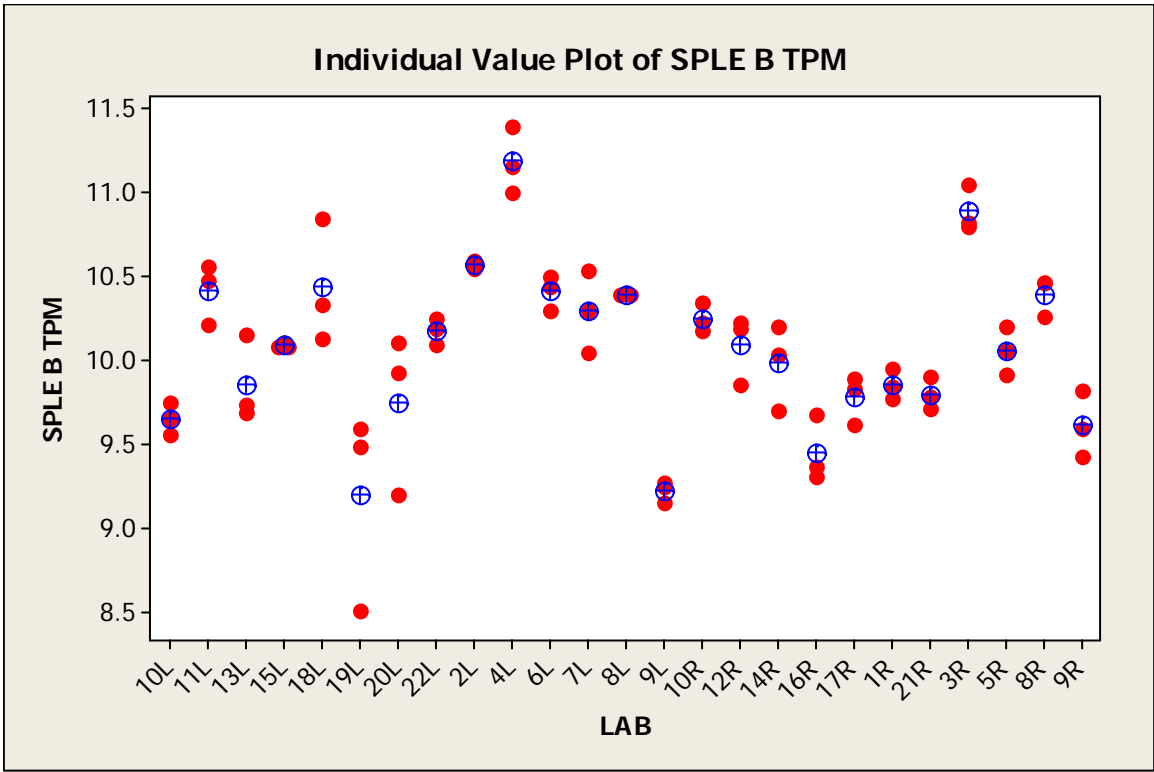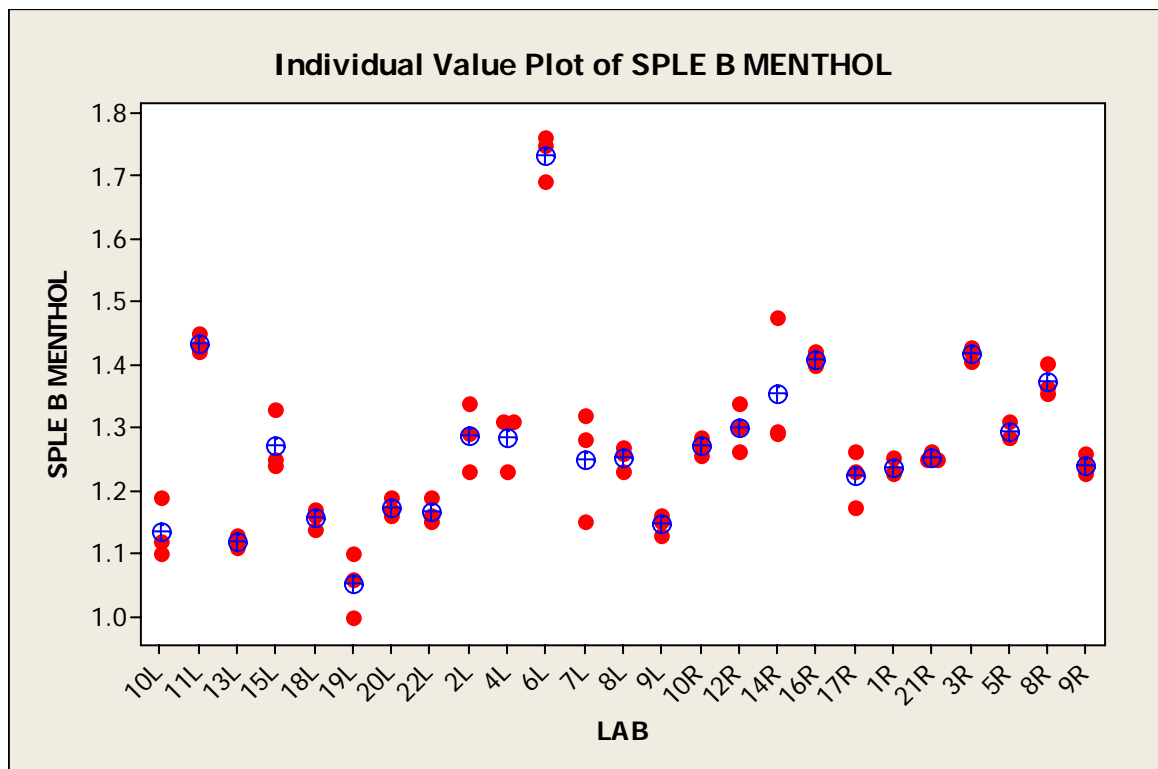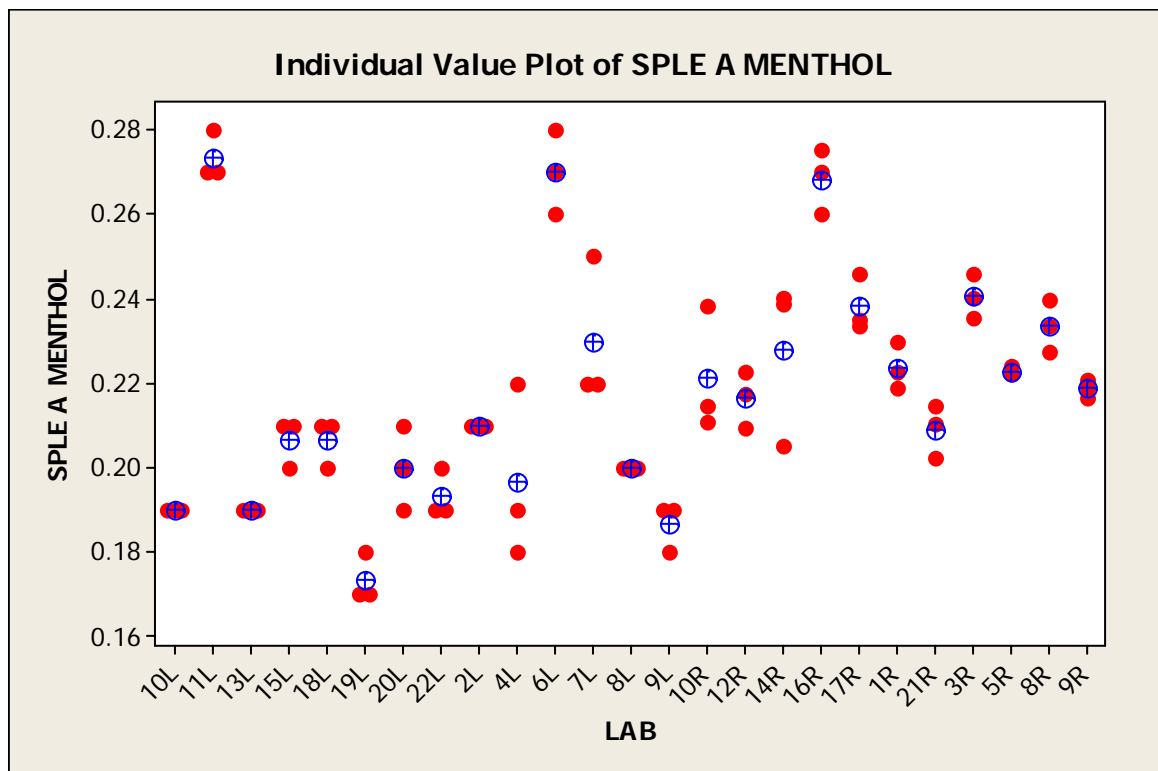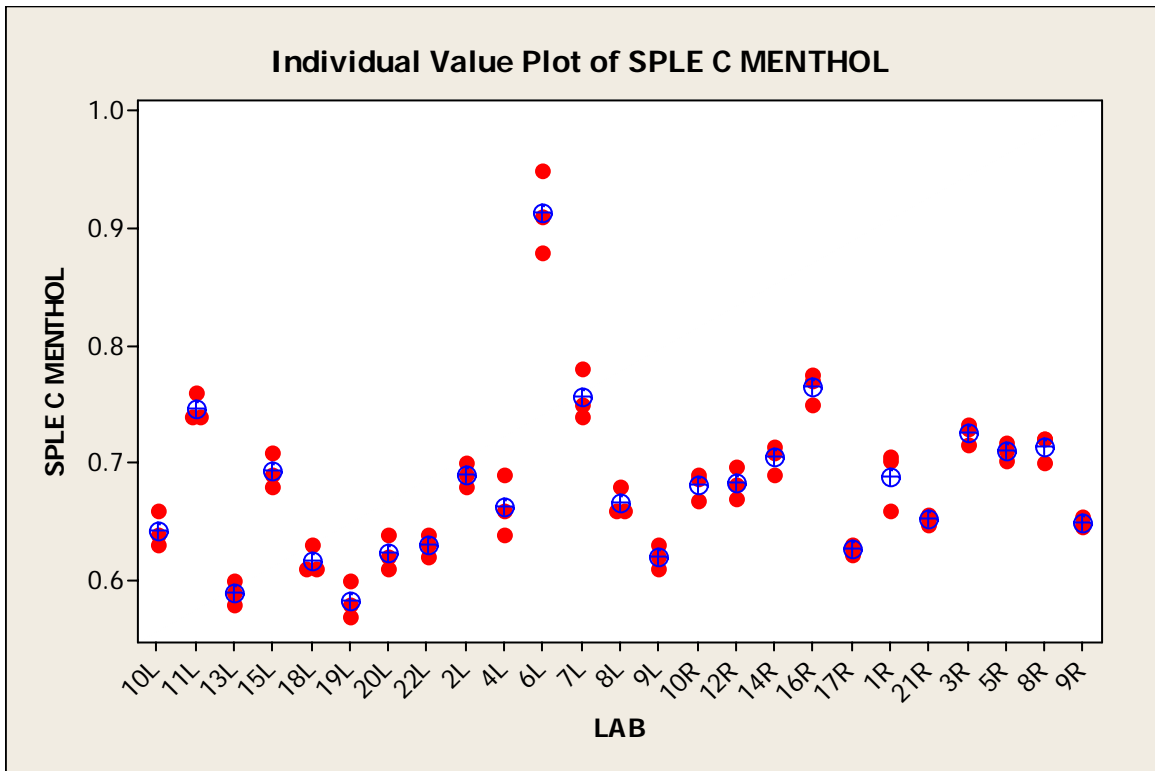
Individual Value Plot of SPLE C PUFF COUNT

5   DATA - STATISTICAL ANALYSIS

5.1   MANDEL'S k and h

Initially the raw data was checked for the presence of outliers (0.99 level) using two graphical data consistency techniques (Mandel's k and h). For convenience in data interpretation the derived k and h values are displayed in APPENDIX E as their corresponding standard deviations (k plots) or mean values (h plots) for each laboratory.

As the graphical data consistency techniques are more likely to indicate outliers than numerical techniques, no further action was taken on excluding data at this stage. However, the graphs in APPENDIX E confirm the presence of outliers as calculated in the next section.

5.2   Exclusion of Outliers

Outlier testing was carried out according to ISO 5725-part 2 using Cochran's test to eliminate within laboratory variance followed by Grubbs' test to eliminate between laboratory outliers.

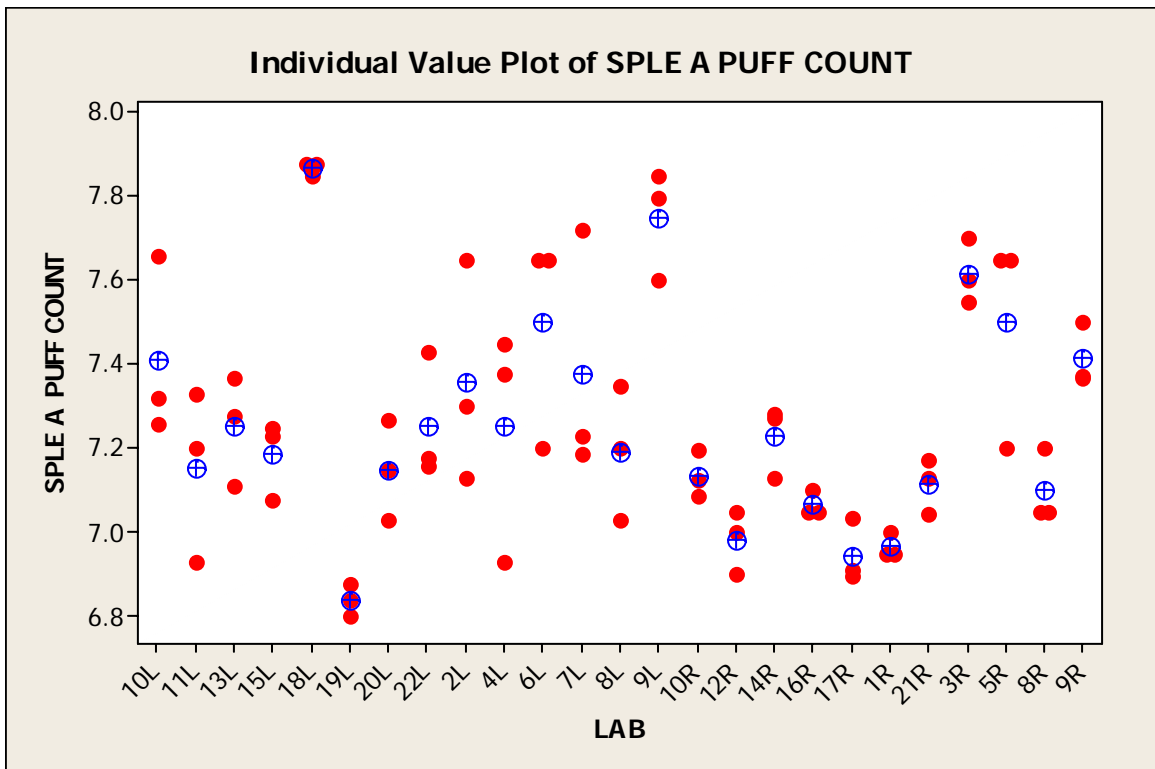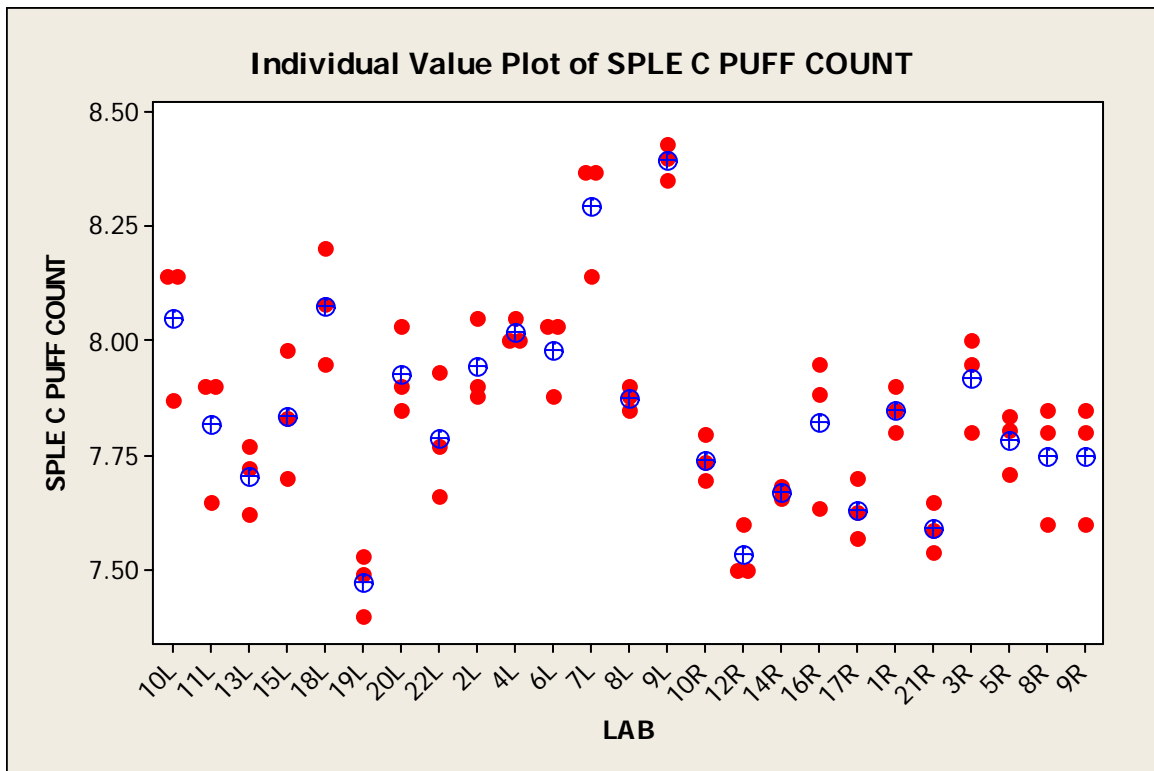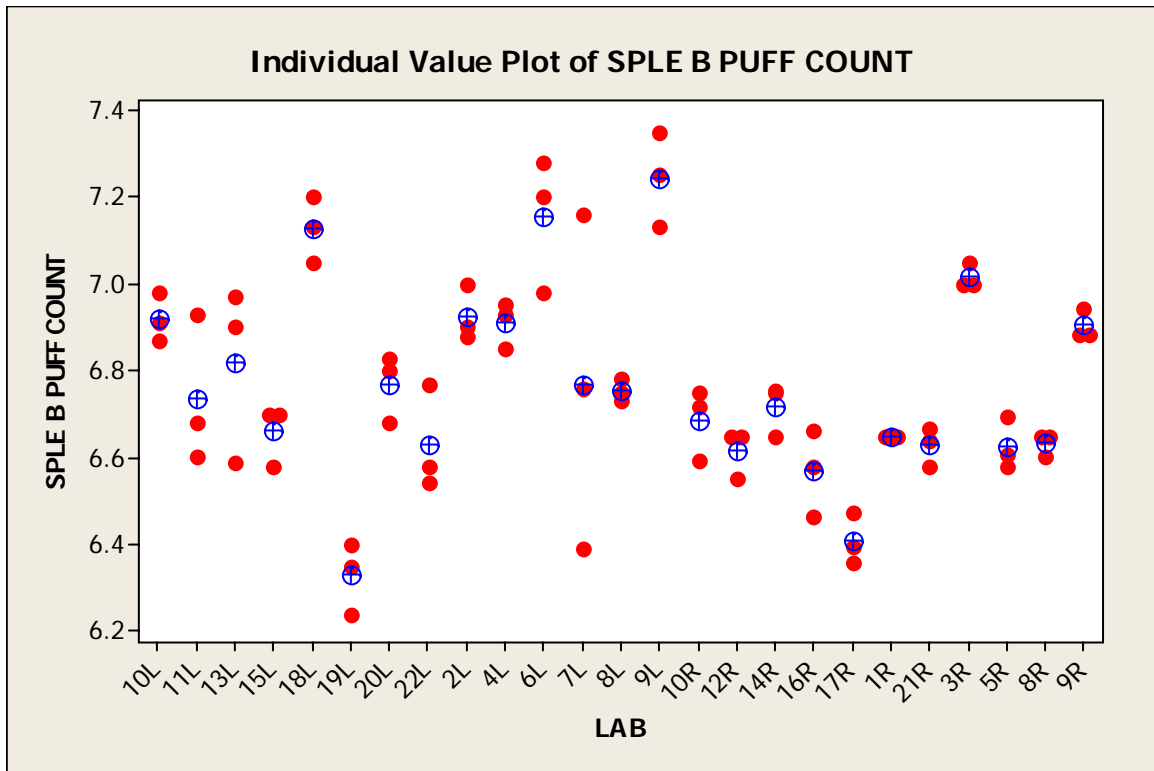| Analyte	Cochran's Outliers	Grubbs' Outliers
Sample A TPM	None	None
Sample A Menthol	None	None
Sample A Puff Count	None	None
Sample B TPM	19L	None
Sample B Menthol	14R	6L
Sample B Puff Count	7L	None
Sample C TPM	None	None
Sample C Menthol	None	6L
Sample C Puff Count	None	None

Table 2: List of Outlying Laboratories

5.3   Calculation of R & r – All Machines

The following table lists repeatability (r) & reproducibility (R) values which were calculated on the remaining data after the exclusion of outliers

Parameter	No of Labs	Mean	Repeatability		Reproducibility	
			r	% CV r	R	% CV R
Sample A TPM	25	7.57	0.55	7.3	0.95	12.5
Sample A Menthol	25	0.22	0.03	13.6	0.06	27.3
Sample A Puff Count	25	7.3	0.4	5.5	0.7	9.6
Sample B TPM	24	10.11	0.54	5.3	1.12	11.1
Sample B Menthol	23	1.25	0.10	8.0	0.24	19.2
Sample B Puff Count	24	6.8	0.2	2.9	0.6	8.8
Sample C TPM	25	16.97	0.86	5.1	2.11	12.4
Sample C Menthol	24	0.67	0.04	6.0	0.12	17.9
Sample C Puff Count	25	7.9	0.3	3.8	0.5	3.8

Table 3: R & r Values for each Parameter – All Machines

5.4 Calculation of R & r – Linear v Rotary Machines

The following table lists repeatability (r) & reproducibility (R) values which were calculated on the remaining data after the exclusion of outliers.

Parameter Machine Type	No of Labs	Mean	Repeatability		Reproducibility	
			r	% CV r	R	% CV R
Sample A TPM Linear	14	7.53	0.62	8.2	1.07	14.2
Sample A TPM Rotary	11	7.61	0.45	5.9	0.81	10.6
Sample A Menthol Linear	14	0.21	0.03	14.3	0.07	33.3
Sample A Menthol Rotary	11	0.23	0.03	13.0	0.04	17.4
Sample A Puff Count Linear	14	7.3	0.53	7.3	0.73	10.0
Sample A Puff Count Rotary	10	7.2	0.19	2.6	0.51	7.1
Sample B TPM Linear	13	10.19	0.60	5.9	1.23	12.1
Sample B TPM Rotary	11	10.01	0.45	4.5	0.98	9.8
Sample B Menthol Linear	13	1.21	0.12	9.9	0.24	19.8
Sample B Menthol Rotary	10	1.30	0.06	4.6	0.17	13.1
Sample B Puff Count Linear	13	6.8	0.31	4.6	0.61	9.0
Sample B Puff Count Rotary	11	6.7	0.16	2.4	0.39	5.8
Sample C TPM Linear	14	17.36	0.96	5.5	2.13	12.3
Sample C TPM Rotary	11	16.97	0.72	4.2	2.08	12.3
Sample C Menthol Linear	13	0.66	0.04	6.1	0.13	19.7
Sample C Menthol Rotary	11	0.69	0.03	4.3	0.09	13.0
Sample C Puff Count Linear	14	7.9	0.29	3.7	0.58	7.3
Sample C Puff Count Rotary	11	7.8	0.24	3.1	0.42	5.4

Table 4: R & r Values for each Parameter per Machine Type.

6   DATA INTERPRETATION

6.1   Comparison to other known studies.

PMI regularly conduct internal collaborative studies for menthol in TPM amongst their laboratories. A significant difference between these studies and this one is that in the PMI laboratories use the same analytical method and in this study laboratories used their own analytical method.

However, PMI have provided typical menthol %CV r and %CV R estimates for their studies as approximately 8% and 20% respectively. The values for menthol from this study agree very well with these values for samples B and C (the medium and higher menthol yielding samples). The lower yielding sample A has higher %CV r (13.6) and %CV R (27.3) but this could be accounted for as the yield of this sample is near the detection limit of the analysis.

6.2   Comparison of Linear V Rotary Machines.

The mean data for menthol in Table 4 indicates that there may be a bias of menthol yield between linear and rotary machines. The data is compared in the plot below.

Menthol Yield - Linear v Rotary Machines
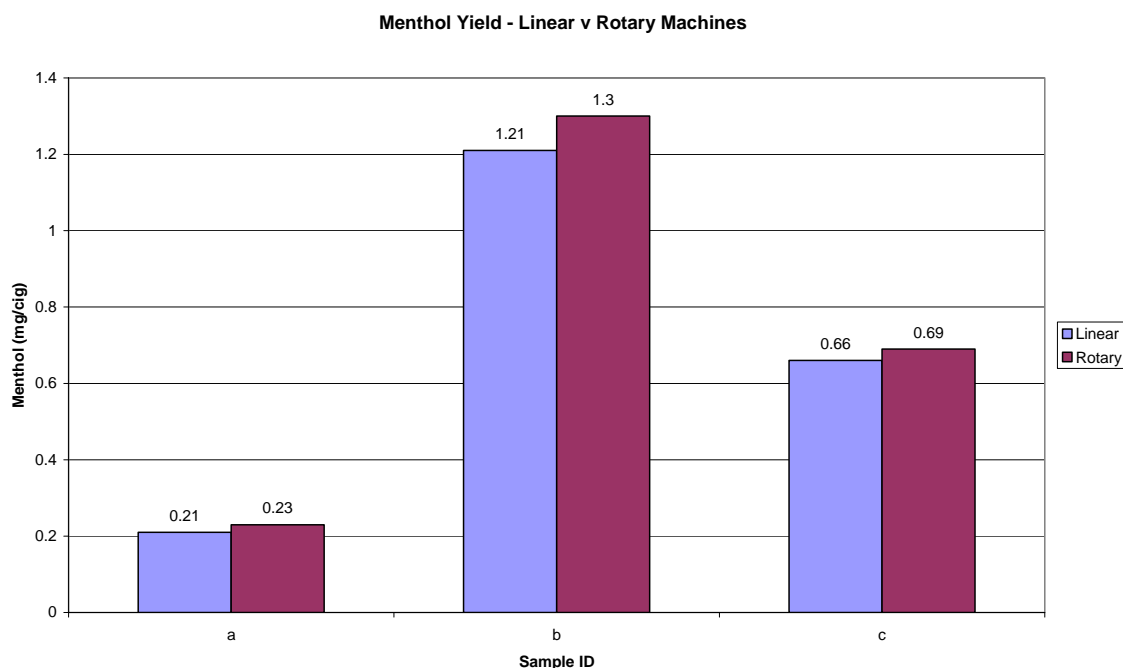
One way analysis of variance (ANOVA) was conducted on the laboratories' mean menthol data. At a 95% level of confidence, data from Sample B is the only set that indicates that there may be a slightly significant difference in menthol yields between rotary and linear smoking machines. However, when this difference is put into context with laboratory repeatability and reproducibility it is not practically significant.

7   RECOMMENDATIONS

When the data from the ISO TC126 WG11 study becomes available it would be useful to compare it with the data from this report.

APPENDIX A

List of Participating Laboratories

Arista Laboratories Europe
Arista Laboratories U.S.
British American Tobacco, Germany
British American Tobacco, Denmark
British American Tobacco, Poland
British American Tobacco, South Africa
British American Tobacco, UK
China National Tobacco Centre
Filtrona Technology Centre
Heintz van Landewyck
ITC, India
Japan Tobacco Inc.
JTI, Germany GmbH
JTI, UK
KT&G Central Research Institute
Labstat International ULC
Lorillard Tobacco Co
Philip Morris International OPS
Philip Morris International R&D
R.J. Reynolds Tobacco Company
Imperial Tobacco France
Papierfabrik Wattens

APPENDIX B

Experimental Protocol

**CORESTA Routine Analytical Chemistry Sub-group
Menthol in Mainstream Smoke – 2009 Collaborative Study**

1. Introduction

The "Routine Analytical Chemistry Sub Group" has been given the responsibility to organize a collaborative study for menthol in mainstream smoke. Because menthol is prone to sublimation under normal laboratory operating conditions, sample handling restrictions and analysis timings have been specified.

2. Objective

The aim of this study is to give an indication of reproducibility and repeatability (R & r) values for menthol in mainstream smoke when laboratories use their own methods of analysis and the contribution due to production variability is minimised.

3. Methods

The following relevant ISO/CORESTA Recommended Methods will be used:-

Smoking machines   ISO 3308   CORESTA Recommended Methods No. 21 and 25

NOTE: Due to the volatility of menthol, samples must not be subjected to ISO conditioning prior to smoking.

The following pre-determined butt lengths should be used for each of the samples:-

```
Coded A     Marlboro Menthol (Switzerland): 35 mm
Coded B     Marlboro Ice Mint          : 35 mm
Coded C     Marlboro Menthol (Singapore) : 29 mm
```

Laboratories will use their own method for the analysis of menthol in mainstream smoke

To avoid possible influence on the smoking results please remind your operators that the side seam of the cigarettes should be positioned at random and not in a fixed position (never always up or down as some operators may normally do).

4. Samples

The study will use the three commercial brands listed below:-

```
Coded A     Marlboro Menthol (Switzerland)
Coded B     Marlboro Ice Mint
Coded C     Marlboro Menthol (Singapore)
```

The samples were kindly procured and despatched by PMI during the week commencing August 17th. Each participating laboratory will receive a sealed 200 'bundle' or 'outer' of each product.

5. Sample Receipt

Samples arriving at some Companies are subjected to 48 hours freezing at –18 °C to comply with local infestation policies. Because this freezing cycle MAY impact on the menthol yield of the samples, **ALL laboratories should freeze the samples for 48 hours at –18 °C as soon after receipt as possible. They should then be removed from the freezer and <u>stored in the original bundles/packaging</u> in conditioning rooms/cabinets under ISO conditions until the time they will be prepared for smoking.**

It is most important that the bundles/outers are kept sealed until immediately before the smoking starts.

6. Smoking timetable

It is very important that the smoking takes place during the same two week time period. This period is to be during weeks 37 & 38 (7th to 19th September).

7. Smoking Plans - Overview

NOTE An important physical characteristic of menthol is that it is prone to sublimation therefore exposure of the cigarettes to the atmosphere during sampling should be minimised.

The smoking plans are shown in figures 1 and 2.

Please note that puff numbers and TPM (mg/cig) data are required for each sample.

For each brand, six runs (pads) of smoking 20 cigarettes will be required when a <u>rotary</u> 20 port machine is used. These will be smoked over a 3 day period at 2 runs per day for each sample.

For each brand, 12 ports of smoking 5 cigarettes per port will be required when a <u>linear</u> machine is used. These will be smoked over a 3 day period.

The smoking plans will therefore use 60 cigarettes for each linear smoking machine and 120 cigarettes for each rotary smoking machine.

8. Smoking Plan for Rotary Machines

2 smoke runs to be carried out on each of 3 days for each brand.

For each brand in turn:-

Remove the sealed 200 bundle from the conditioning room immediately prior to smoking and select 2 packs of 20 from the bundle for smoking on day 1. Return the unopened packs to the conditioning room.

To minimise the exposure of the cigarettes to the atmosphere during smoking assign 1 unopened pack of 20 to each of the 2 daily runs (pads) to be smoked per sample.

Remove the outer film from each pack <u>immediately before smoking</u>. Load all the 20 cigarettes from the one pack onto the rotary machine immediately prior to smoking. When smoking is complete extract the pad in the appropriate solvent and analyse for menthol according to your laboratory's routine method. Repeat for the second sample of the first brand.

This procedure should be followed for the other 2 brands.

9. Smoking Plans for Linear Machines

To minimise the exposure of the cigarettes to the atmosphere during smoking it is advisable to assign 1 unopened pack of 20 to each of the 4 ports to be smoked and remove the outer films from each pack immediately before smoking. Each cigarette should be removed from its pack immediately prior to smoking, butt marked then inserted into the port for smoking. The pack lids should be closed between sampling.

Ports (on the linear smoking machines) not occupied are shown as X, which could be usefully employed with the laboratory's internal monitor samples.

Codes for the plans below:-

```
Coded A      Marlboro Menthol (Switzerland)
Coded B      Marlboro Ice Mint
Coded C      Marlboro Menthol (Singapore)
```

Figure 1 Smoking Plan for Linear 20-Port Machines

Run No.	Port number																			
	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20
1	A	A	A	A	X	X	X	X	B	B	B	B	X	X	X	X	C	C	C	C
2	B	B	B	B	X	X	X	X	A	A	A	A	C	C	C	C	X	X	X	X
3	X	X	X	X	C	C	C	C	X	X	X	X	B	B	B	B	A	A	A	A

1 smoke run to be carried out on each of 3 days where possible using the same smoke engine and same operator.

Figure 2 Smoking Plan for Linear 16-Port Machines

Run No.	Port number															
	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16
1	A	A	A	A	x	x	x	x	B	B	B	B	C	C	C	C
2	B	B	B	B	A	A	A	A	C	C	C	C	x	x	x	x
3	x	x	x	x	C	C	C	C	B	B	B	B	A	A	A	A

1 smoke run to be carried out on each of 3 days where possible using the same smoke engine and same operator.

10. Target variables

The variables to be analysed are menthol yield in mainstream TPM (mg/cig). Ancillary measurements will be puff number and total particulate matter (TPM).

11. Variables to be reported

The variables to be reported fall into two categories; those ancillary to the test, and those which will form the basis of the statistical evaluation of the data.

The ancillary variables are:
- Type of smoking machine used
- Laboratory temperature during smoking
- Relative humidity (RH%) in the laboratory during smoking
- Atmospheric pressure in the laboratory during smoking
- Date of test
- Ambient air flow

The variables to be reported for statistical analysis are:

- Puff number per pad
- TPM per pad
- Menthol per pad

12. Dimensions and rounding of test results

Ancillary variables:

- Laboratory temperature	degrees Celsius.	##.#
- Laboratory humidity	percent RH.	##.#
- Laboratory pressure	kPa.	###.#

Analytical variables:

- Puff number	number	##.#
- TPM	mg per sample	##.##
- Menthol	mg per sample	##.###

Please note that the rounding of the data to the formats specified above must take place after any calculations that may be involved. All calculations will use the laboratory data as recorded using the maximum number of digits available.

All data must be submitted using the spreadsheets that will be circulated by the Study Coordinator.

13. Data Submission

Data should be sent to the study coordinator, Linda Drake, (Linda_Drake@bat.com) not later than Tuesday 22nd September to allow sufficient time for the basic statistics to be prepared in time for the next meeting of the Routine Analytical Chemistry sub-group meeting on October 6th 2009.

14. Exchange of data

All data arising from this study will be made available to the laboratories participating in the study. The distribution of data will be done after coding, collation and statistical evaluation.

To facilitate the statistical evaluation, all results must be reported according to the specifications given in the reporting spreadsheet's "Reporting format" and the data must be exchanged by electronic means i.e. by e-mail. **Please use the recommended reporting format of the spreadsheet.**

The statistical analysis of the results will follow as closely as possible the recommendations of ISO 5725 part 2.

Should any questions arise please contact the Study Coordinator.

All results must be sent to:

Linda Drake (e-mail Linda_Drake@bat.com)

APPENDIX C

Departures from Experimental Protocol

Lab number	Smoke Machine	Chromatography Column (diameter/length/packing or coating)	Internal standard used	Number of standards (levels)	Range of standards' concentrations (mg/ml)	Extraction volume in mls	Extraction method	Extraction timing
1	RM20 mod	15m×0.53mm i.d., DB-WAX, 1μm thickness	1,3-butanediol	5	0.002004 to 0.6009 (mg/ml)	50	Shaker	20 minutes
2	SM350	2m x 1.8" x 2.0mm nickel column packed with 10% Carbowax 20M + 2% KOH on Chromosorb WHP 80-100 Mesh	Heptadecane	6	0.0396 to 2.3737 (mg/ml)	20	Shaker	25 minutes
3	RM20CSR	DB-Wax (30m x 0.53mm x 1μm)	n-heptadecane	8	0.05 to 0.75 (mg/ml)	50	Shaker	20 minutes
4	KC20X	5% CW20M 100/120 Supelcoport Column 2M*1/8in Nickel	Heptadecane	4	0.05 to 0.20 (mg/ml)	20	Shaker	20 minutes
5	RM200	Innowax 15m ×0,53 mm x 1	Decanol	5	0.10 to 0.75 (mg/ml)	50	Shaker	45 minutes
6	SM450	DB-Waxetr （30m×0.25mm×0.25um)	Heptadecane	6	0.027 to 0.489 (mg/ml)	20	Shaker	90 minutes
7	ASM500	0.25mm x 15 m x 0.25μm Rtx-5MS	n-tetradecane	5	0.025 to 0.5 (mg/ml)	40	Shaker	30 minutes
8	RM200 SM450	Megabore , CP-wax 52 CB; 0,53 MM ID	n-heptadecane	6	0.24 to 3.00 (mg/cig)	20	Shaker	20 minutes
9	RM200 SM450	DB - WAX (530 um x 30 metres ×1.00 um)	n-heptadecane	9	0.02 to 0.80 (mg/ml)	Linear = 25 ml Rotary = 50 ml	Shaker	60 minutes
10	SM450 RM20CSR	HP Carbowax 20 M	n-heptadecane	7	0.00796 to 0.49720 (mg/ml)	Linear = 10 ml Rotary = 50 ml	Shaker	30 minutes
11	SM450	10m x 0.1mm x 0.1um Wax SGE	Trans-anethole	3	0.05 to 0.15 (mg/ml)	20	Shaker	30 minutes
12	RM20H	DB-WAX (0.32mm,30m,0.25μm)	Anethol	4	0.05 to 0.30 (mg/ml)	100	Stand overnight	16 hours
13	SM450	DB-Wax Fused Silica 0.53 mm/15m/1um	Trans-anethole	3	0.0103 to 0.2587 (mg/ml)	20	Shaker	45 minutes
14	RM200	Column DB-wax, length 30m, thickness 1μm, internal diameter 0.53 mm	n-heptadecane	5	0.04 to 0.80 (mg/ml)	50	Shaker	60 minutes
15	SM400	CARBOWAX 30m x 0.32mm x 0.5μm	Octadecane	5	0.25 to 4 (mg/ml)	10	maceration/soaking (overnight) and followed by bath ultrasound (10 minutes)	16 hours then 10 minutes
16	RM20	HP-5MS (Crosslinked 5%PHME Siloxane) 30m x 0,25mm x 0,25μm Film Thickness	Heptadecane	1	0.2000 [mg/ml]	100	Shaker	30 minutes
17	RM20	Capillary HP-INNOWAX (Polyethylene Glycol 0.25um/320um/30m)	n-heptadecane	4	0.20 to 0.80 mg/ml	100	Shaker	30 minutes
18	SM450	J&W DB WaxETR/5mx0.2IDx0.4um	1,3-butanediol	5	0.119 to 4.95 (mg/ml)	10	Shaker	60 minutes
19	SM450	Restek Rtx-5 (15m, 0.25mmID, 0.25um df) Crossbond 5% diphenyl/ 95%	Anethole	8	0.01033 to 3.22934 (mg/ml)	20	Shaker	35 minutes
20	SM450	ZB Wax, 3m x 0.18mm i.d., and 0.18μ film thickness	n-heptadecane	4	0.075 to 1.875 (mg/ml)	20	Shaker	20 minutes
21	RM200A	Column DB-WAX, 30 m long, 1 micron film thickness, ID= 0.53 mm	n-heptadecane	5	0.04 to 0.80 (mg/ml)	50	Shaker	60 minutes
22	SM400	Chromosorb Custom 6' x 1/8" 7% Carbowax 20M, 3% OS-138 2% KOH	n-heptadecane	7	0.020 to 1.611 (mg/ml)	20	Shaker	30 minutes

APPENDIX D

Full Data Set - Sample A

Lab No.	Sample A TPM			Sample A Menthol			Sample A Puff Count		
	Rep 1	Rep 2	Rep 3	Rep 1	Rep 2	Rep 3	Rep 1	Rep 2	Rep 3
10L	7.20	7.30	7.00	0.19	0.19	0.19	7.7	7.3	7.3
11L	8.01	7.59	7.32	0.28	0.27	0.27	7.3	7.2	6.9
13L	7.58	7.22	7.36	0.19	0.19	0.19	7.4	7.3	7.1
15L	7.15	7.55	7.30	0.21	0.20	0.21	7.2	7.1	7.3
18L	8.18	7.85	8.03	0.21	0.21	0.20	7.9	7.9	7.9
19L	6.83	6.90	7.07	0.18	0.17	0.17	6.8	6.9	6.8
20L	6.80	7.10	7.68	0.19	0.20	0.21	7.0	7.2	7.3
22L	7.81	7.64	7.87	0.19	0.20	0.19	7.2	7.2	7.4
2L	7.76	7.81	7.92	0.21	0.21	0.21	7.7	7.3	7.1
4L	8.53	8.38	8.39	0.22	0.19	0.18	7.5	7.4	6.9
6L	7.63	7.63	7.30	0.27	0.28	0.26	7.7	7.2	7.7
7L	8.15	7.57	7.57	0.25	0.22	0.22	7.7	7.2	7.2
8L	7.60	7.43	7.31	0.20	0.20	0.20	7.4	7.2	7.0
9L	7.20	6.82	7.06	0.19	0.18	0.19	7.8	7.6	7.9
10R	7.58	7.85	7.43	0.24	0.21	0.21	7.2	7.1	7.1
12R	7.84	7.48	7.64	0.21	0.22	0.22	6.9	7.0	7.1
14R	7.33	7.45	7.27	0.24	0.24	0.21	7.1	7.3	7.3
16R	7.21	7.32	7.30	0.26	0.28	0.27	7.1	7.1	7.1
17R	7.63	7.54	7.97	0.24	0.23	0.25	6.9	6.9	7.0
1R	7.49	7.75	7.91	0.22	0.22	0.23	7.0	7.0	7.0
21R	7.33	7.29	7.31	0.21	0.21	0.20	7.1	7.0	7.2
3R	8.45	8.45	8.00	0.24	0.25	0.24	7.6	7.7	7.6
5R	7.79	7.83	7.83	0.22	0.22	0.22	7.7	7.2	7.7
8R	7.77	7.95	7.69	0.23	0.24	0.23	7.2	7.1	7.1
9R	7.32	7.17	7.15	0.22	0.22	0.22	7.5	7.4	7.4

Full Data Set - Sample B

Lab No.	Sample B TPM			Sample B Menthol			Sample B Puff Count		
	Rep 1	Rep 2	Rep 3	Rep 1	Rep 2	Rep 3	Rep 1	Rep 2	Rep 3
10L	9.65	9.75	9.55	1.12	1.10	1.19	6.9	7.0	6.9
11L	10.21	10.56	10.47	1.45	1.42	1.43	6.9	6.7	6.6
13L	10.15	9.74	9.69	1.13	1.12	1.11	6.9	7.0	6.6
15L	10.11	10.08	10.08	1.24	1.25	1.33	6.7	6.7	6.6
18L	10.33	10.13	10.85	1.14	1.17	1.16	7.1	7.1	7.2
19L	8.50	9.59	9.49	1.00	1.10	1.06	6.2	6.4	6.4
20L	9.20	9.93	10.10	1.16	1.17	1.19	6.8	6.7	6.8
22L	10.09	10.19	10.25	1.15	1.16	1.19	6.6	6.8	6.5
2L	10.56	10.55	10.60	1.23	1.34	1.29	7.0	6.9	6.9
4L	11.00	11.40	11.16	1.31	1.31	1.23	6.9	7.0	6.9
6L	10.50	10.30	10.44	1.76	1.69	1.75	7.2	7.3	7.0
7L	10.54	10.30	10.05	1.32	1.28	1.15	7.2	6.8	6.4
8L	10.40	10.39	10.39	1.26	1.23	1.27	6.7	6.8	6.8
9L	9.27	9.15	9.24	1.15	1.13	1.16	7.4	7.3	7.1
10R	10.35	10.23	10.18	1.29	1.27	1.26	6.7	6.8	6.6
12R	10.19	10.23	9.86	1.30	1.34	1.26	6.7	6.7	6.6
14R	10.03	10.21	9.71	1.48	1.29	1.30	6.8	6.8	6.7
16R	9.37	9.67	9.31	1.41	1.42	1.40	6.5	6.7	6.6
17R	9.89	9.83	9.62	1.26	1.23	1.17	6.5	6.4	6.4
1R	9.85	9.77	9.95	1.23	1.23	1.25	6.7	6.7	6.7
21R	9.78	9.90	9.71	1.25	1.25	1.26	6.7	6.6	6.6
3R	11.05	10.80	10.83	1.43	1.40	1.42	7.1	7.0	7.0
5R	10.06	9.92	10.20	1.31	1.28	1.28	6.6	6.6	6.7
8R	10.26	10.47	10.46	1.40	1.36	1.36	6.7	6.6	6.7
9R	9.82	9.60	9.43	1.26	1.24	1.23	6.9	6.9	6.9

Full Data Set - Sample C

| Lab No.	Sample C TPM			Sample C Menthol			Sample C Puff Count		
	Rep 1	Rep 2	Rep 3	Rep 1	Rep 2	Rep 3	Rep 1	Rep 2	Rep 3
10L	17.00	16.45	16.50	0.64	0.63	0.66	8.1	8.1	7.9
11L	17.09	17.33	17.59	0.76	0.74	0.74	7.9	7.7	7.9
13L	17.05	15.80	16.85	0.59	0.58	0.60	7.7	7.8	7.6
15L	18.04	17.53	17.29	0.71	0.69	0.68	8.0	7.8	7.7
18L	18.60	17.65	18.05	0.63	0.61	0.61	8.2	8.1	8.0
19L	16.22	15.92	16.11	0.60	0.57	0.58	7.5	7.4	7.5
20L	16.24	16.33	16.72	0.61	0.62	0.64	8.0	7.9	7.9
22L	17.47	17.28	17.90	0.64	0.62	0.63	7.8	7.7	7.9
2L	18.55	18.07	18.26	0.69	0.68	0.70	8.1	7.9	7.9
4L	17.78	17.91	18.16	0.69	0.64	0.66	8.0	8.0	8.1
6L	17.49	17.45	17.68	0.95	0.88	0.91	8.0	7.9	8.0
7L	19.03	19.51	18.85	0.75	0.78	0.74	8.4	8.4	8.1
8L	17.84	17.64	16.93	0.68	0.66	0.66	7.9	7.9	7.9
9L	16.43	16.15	16.34	0.63	0.61	0.62	8.4	8.4	8.4
10R	16.98	17.23	17.15	0.67	0.69	0.69	7.7	7.8	7.7
12R	16.72	16.86	16.31	0.68	0.70	0.67	7.5	7.6	7.5
14R	16.08	16.32	16.18	0.72	0.71	0.69	7.7	7.7	7.7
16R	15.47	15.93	15.91	0.75	0.77	0.78	7.6	7.9	8.0
17R	15.86	16.10	15.89	0.62	0.63	0.63	7.7	7.6	7.6
1R	16.14	16.84	16.95	0.66	0.70	0.71	7.9	7.9	7.8
21R	16.03	16.68	15.88	0.65	0.65	0.66	7.5	7.6	7.7
3R	17.58	17.98	17.65	0.73	0.73	0.72	8.0	8.0	7.8
5R	16.38	16.66	16.75	0.70	0.71	0.72	7.8	7.7	7.8
8R	16.75	16.27	16.60	0.72	0.70	0.72	7.9	7.6	7.8
9R	15.95	15.71	15.65	0.65	0.65	0.65	8.1	8.1	8.1

APPENDIX E

MANDEL'S STATISTICS

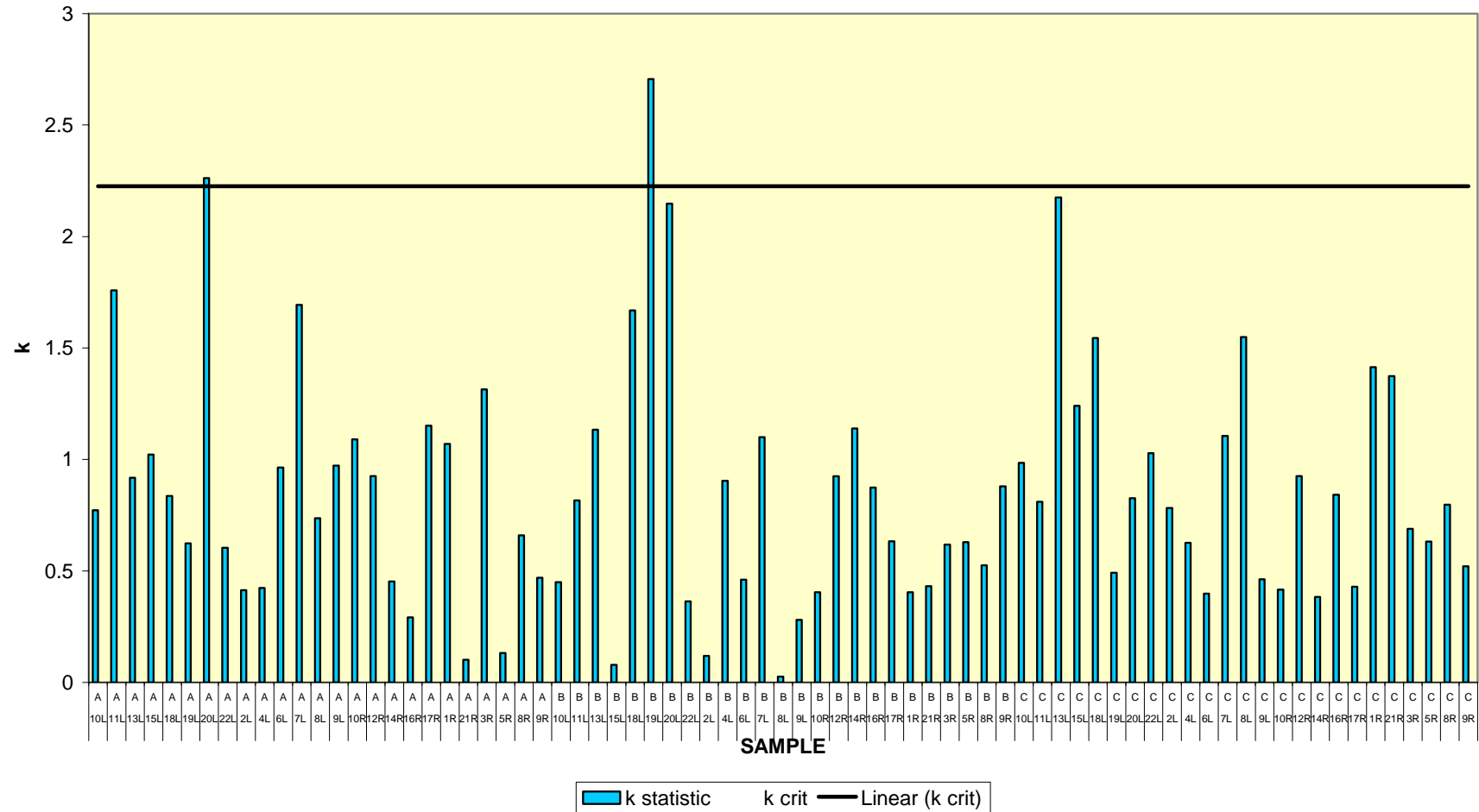**MANDEL'S k STATISTIC BY SAMPLE - PUFF**
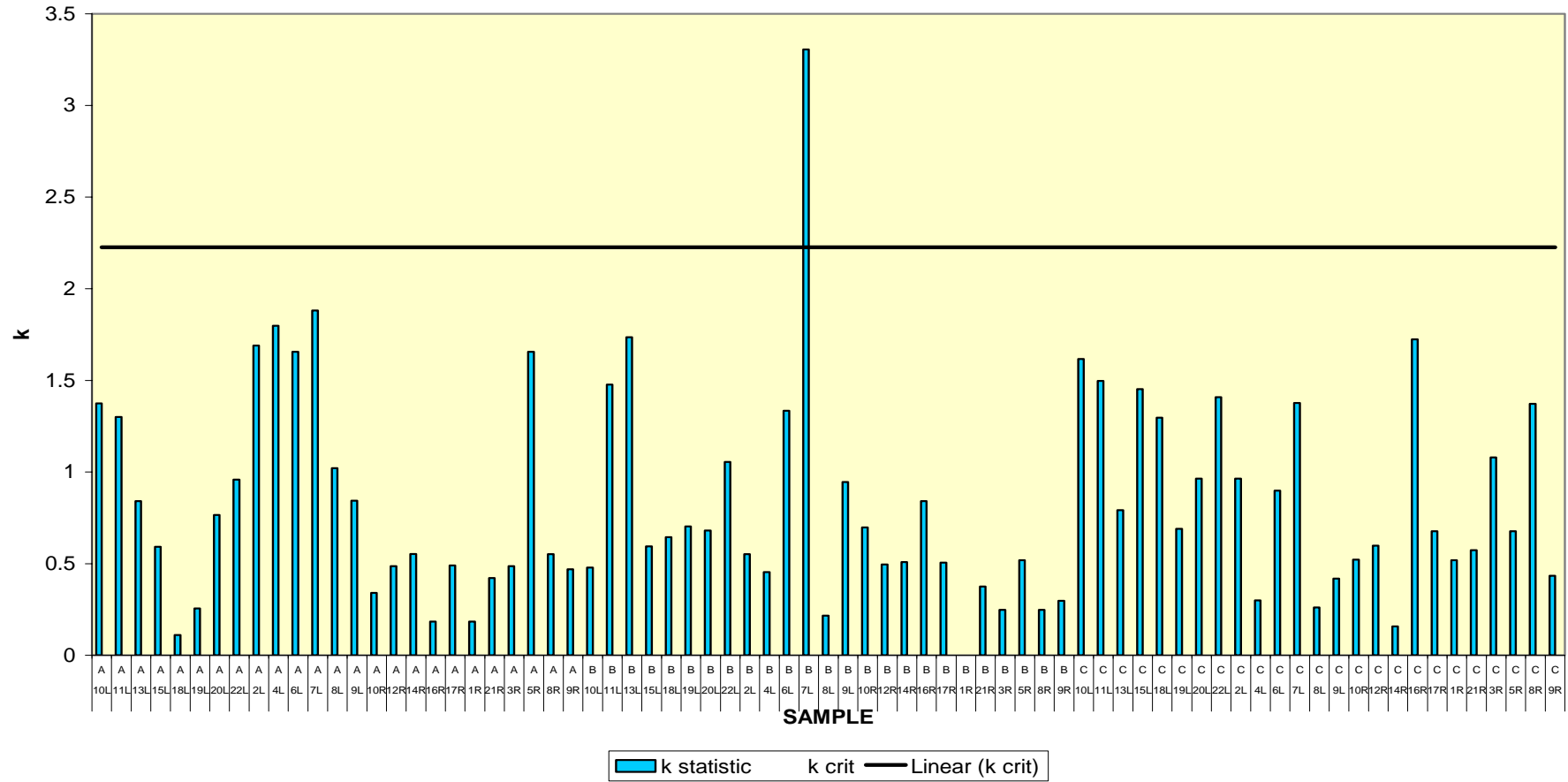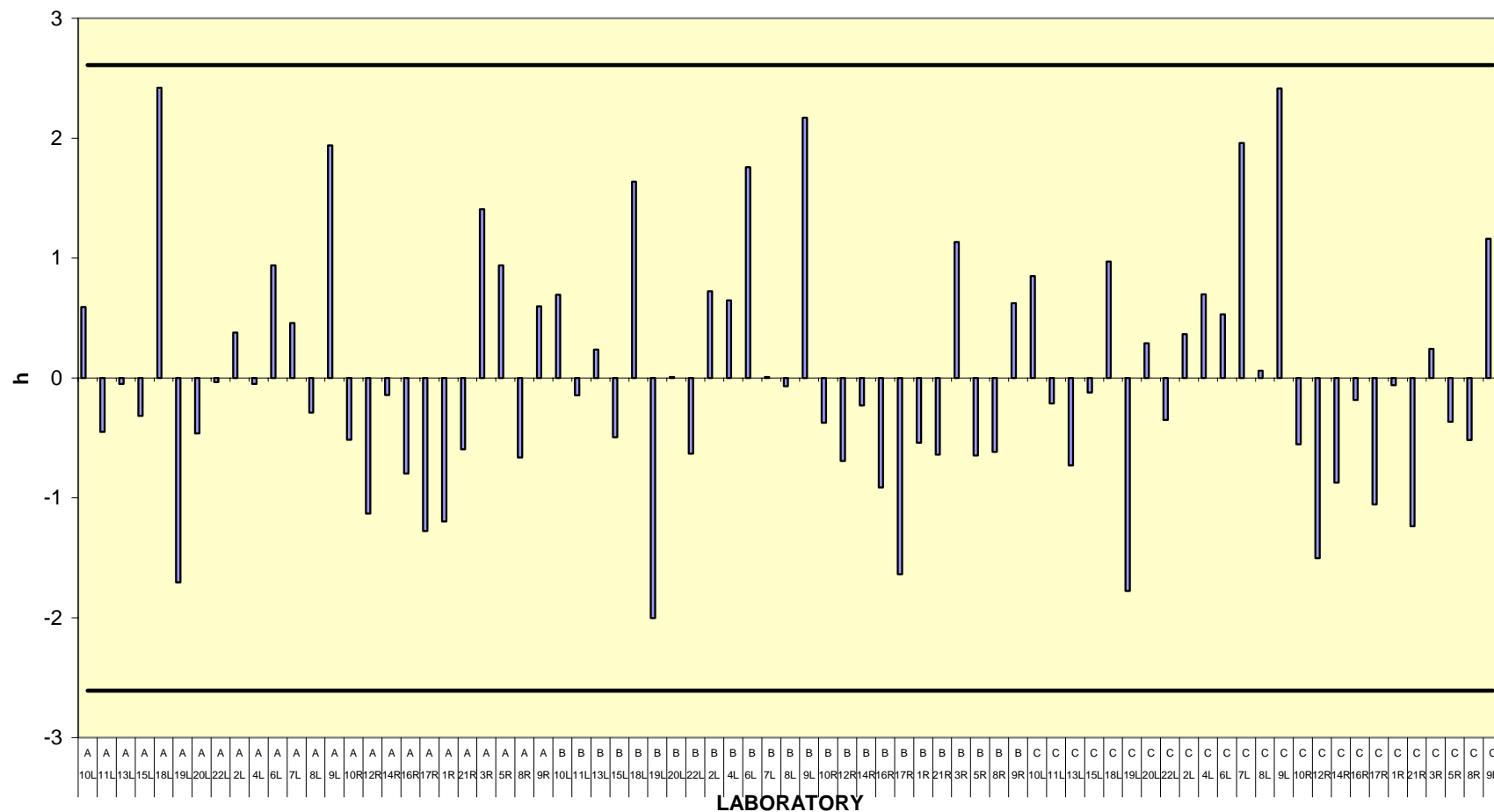
MANDEL'S k STATISTIC BY SAMPLE - MENTHOL
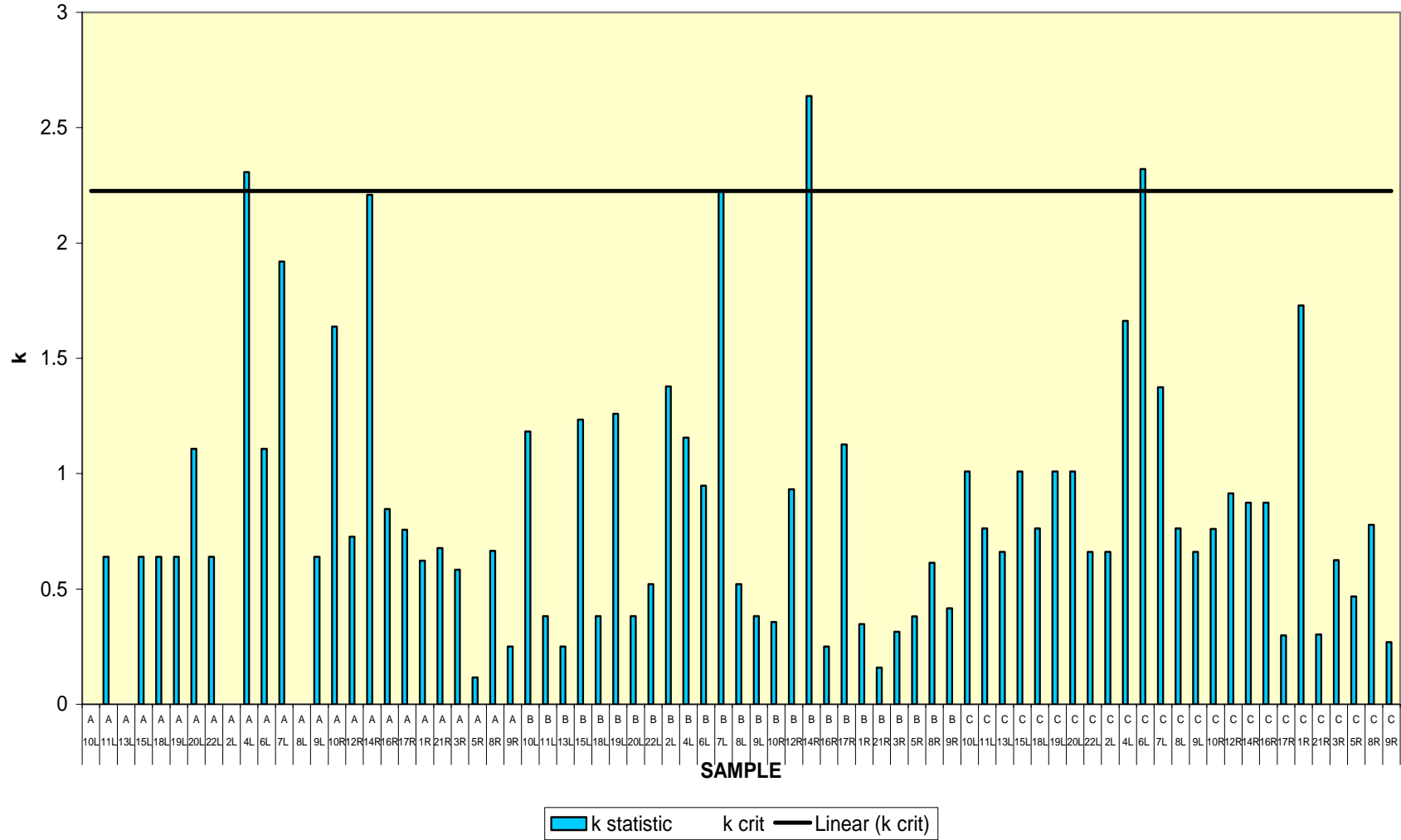
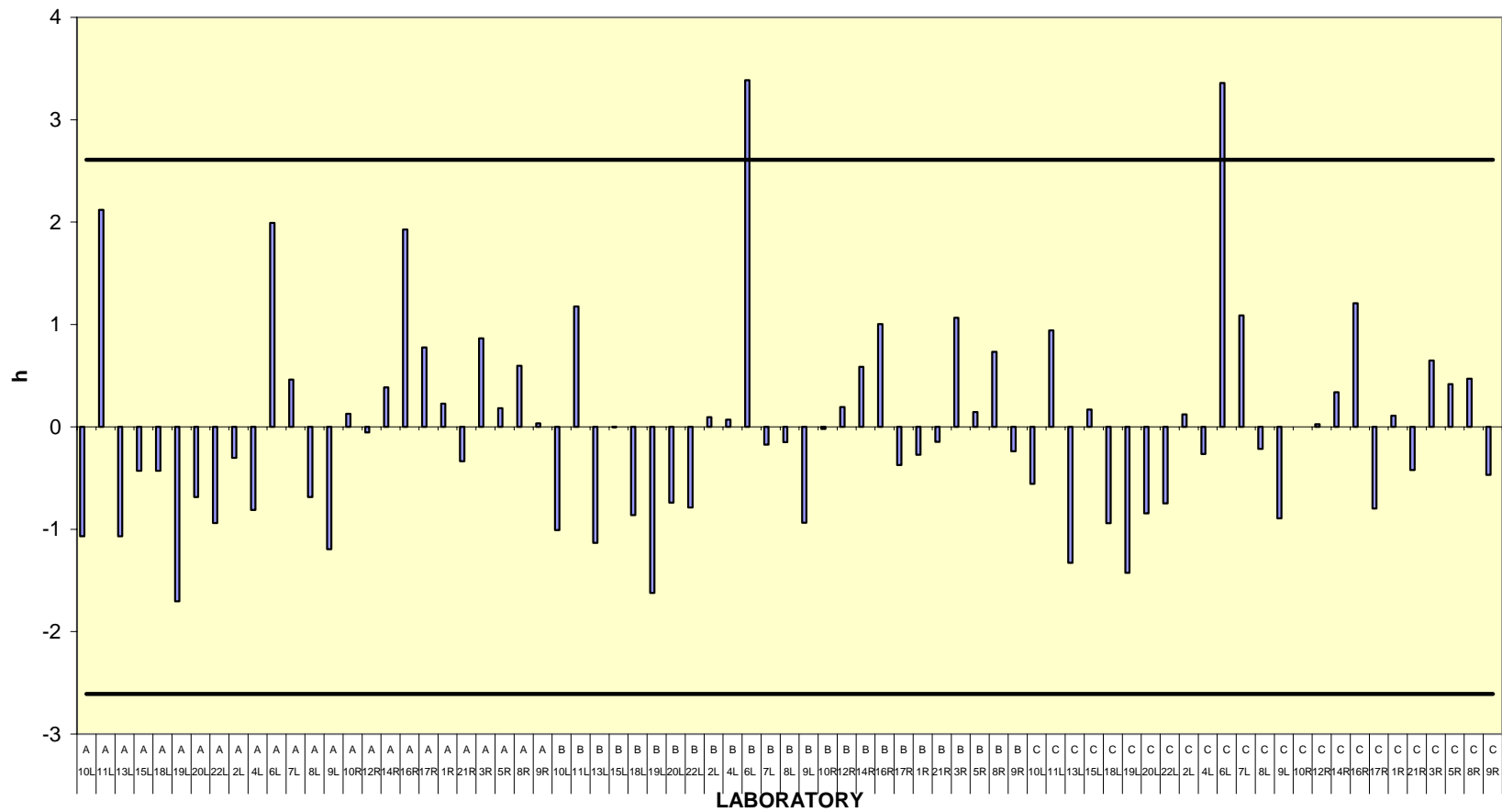MANDEL'S k STATISTIC BY SAMPLE - TPM

MANDEL'S h STATISTIC BY SAMPLE - PUFF

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MANDEL'S h STATISTIC BY SAMPLE - MENTHOL

MANDEL'S h STATISTIC BY SAMPLE - TPM