

Coresta Sub Group
Monitoring and Maintenance of Physical Test Methods
Interlaboratory Proficiency Test 2009

**CORESTA Monitoring and Maintenance of
Physical Test Methods Sub - Group**

2009 Interlaboratory Proficiency Test

Issued by:
Graham Errington
R & D Centre
British American Tobacco
Regents Park Road
Millbrook
Southampton
United Kingdom
SO15 8TL
Phone: 02380 793540
e-mail: graham_errington@bat.com

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PARTICIPATING LABORATORIES

British American Tobacco (Germany) GmbH, Weistrasse 26, 95448 Bayreuth, Germany

British American Tobacco, Denmark

Cerulean, Rockingham Drive, Linford Wood East, Milton Keynes, MK14 6LY, UK

Hauri

Imperial Tobacco - HH

Imperial Tobacco - LA

Product Research Division, Japan Tobacco Inc., JT Sumida Building, 17-7, Yokogawa 1-chome, Sumida-ku,
Tokyo 130-8603, Japan

JT International Germany GmbH, Zentrale Warenaufnahme, R & D Building/GQA-Laboratories,
Diedenhofener Str. 20, D-54294 Trier, Germany

Rhodia Acetow GmbH, Engesserstr. 8, D-79123 Freiburg, Germany

SODIM SA, 4 rue André Dessaux, 45404 Fleury les Aubrais cedex, France

ST Poland, 32-400 Myslenice, Jawornik 360, Poland

Papierfabrik Wattens GmbH & Co KG, Ludwig-Lassl-Strasse 15, A-6112 Wattens, Austria

Zhengzhou Tobacco Research Institute of CNTC, No. 2 Fengyang St., Zhengzhou New & High-Tech Industries,
Development Zone, Henan 450001, China

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2009 CORESTA MMPTM SUB – GROUP INTERLABORATORY PROFICIENCY TEST

Introduction

The purpose of an interlaboratory proficiency testing scheme¹ is to provide participating laboratories with an objective means of assessing and demonstrating the reliability of the data they are producing, and is an important component of any laboratory quality assurance scheme. The importance of proficiency testing is recognised in ISO/IEC Guide 17025² where “*participation in interlaboratory comparison or proficiency testing programmes*” is listed as an important component of quality assurance results.

Outline of Test Protocol

Five filter types and five cigarette brands were selected to cover the range of interest for Individual Weight, Circumference, Pressure Drop and Ventilation.

The samples used in the study are presented in Table 1.

Table 1.

SAMPLE TYPE	SAMPLE ID	VARIANT	Pressure Drop Type
Monoacetate Filter	F1	Semi - Slim	
	F2	Slim	
	F3	King Size/ Regular	High
	F4	King Size/ Regular	Low
	F5	King Size/ Regular	Medium
Cigarette	C1	King Size/ Regular	
	C2	King Size/ Regular	
	C3	Slim	
	C4	Super Slim	
	C5	Super Slim	

Participating laboratories were instructed to measure a sub – group of 10 specimens under their normal operating conditions, repeated 3 times for each sample. Mean values of each replicate were reported in an Excel spreadsheet supplied with the protocol.

After completion, the spreadsheets were returned to the author for data analysis.

Data Analysis

Statistical analysis was carried out using Microsoft Excel and MINITAB v15. All graphs were produced with MINITAB v15.

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Summary statistics were calculated for each sample, followed by individual value plots by laboratory, to identify potential outliers.

Precision Statistics

Following identification and removal of outliers, Reproducibility, R , and repeatability, r , were calculated for each sample and tabulated at the beginning of each section.

Mandel Plots

h statistics for between laboratory consistency, and k statistics for within laboratory consistency were calculated and plotted according to ISO 5725 – 2³, for each product characteristic

Repeatability Variation (Cochran's Test)

Cochran's test⁴ was used to evaluate the within – laboratory performance of each laboratory

$$C = \frac{s_{\max}^2}{\sum_{i=1}^p s_i^2}$$

where s_{\max} is the highest standard deviation in a set of p laboratories. The following criteria were used to assess each laboratory:

If the test statistic, c , is:

- | | |
|--|-----------|
| - \leq 5% critical value | Accept |
| - $>$ 5% critical value $<$ 10% critical value | Straggler |
| - $>$ 10% critical value | Outlier |

Z - scores

After removal of outliers, robust estimates of means of all laboratories were used as the reference for computing z – scores

$$Z = \frac{(x - X)}{s}$$

x = Single mean value

X = Total mean value

s = overall standard deviation adjusted for outliers

Z – scores are plotted in ascending order. Laboratory performance is indicated using the following criteria:

- | | | |
|-------------|--|-----------------------|
| z < 1 | | = good |
| 1 < z < 2 | | = satisfactory |
| 2 < z < 3 | | = questionable |
| z > 3 | | = highly questionable |

Results are presented separately for Filters and cigarettes.

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FILTER DATA ANALYSIS

Table 2. Filter Summary Statistics

Variable	Sample ID	N	N*	Mean	StDev	CoefVar	Minimum	Median	Maximum	Range
Individual Wt Mean mg	F2	42	0	461.45	3.660	0.79	452.50	462.73	466.00	13.500
	F1	41	1	510.45	3.770	0.74	501.30	512.00	518.00	16.700
	F4	42	0	798.88	6.490	0.81	784.60	800.00	810.50	25.900
	F5	42	0	894.37	6.400	0.72	878.40	895.23	903.00	24.600
	F3	42	0	1044.20	8.000	0.77	1025.30	1047.00	1055.80	30.500
Circum Mean mm	F2	42	0	18.00	0.028	0.16	17.93	18.01	18.04	0.117
	F1	41	1	21.18	0.034	0.16	21.10	21.19	21.24	0.143
	F5	42	0	24.47	0.037	0.15	24.39	24.47	24.55	0.165
	F4	42	0	24.43	0.038	0.15	24.34	24.45	24.49	0.146
	F3	42	0	24.43	0.049	0.20	24.23	24.45	24.49	0.260
PD (fully encapsulated)	F4	39	3	243.54	6.070	2.49	222.80	244.00	253.00	30.200
	F1	38	4	396.98	12.760	3.21	351.90	396.60	414.30	62.400
	F2	36	6	412.96	13.300	3.22	370.90	416.00	429.00	58.100
	F5	39	3	538.44	25.480	4.73	432.40	544.70	565.00	132.600
	F3	39	3	779.62	47.290	6.07	598.60	791.40	818.00	219.400

Instrument Manufacturer/ Type Codes for Individual Value Plots

Instrument Manufacturer/Type	Code
Borgwaldt	1
Cerulean	2
Sodiline	3
Sodimat	4
Sodimax	5
Sodimax CCD	6

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Filter Rod Individual Weights

Laboratory N did not measure weights on any filter samples.

Precision Statistics

Individual weights data were screened for outliers and precision statistics calculated for each filter sample.

Table 3. Filter Rod Individual Weights Precision Statistics.

Filter Type	Sample ID	Mean	sr	sR	r	R	CoV
SemiSlim	F1	510.5	1.48	3.77	4.14	10.56	0.74
Slim	F2	461.5	1.27	3.66	3.56	10.25	0.79
KS	F3	1044.2	2.92	8.00	8.18	22.40	0.77
KS	F4	798.9	3.36	6.49	9.41	18.17	0.81
KS	F5	894.4	2.54	6.40	7.11	17.92	0.72

Individual value plots for each sample are shown in Fig.1. Individual data points are represented by solid symbols, and laboratory means by open circles.

Mandel Plots

The h statistic Mandel Plots (Fig.2) show that no laboratory is significantly different.

There are no apparent exceptional values in the k – statistic plots in Fig. 3.

Repeatability Variation

Cochran's test did not identify any exceptional variances.

Z – scores (Fig.4)

Results for Laboratory C are questionable for all filters.

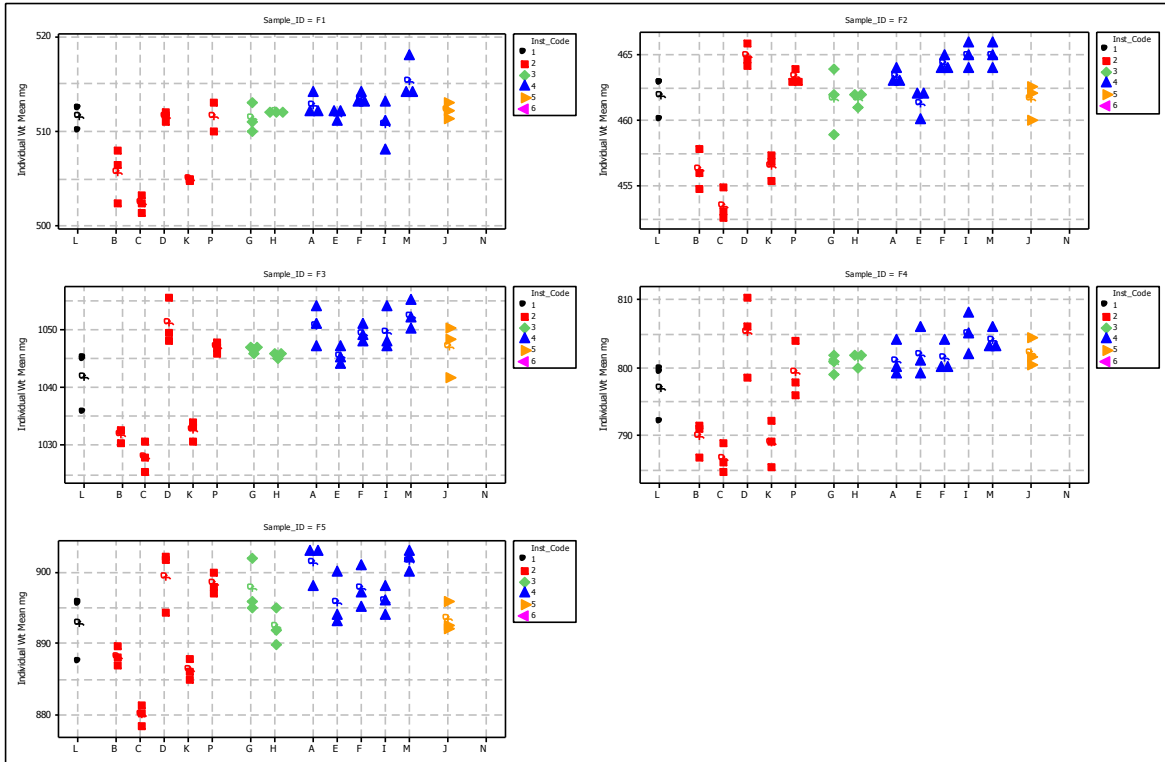
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Figure 1. Filter Rod Individual Weights – Individual Value Plots

Individual Values represented by solid symbols, mean values represented by open circles.

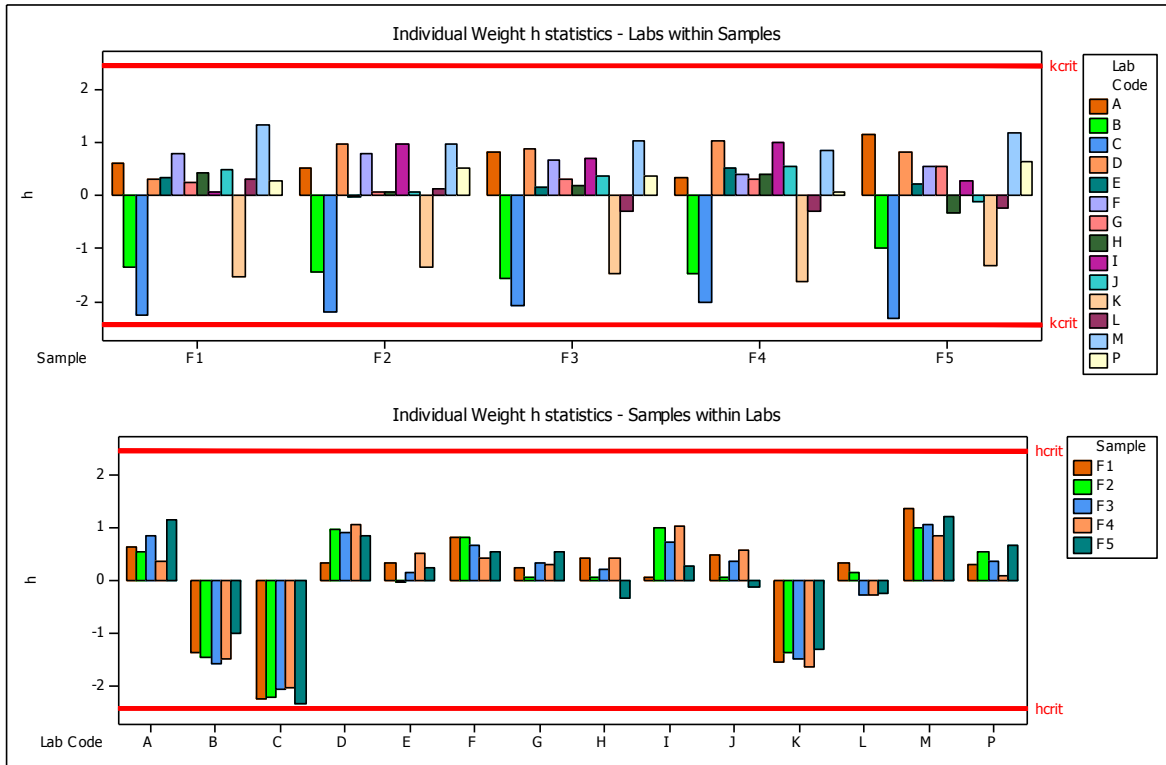


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Figure 2. Filter Rod Individual Weights – *h* statistic Mandel Plots

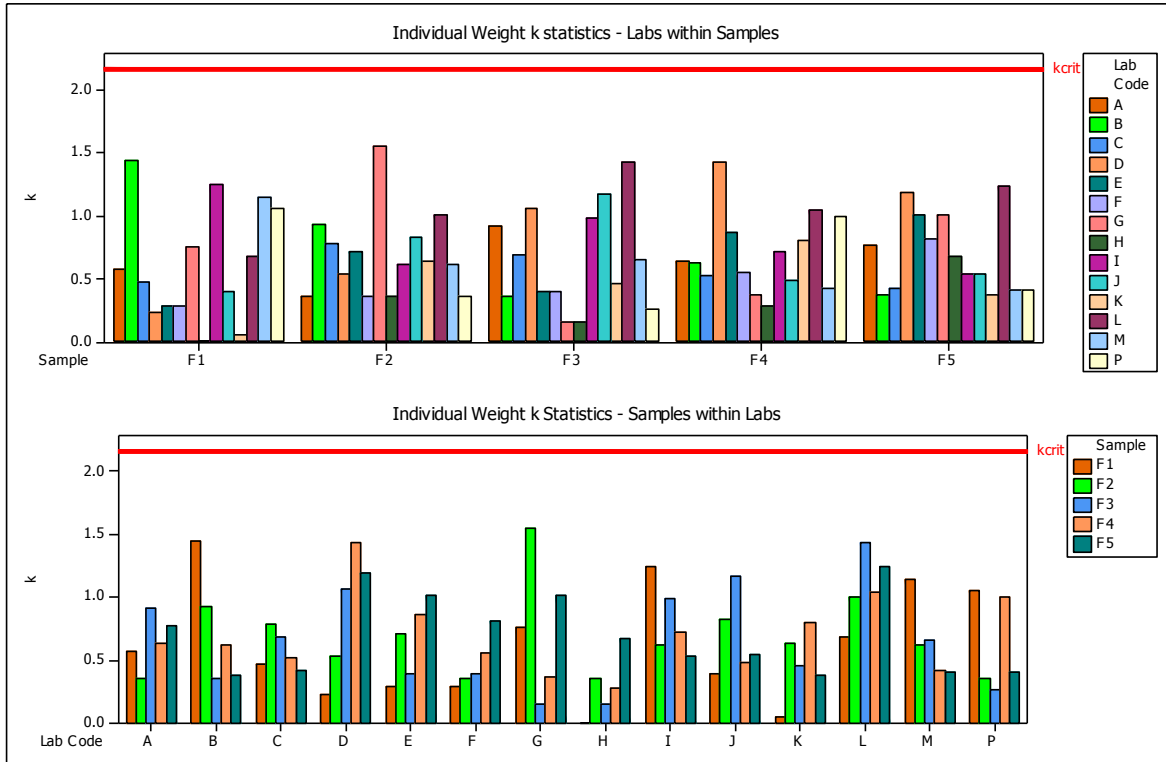


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Figure 3. Filter Rod Individual Weights – k statistic Mandel Plots

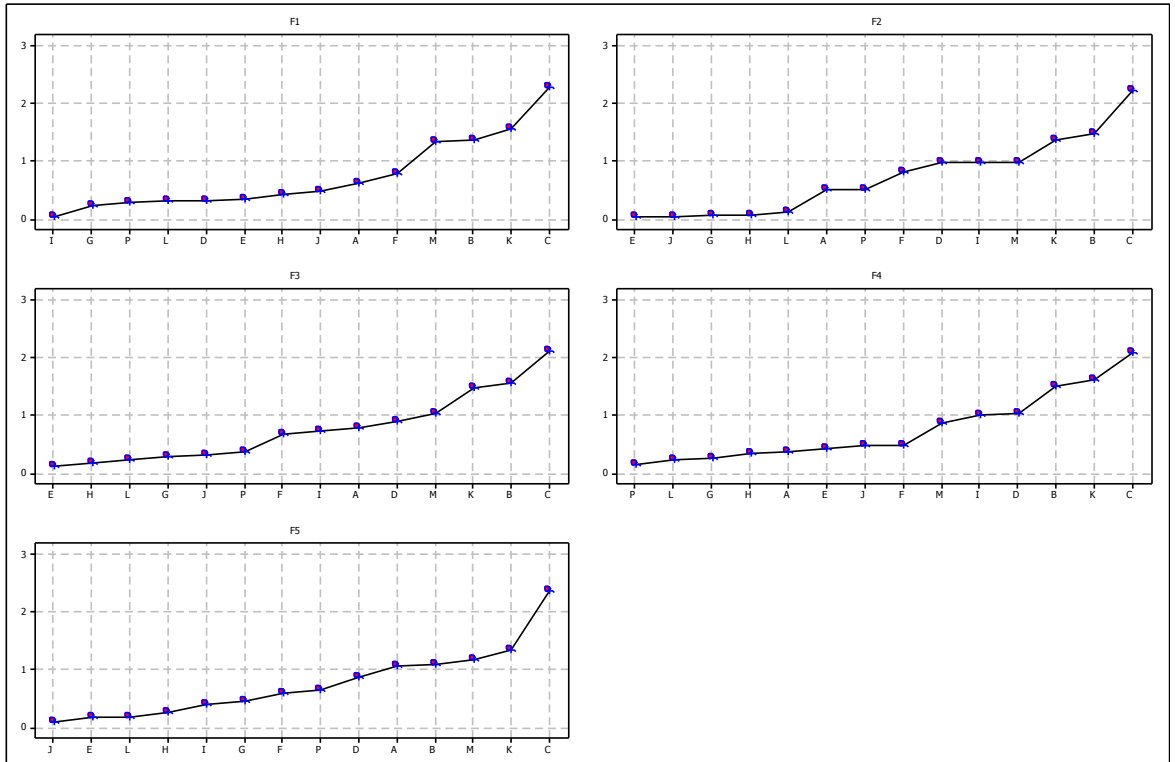


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Figure 4. Filter Rod Individual Weights – z -Score Plots



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Filter Circumference

Table 4. Filter Circumference Precision Statistics.

Results for Lab N excluded. One outlier removed sample F3, laboratory J.

Filter Type	Sample ID	Mean	sr	sR	r	R	CoV
SemiSlim	F1	21.180	0.015	0.034	0.041	0.096	0.16
Slim	F2	18.000	0.009	0.028	0.024	0.078	0.16
KS	F3	24.440	0.016	0.037	0.044	0.104	0.15
KS	F4	24.430	0.014	0.038	0.039	0.106	0.15
KS	F5	24.470	0.019	0.037	0.052	0.102	0.15

Individual value plots for each sample are shown in Fig.5. Individual data points are represented by solid symbols, and laboratory means by open circles.

Mandel Plots

Mandel plots do not show any laboratories beyond critical values (Note Laboratory N was not included in any of the filter analysis, and one measurement removed from sample F3, laboratory J).

Repeatability Variation

Cochran's test did not show any labs as outliers (one measurement removed from laboratory J).

Z – scores

Results for Laboratory C (Fig.8) are questionable for Filters F1 and F2.

Results for Laboratory B, Filters F2 and F4 are questionable.

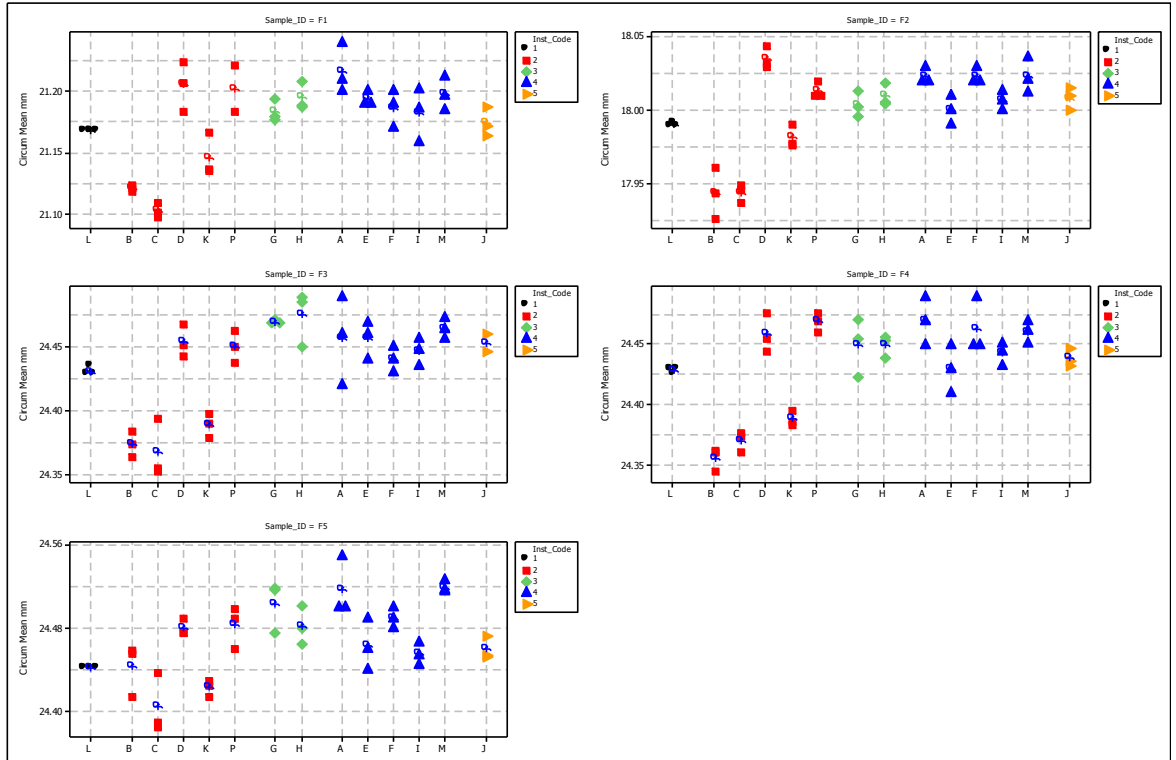
Laboratories with missing data points did not, or were unable to, measure the samples.

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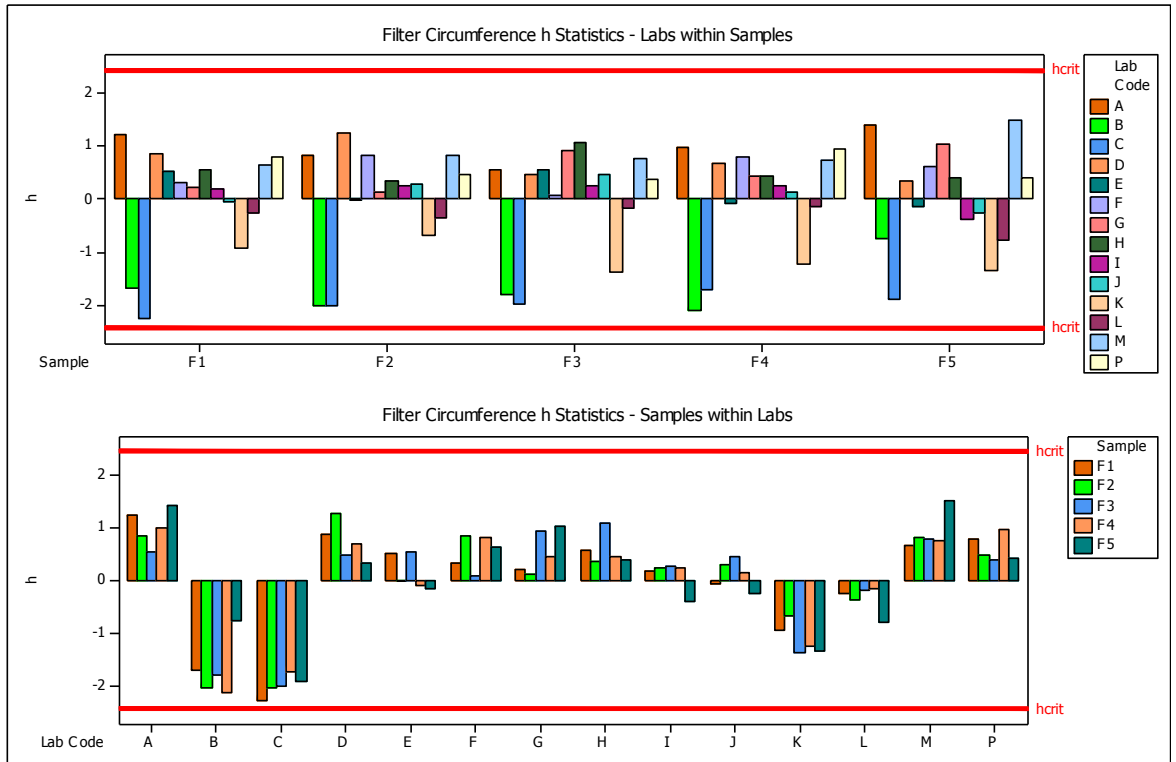
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Figure 5. Filter Rod Circumference – Individual Value Plots



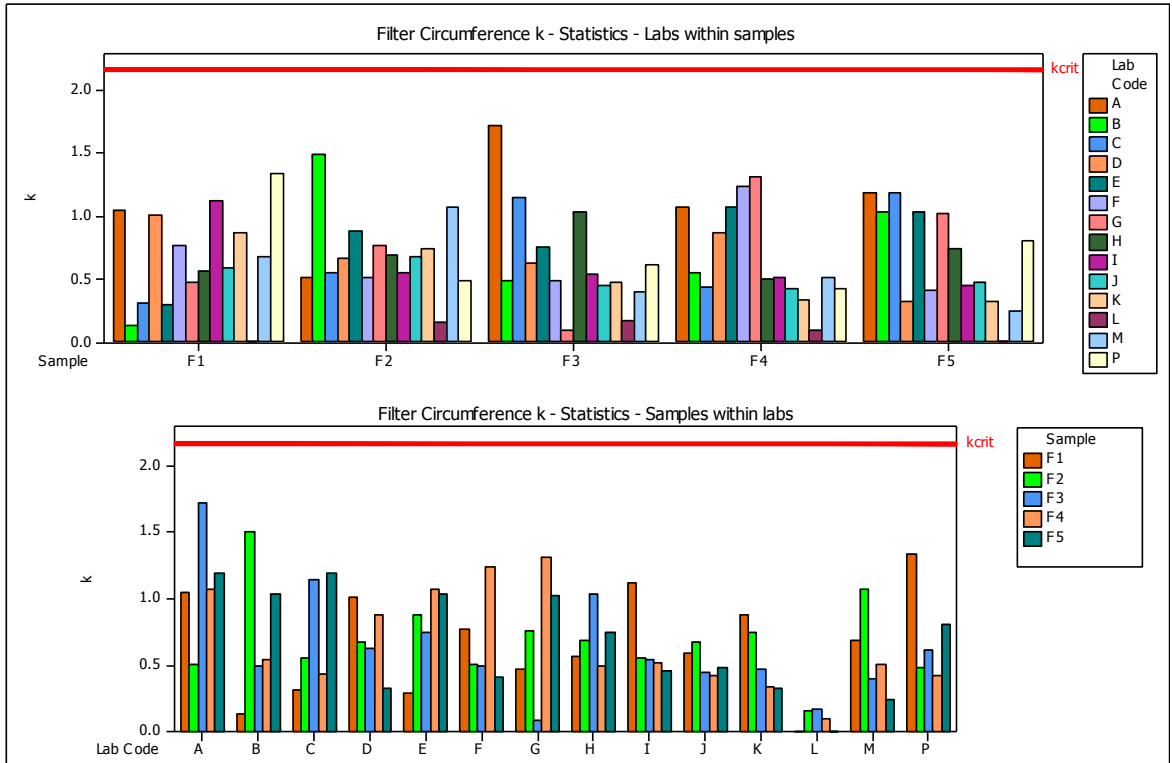
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Figure 6. Filter Rod Circumference: h – statistic Mandel Plots



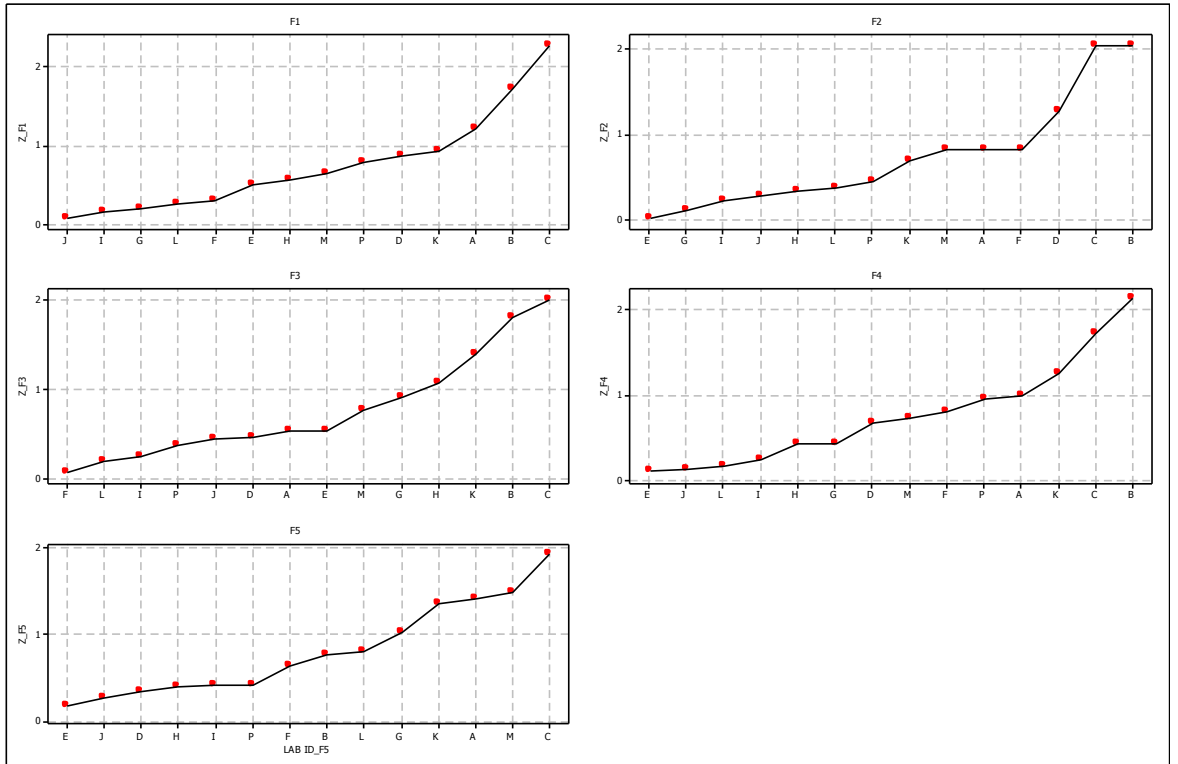
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Figure 7. Filter Rod Circumference: k – statistic Mandel Plots



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Figure 8. Filter Rod Circumference z -score Plots



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FILTER ROD PRESSURE DROP

Table 5. Filter Rod Pressure Drop Precision Statistics

Laboratories N and B not included.

Filter Type	Sample ID	Mean	sr	sR	r	R	CoV
SemiSlim	F1	400.030	2.603	6.770	7.288	18.956	1.69
Slim	F2	416.170	2.297	7.720	6.432	21.616	1.86
KS	F3	792.420	5.513	12.880	15.436	36.064	1.63
KS	F4	244.620	2.519	3.960	7.053	11.088	1.62
KS	F5	545.230	3.266	6.950	9.145	19.460	1.27

Individual value plots for each sample are shown in Fig.9. Individual data points are represented by solid symbols, and laboratory means by open circles.

Mandel Plots

The h statistic Mandel Plots (Fig.10) show that Laboratory B is significantly different from the grand mean for all filters.

The k statistic plot (Fig.11) shows that Laboratory B's within – laboratory variation is significantly different for all samples

Repeatability Variation

Cochran's test identifies the results for all filters for laboratory B as outliers.

Z – scores

Results for all filters for Laboratory B were identified as outliers

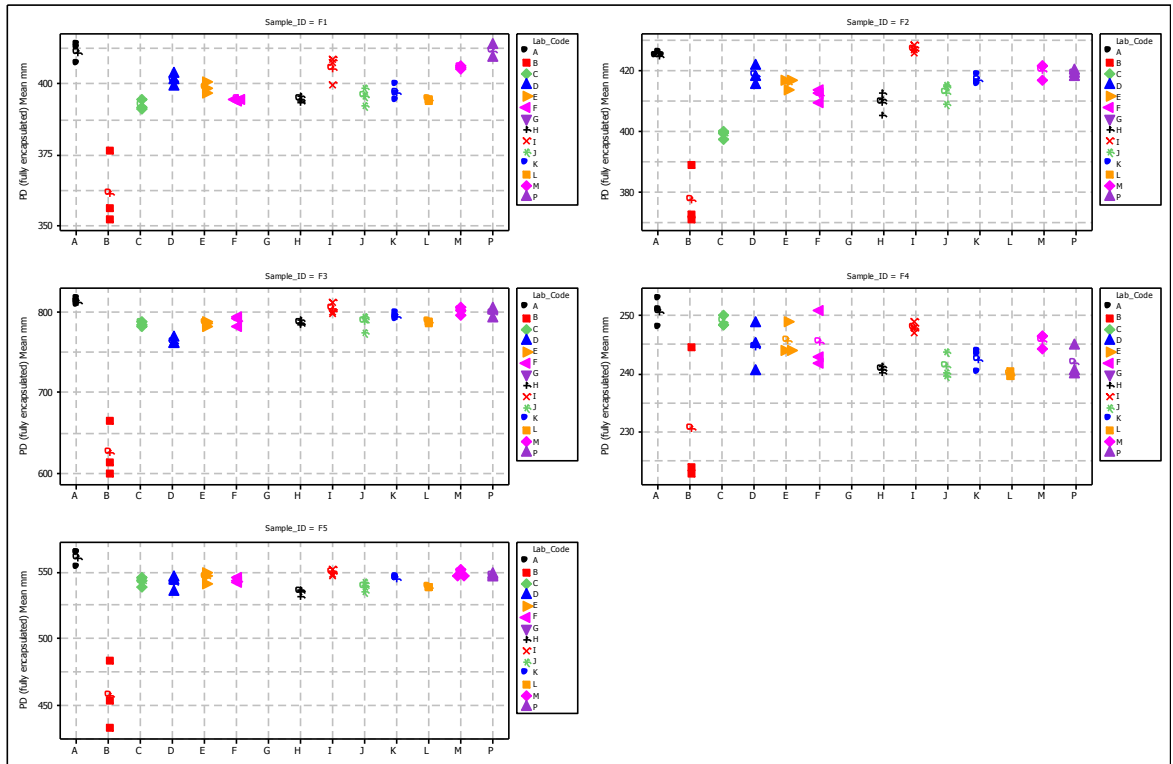
Laboratories with missing data points did not, or were unable to, measure the samples.

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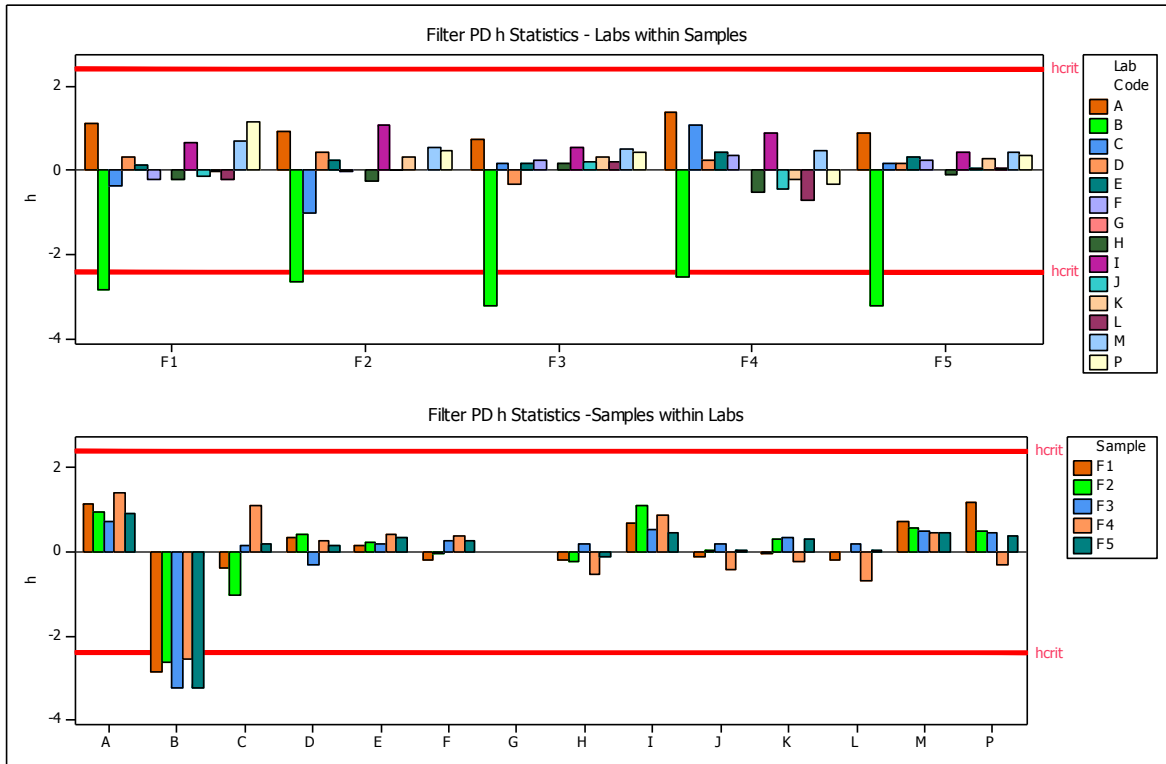
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Figure 9. Filter Rod Pressure Drop – Individual Value Plots



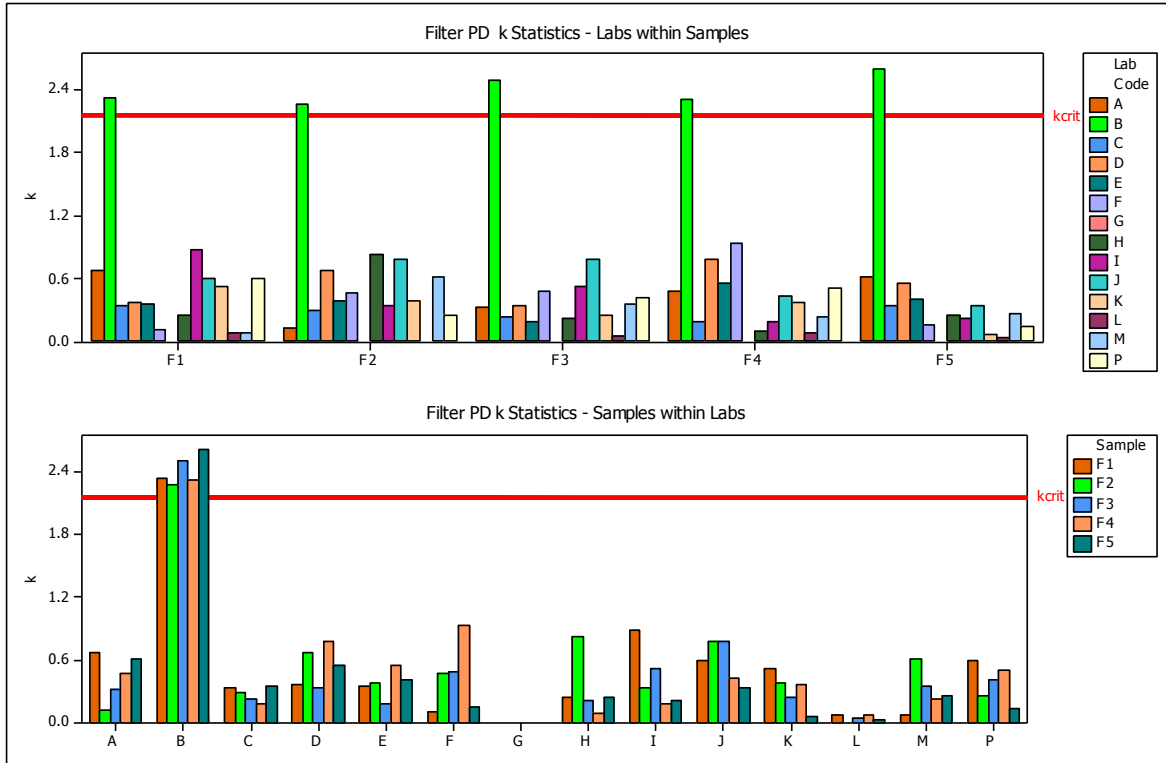
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Figure 10. Filter Rod Pressure Drop: h - statistic Mandel Plots



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Figure 11. Filter Rod Pressure Drop: k - statistic Mandel Plots

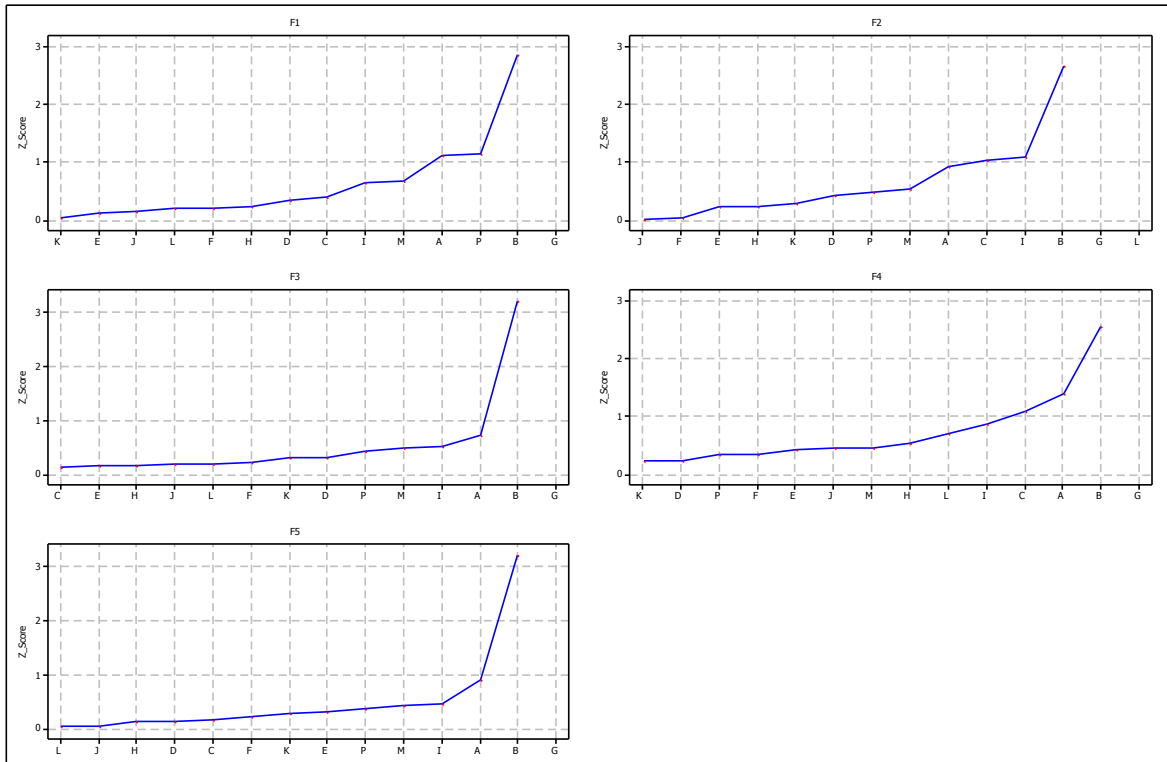


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Filter Rod Pressure Drop – Repeatability Variation

Laboratory G did not submit data. All samples from Laboratory B were outliers.

Figure 12. Filter Rod Pressure Drop: z – score Plots



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CIGARETTE DATA ANALYSIS

Laboratory P did not measure cigarettes. Laboratory N only measured circumference, but the values are not included in the analysis because they are wildly different from the rest of the laboratories. Laboratory I did not report PDo or Ventilation results.

Table 6. Cigarette Summary Statistics

Variable	Sample_ID	N	N*	Mean	StDev	CoefVar	Minimum	Median	Maximum
Individual Wt Mean mg	C1	39	0	954.86	13.410	1.4	921.00	954.00	993.90
	C2	39	0	958.76	12.270	1.3	936.00	959.00	986.00
	C3	39	0	716.26	10.620	1.5	688.00	717.00	741.60
	C4	39	0	664.49	14.390	2.2	636.00	666.00	695.50
	C5	39	0	657.69	12.580	1.9	633.00	661.00	680.70
Circum Mean mm	C1	39	0	24.98	0.488	2.0	24.80	24.90	27.92
	C2	39	0	25.01	0.491	2.0	24.84	24.94	27.97
	C3	39	0	21.46	0.075	0.4	21.24	21.45	21.71
	C4	39	0	18.02	0.027	0.2	17.97	18.01	18.08
	C5	39	0	18.05	0.324	1.8	17.93	17.99	19.97
PD open Mean mmWG	C1	36	3	118.71	6.200	5.2	100.10	119.75	127.80
	C2	36	3	108.61	4.270	3.9	95.90	108.95	117.50
	C3	36	3	120.00	3.130	2.6	115.00	119.50	126.80
	C4	36	3	182.98	9.100	5.0	175.00	180.05	212.40
	C5	36	3	98.32	31.250	31.8	85.00	88.75	204.10
Vent % Mean	C1	36	3	20.86	6.580	31.5	17.43	19.00	42.70
	C2	36	3	29.53	5.780	19.6	26.53	27.84	48.70
	C3	36	3	42.58	2.844	6.7	40.60	41.80	52.30
	C4	36	3	46.10	4.570	9.9	41.85	45.05	61.20
	C5	36	3	81.17	0.836	1.0	78.80	81.40	82.60

Instrument Manufacturer/ Type Codes for Individual Value Plots

Instrument Manufacturer/Type	Code
Borgwaldt	1
Cerulean	2
Sodiline	3
Sodimat	4
Sodimax	5

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CIGARETTE INDIVIDUAL WEIGHTS

Table 7. Cigarette Individual Weights – Precision Statistics

Cigarette Type	Sample ID	Mean	sr	sR	r	R	CoV
SuperSlim	C5	657.7	4.53	12.58	12.68	35.22	1.9
SuperSlim	C4	664.5	6.57	14.39	18.40	40.29	2.2
Slim	C3	715.7	4.38	6.53	12.26	18.28	0.9
KS	C1	955.5	7.09	9.86	19.85	27.61	1.0
KS	C2	958.8	0.57	12.27	1.59	34.36	1.3

Individual value plots for each sample are shown in Fig.13. Individual data points are represented by open circles, and laboratory means by solid circles.

Mandel Plots

The h statistic Mandel Plots (Fig.14) do not show any laboratories significantly different from the reference value.

The k statistic plot (Fig.15) does not suggest exceptional variation for any single laboratory.

Repeatability Variation

Cochran's test shows repeatability variation is acceptable for all laboratories.

Z – scores

Results for Laboratory D (Fig.16), cigarette 1 and 3 are questionable. Results for Laboratory C are questionable.

There are no questionable results for cigarettes 2, 4 and 5.

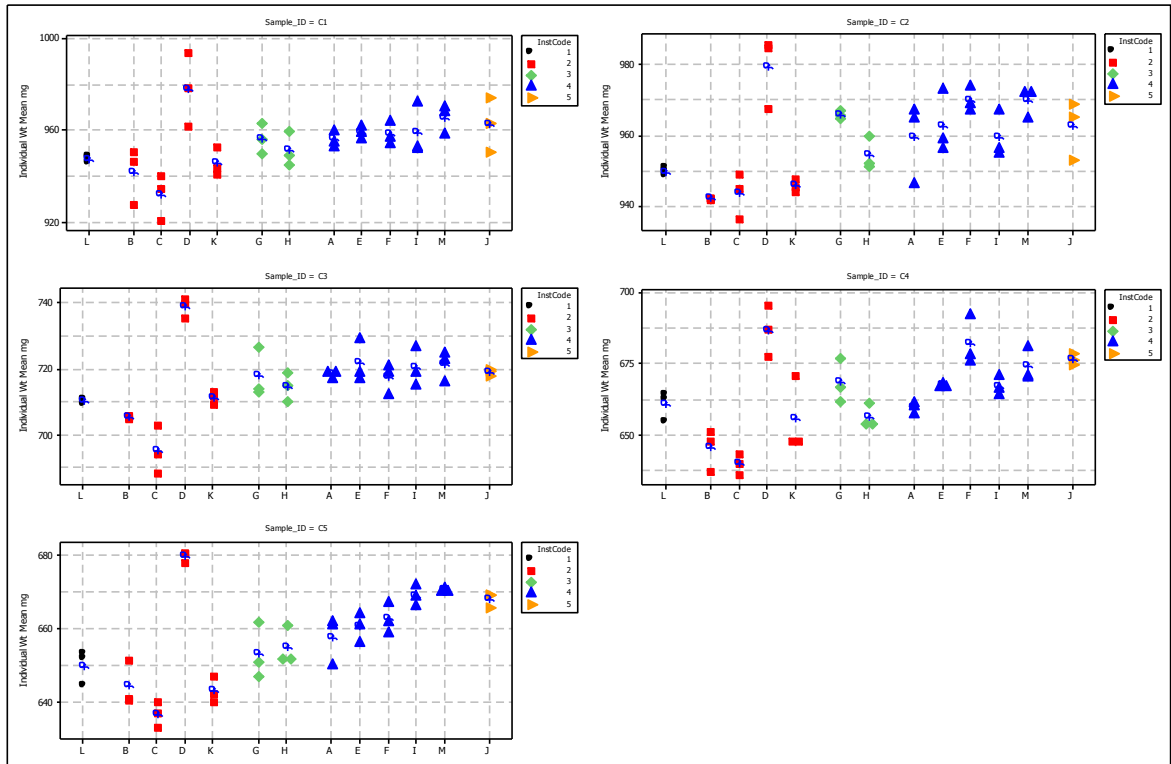
Laboratories with missing data points did not, or were unable to, measure the samples.

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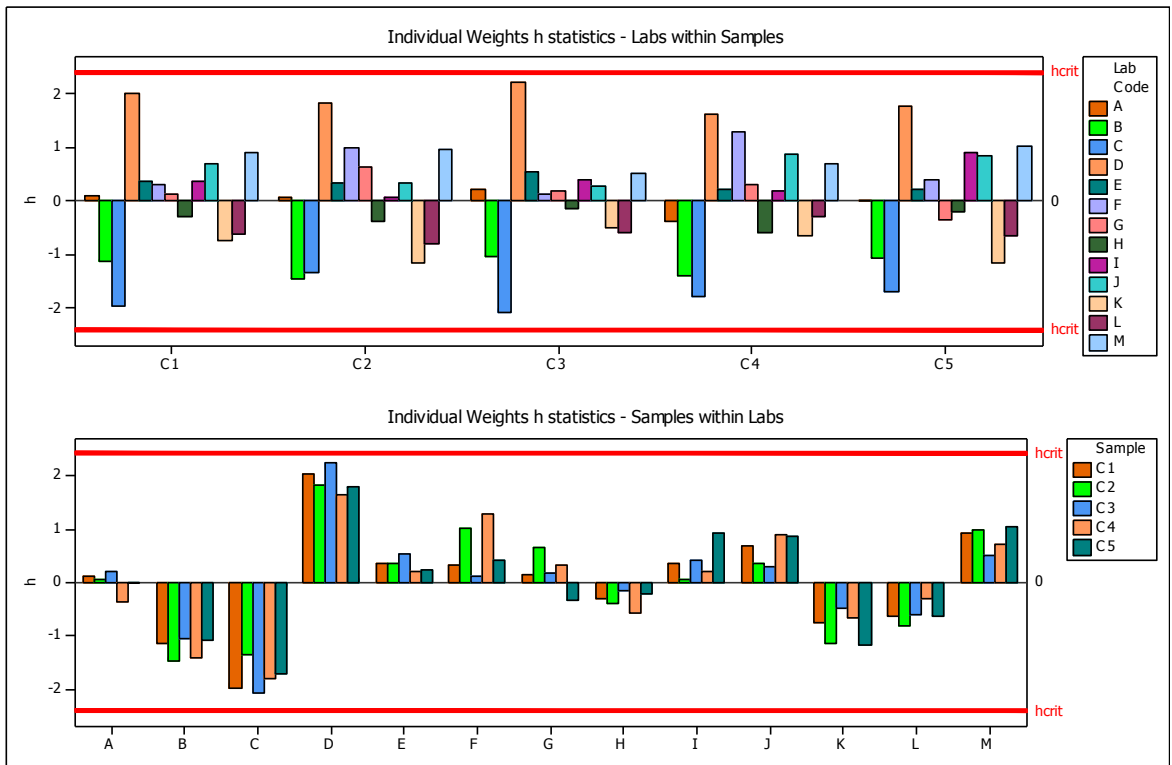
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Figure 13. Cigarette Individual Weights – Individual Value Plot



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Figure 14. Cigarette Individual Weights: h – statistic Mandel Plots

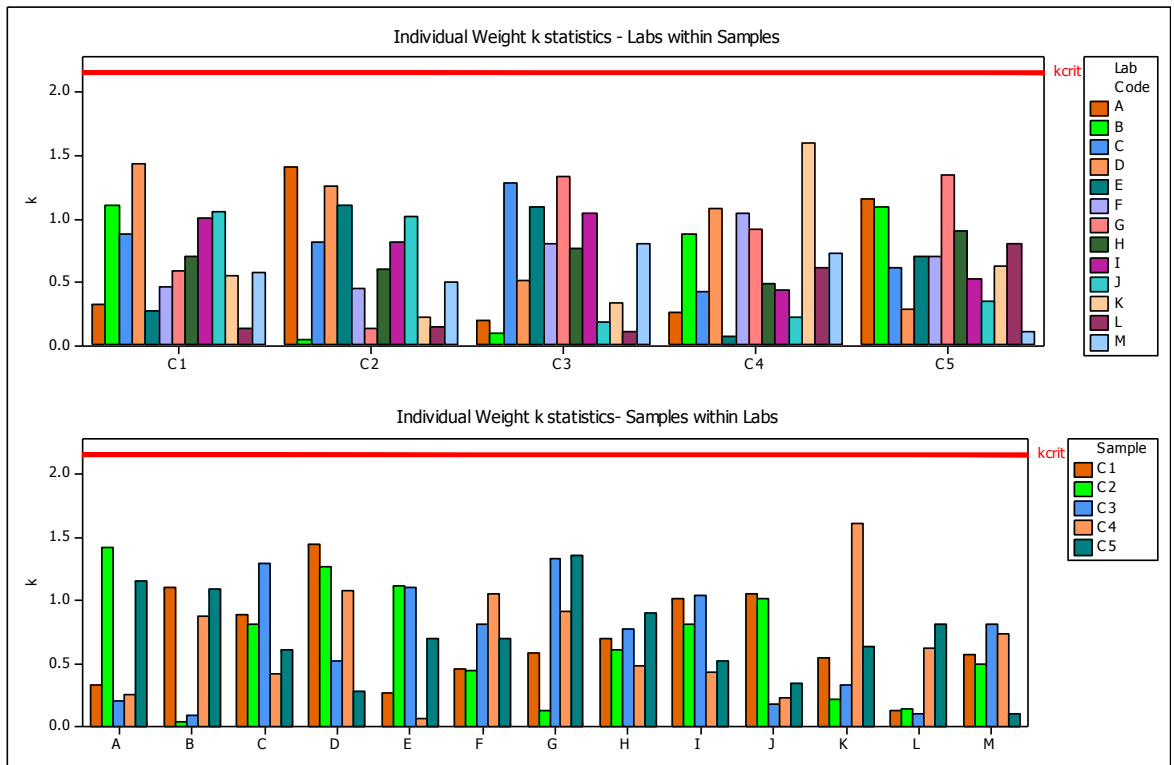


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Figure 15. Cigarette Individual Weights: k – statistic Mandel Plots



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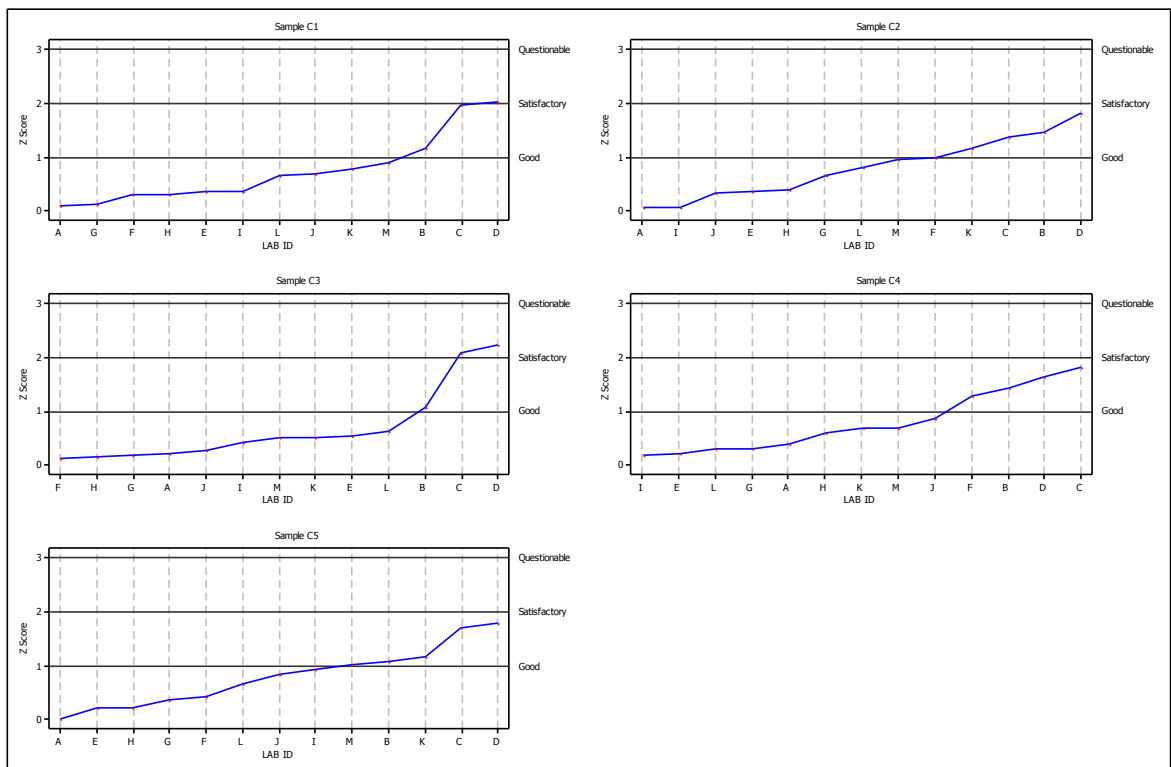
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Cigarette Individual Weights; Repeatability Variation

Cochran's test did not show any laboratories with exceptional variation.

Figure 16. Cigarette Individual Weights: z -score Plots



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CIGARETTE CIRCUMFERENCE

Table 8. Cigarette Circumference Precision Statistics

Cigarette Type	Sample ID	Mean	sr	sR	r	R	CoV
SuperSlim	C5	17.99	0.020	0.026	0.057	0.072	0.14
SuperSlim	C4	18.02	0.024	0.027	0.066	0.076	0.15
Slim	C3	21.45	0.022	0.046	0.063	0.129	0.21
KS	C1	24.90	0.037	0.058	0.104	0.162	0.23
KS	C2	24.92	0.040	0.051	1.588	0.143	0.21

Individual value plots for each sample are shown in Fig. 17. Individual data points are represented by open circles, and laboratory means by solid circles.

Mandel Plots

The h statistic Mandel Plots (Fig.18) shows that Laboratory L is significantly different for cigarette C5

The k statistic plot (Fig.19) identifies Laboratory A as significantly different for cigarette C3, and Laboratory L for cigarette C5.

Repeatability Variation

Cochran's test shows Laboratory A cigarette C3, and Laboratory L, cigarette C5 as outliers, and Laboratory A cigarette C4 as straggler.

Z – scores

Results for Laboratory L, cigarette C5 (Fig.20) are highly questionable, results for Laboratory A cigarettes C1 and C2, are questionabel.

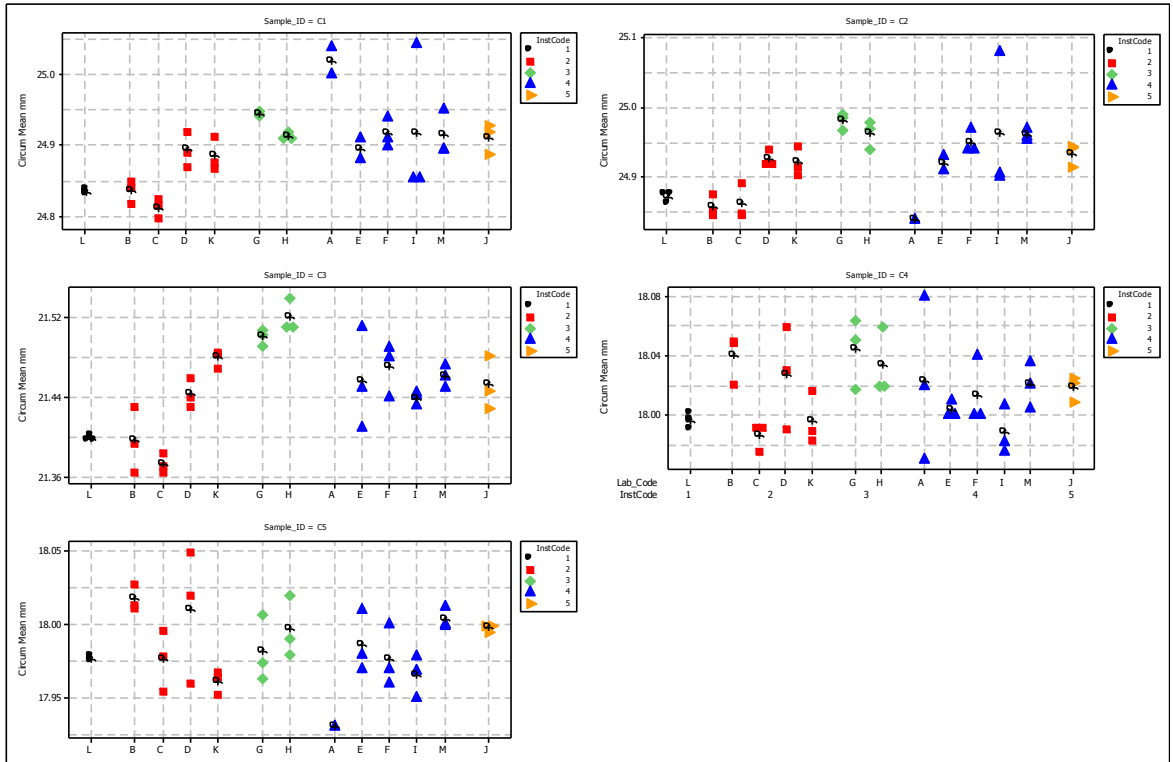
Laboratories with missing data points did not, or were unable to, measure the samples.

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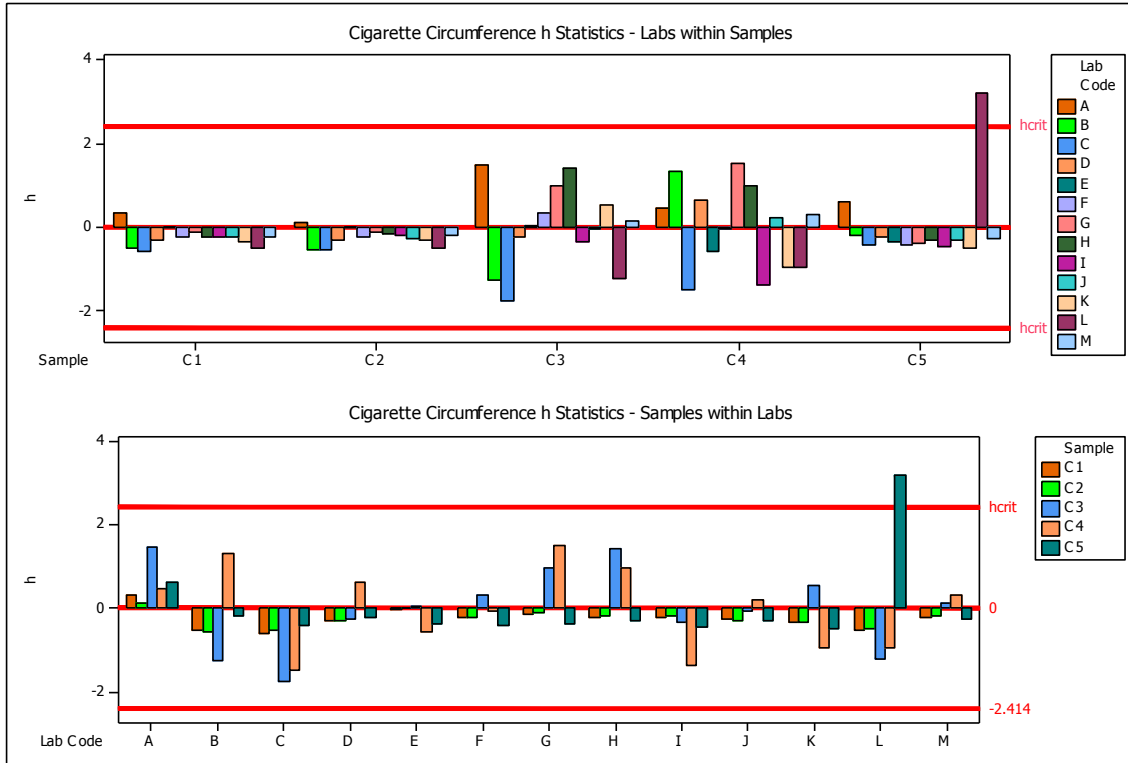
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Figure 17. Cigarette Circumference Individual Value Plots



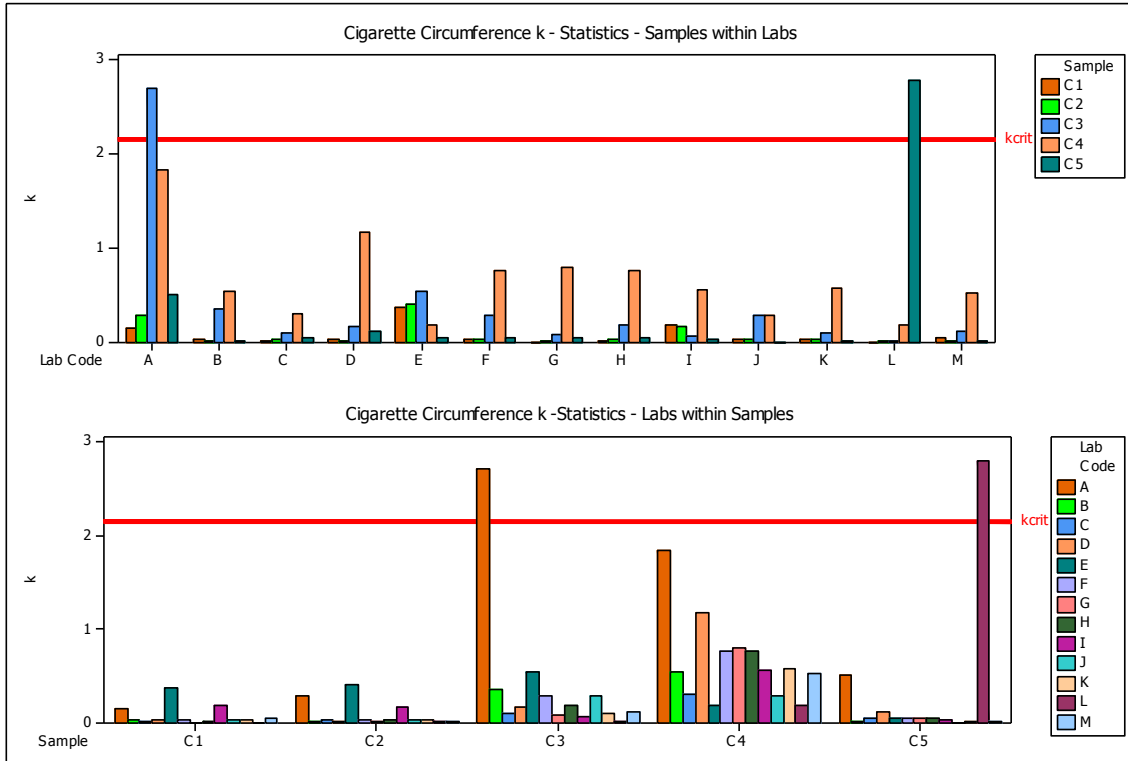
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Figure 18. Cigarette Circumference: h – statistics Mandel Plots



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Figure 19. Cigarette Circumference: k – statistics Mandel Plots

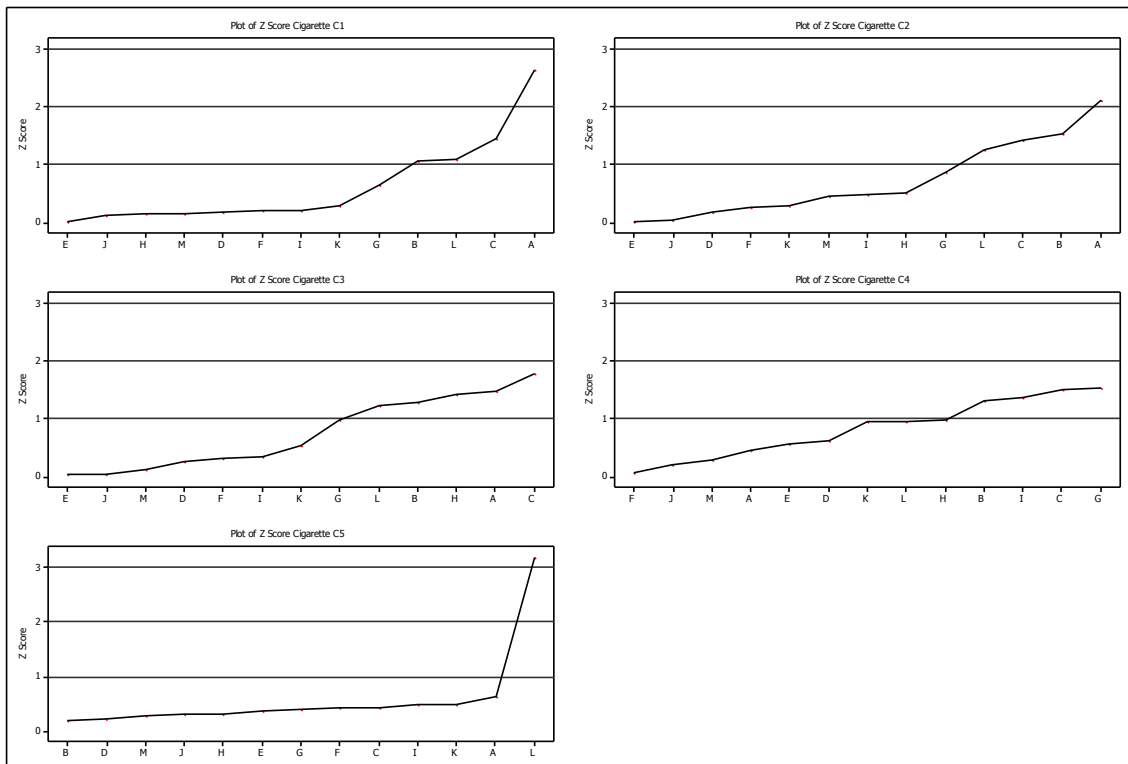


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Cigarette Circumference – Repeatability Variation

Laboratory A cigarette C3 and Laboratory L, cigarette C3 as outliers, and Laboratory A cigarette C4 as a straggler.

Figure 20. Cigarette Circumference: z -score Plots



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CIGARETTE PRESSURE DROP (OPEN)

Table 9. Cigarette Pressure Drop (Open) – Precision Statistics

Cigarette Type	Sample ID	Mean	sr	sR	r	R	CoV
SuperSlim	C5	88.8	1.40	2.95	3.92	8.26	3.32
SuperSlim	C4	180.6	3.35	4.35	9.38	12.18	2.41
Slim	C3	120.0	1.57	3.13	4.40	8.76	2.61
KS	C1	120.3	1.48	3.18	4.14	8.90	2.64
KS	C2	109.4	1.19	2.23	3.33	6.24	2.04

The Individual values plots for cigarette pressure drop identified Laboratory C as different enough to distort the scale on the graphs. The plots were redrawn without these values (Fig.21).

Mandel Plots

The h statistic Mandel Plots (Fig.22) shows that Laboratory C is significantly different from the reference value, for all cigarettes except C3.

The k statistic plot (Fig.23) does not suggest exceptional variation for any single laboratory.

Repeatability Variation

Cochran’s test did not identify any outliers.

Z – scores

Laboratory C gave questionable results for cigarettes C1, C2, C4 and C5 (Fig.24). There were no questionable results for cigarette C3

Laboratories with missing data points did not, or were unable to, measure the samples.

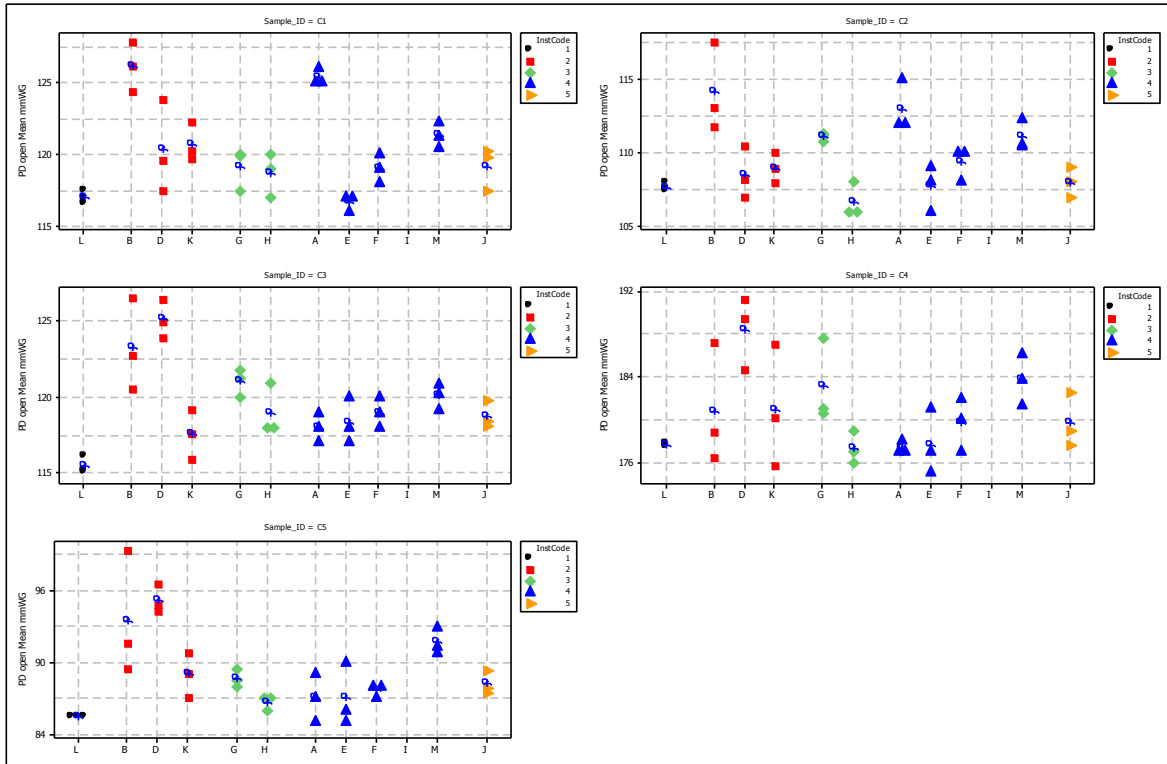
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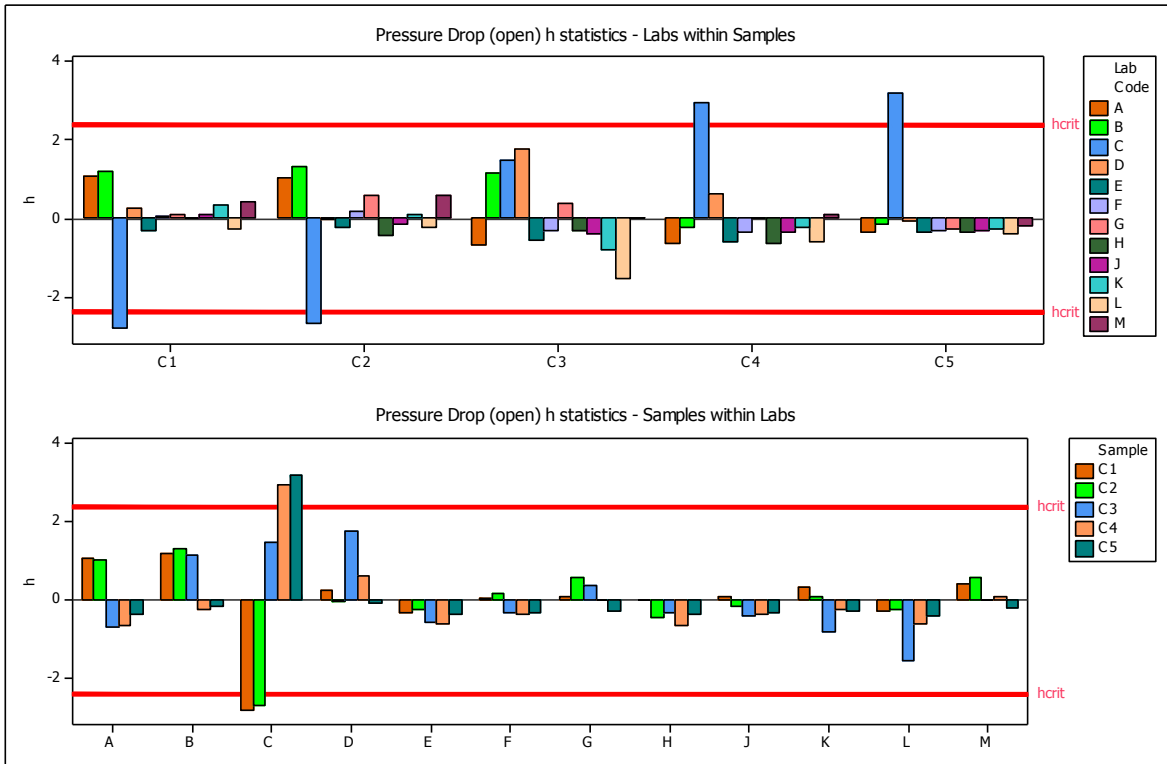
Figure 21. Cigarette Pressure Drop: Individual Value Plots

Labs N. I. P did not measure cig Pressure Drop, Lab C results removed from individual value plots.



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Figure 22. Cigarette Pressure Drop: *h* – statistics Mandel Plots

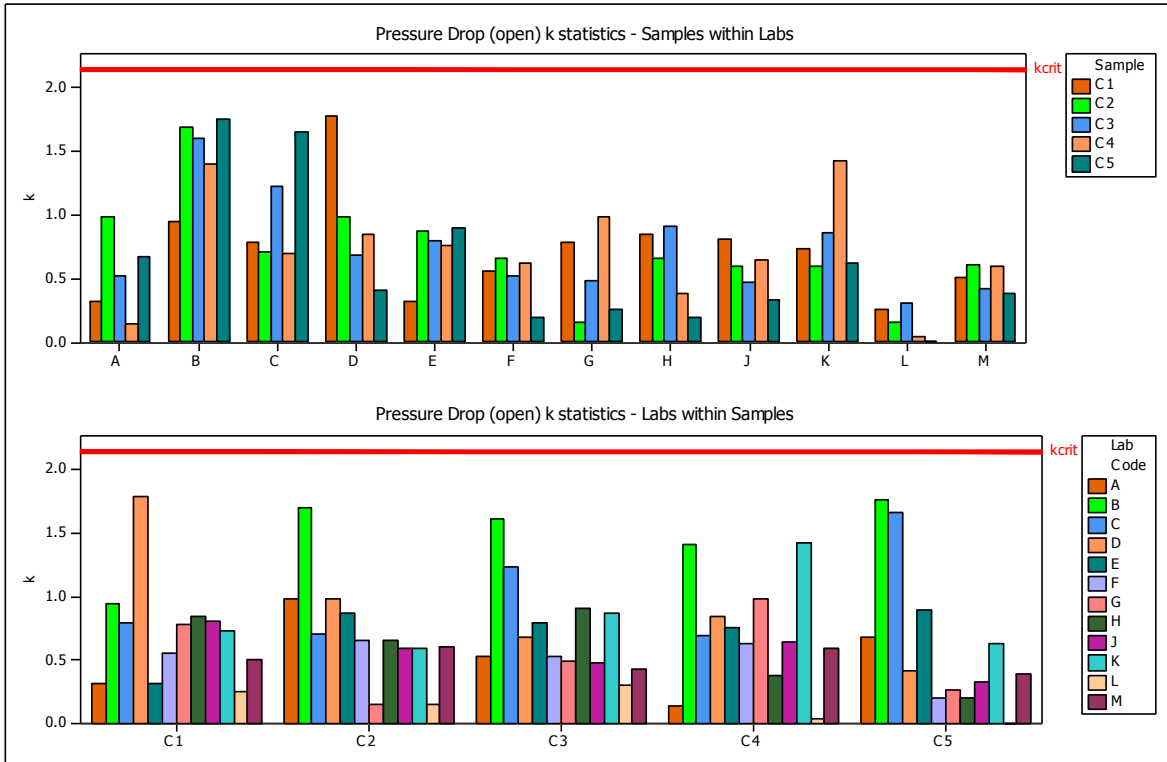


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Figure 23. Cigarette Pressure Drop: k – statistics Mandel Plots



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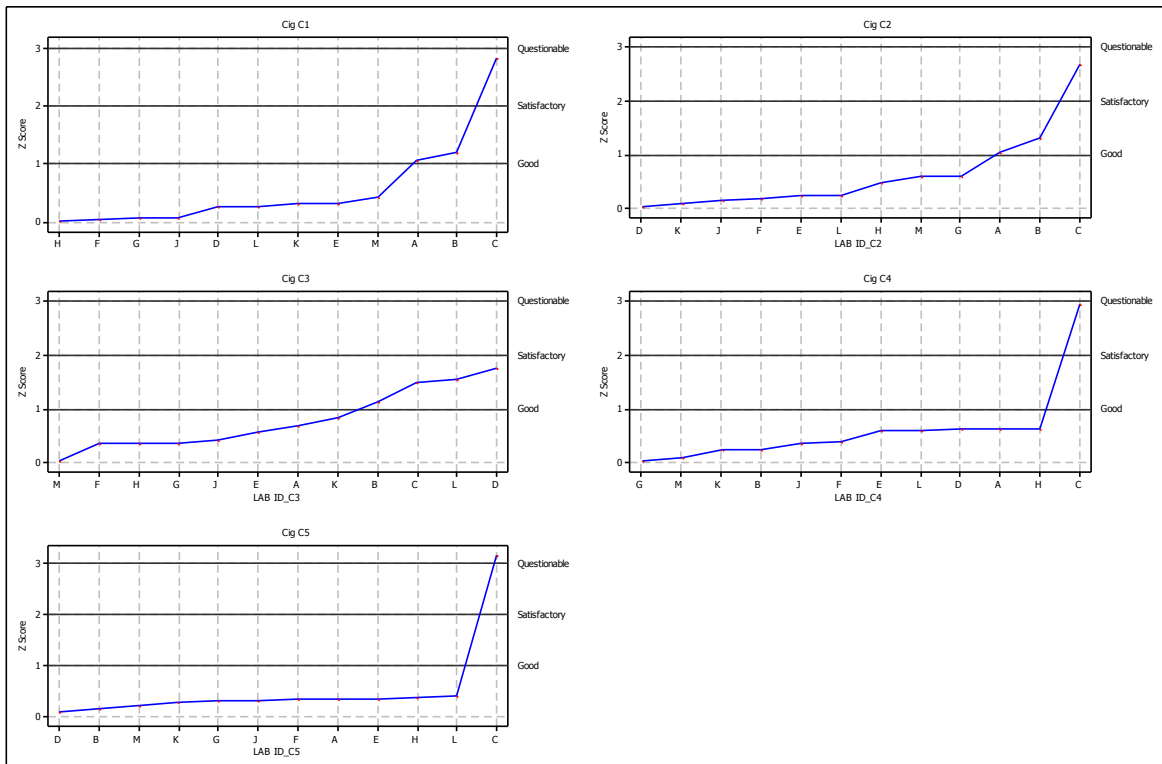
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Cigarette Pressure Drop (open) – Repeatability Variation: Cochran’s Test

Cochran’s test identified Labs B and C as stragglers for sample C5. There were no unusual results for the other 4 samples

Figure 24. Cigarette Pressure Drop: z - score Plots



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CIGARETTE VENTILATION

Table 10. Cigarette Ventilation: Precision Statistics

Cigarette Type	Sample ID	Mean	sr	sR	r	R	CoV
SuperSlim	C5	81.4	0.394	0.498	1.103	1.394	0.61
SuperSlim	C4	44.9	0.643	0.938	1.800	2.626	2.09
Slim	C3	41.8	0.334	0.428	0.935	1.198	1.02
KS	C2	27.8	0.567	0.861	1.588	2.411	3.09
KS	C1	18.9	0.399	0.573	1.117	1.604	3.03

The Individual values plots for cigarette pressure drop identified Laboratory C as different enough to distort the scale on the graphs. The plots were redrawn without these values (Fig.25).

Mandel Plots

Laboratory C was excluded from the Mandel plots because of distortion of the graphs.

The h statistic Mandel Plots (Fig.26) shows that Laboratory B is significantly different from the reference value, for cigarettes C4 and C5. Laboratories A and B, in general, show a greater deviation from the reference values

The k statistic plot (Fig.27) does not show any exceptional variation

Repeatability Variation

Cochran's test did not suggest any exceptional variation.

Z – scores

Results for Laboratory C (Fig.28) are highly questionable for C1, C2, C3 and C4, and questionable for C5.

Results for Laboratory A, cigarette C2 are questionable.

Results for Laboratory B cigarette C4 are questionable.

Laboratories with missing data points did not, or were unable to, measure the samples, or had extreme values which distorted the plots.

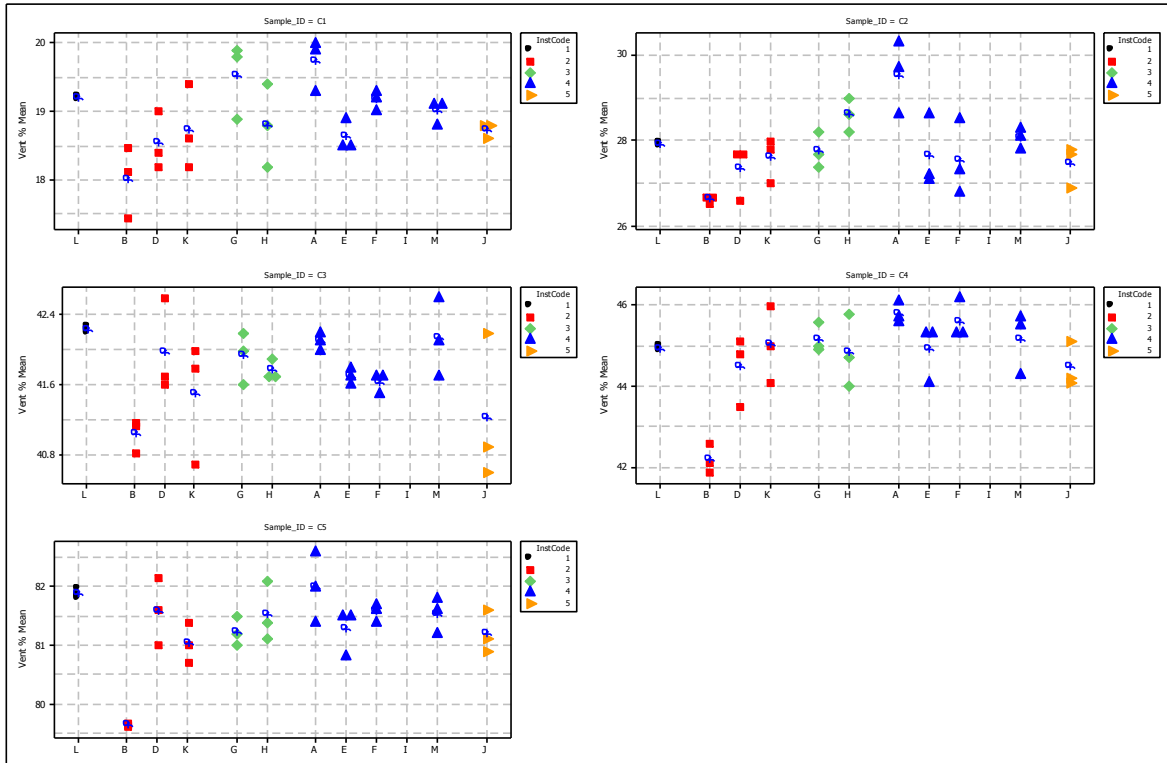
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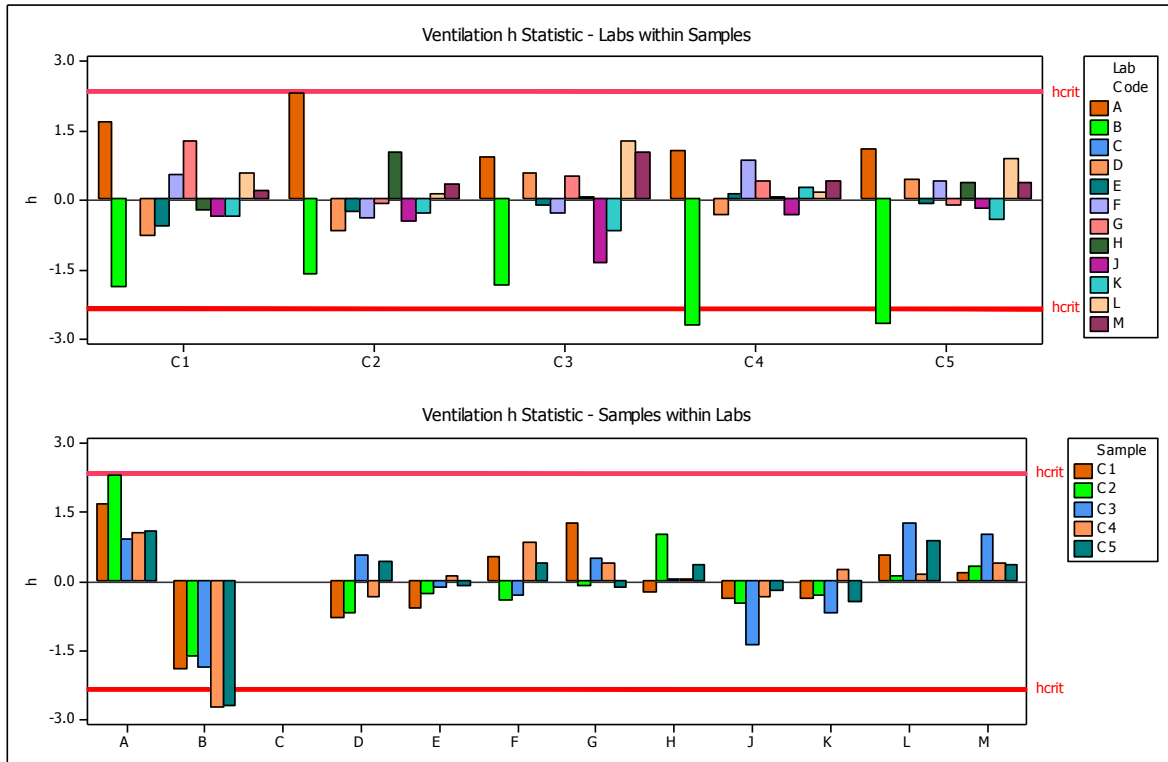
Figure 25. Cigarette Ventilation: Individual Value Plots

Laboratory C excluded from graphs because of wildly different results.



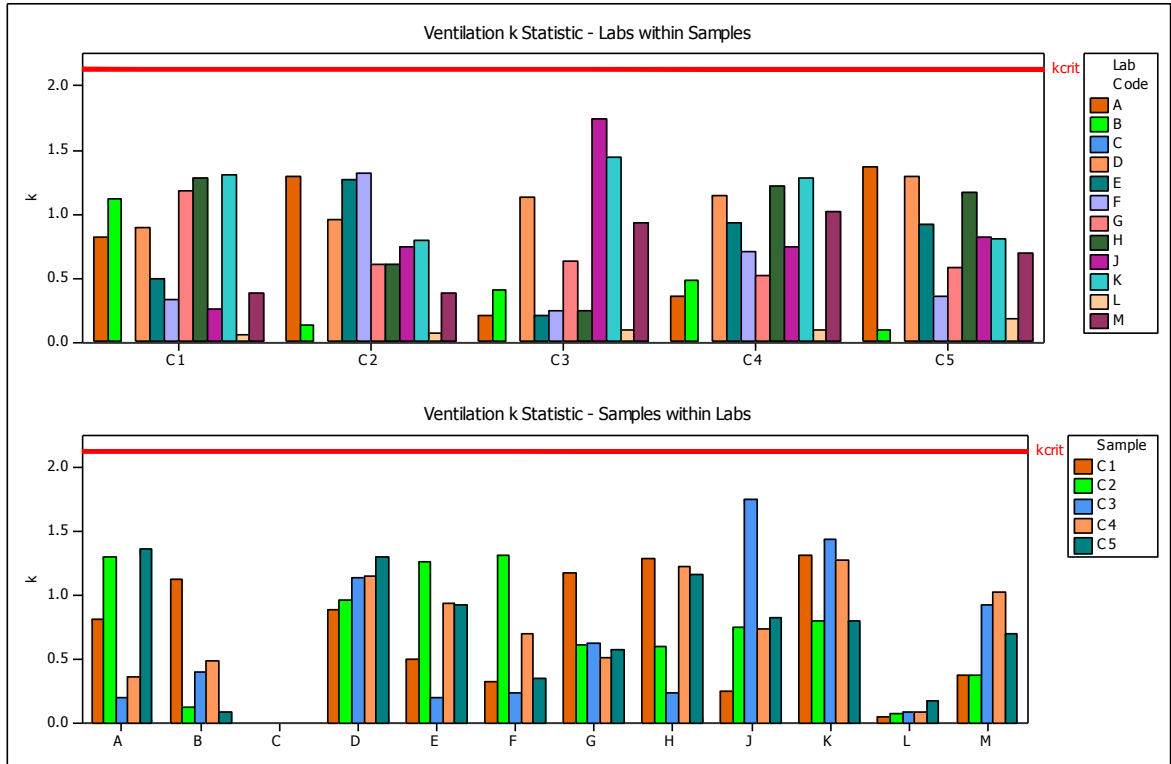
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Figure 26. Cigarette Ventilation: h – statistics Mandel Plots



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Figure 27. Cigarette Ventilation: k – statistics Mandel Plots

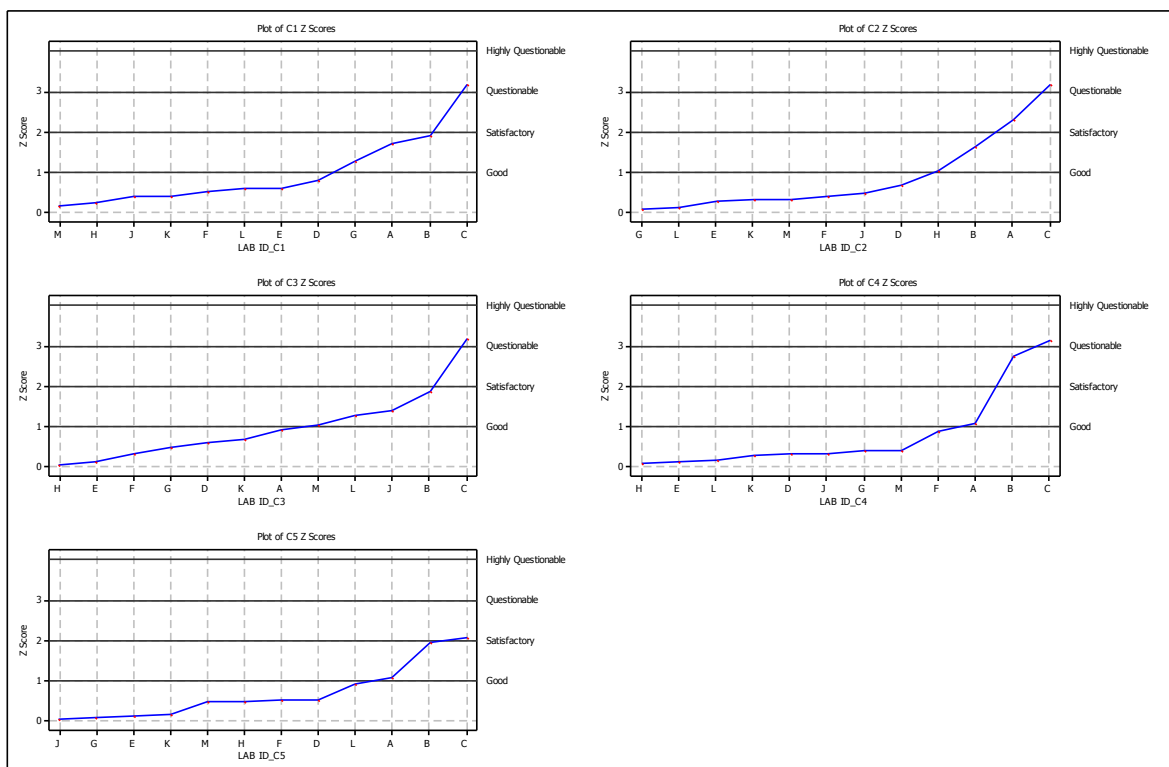


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Cigarette Ventilation: Repeatability Variation

All results were acceptable using Cochran's test.

Figure 28. Cigarette Ventilation: z –score Plots



¹ ISO/IEC Guide 43 – 1: 1997: Proficiency testing by interlaboratory comparisons – Part 1: Development and operation of proficiency testing schemes

² ISO/IEC 17025: 2005 General requirements for establishing the competence of calibration and testing laboratories

³ ISO 5725 – 2 Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method

⁴ *ibid.*