

CORESTA Sub-Group - Physical Test Methods

Interlaboratory Proficiency Test 2010



Physical Test Methods Sub-Group

2010 Interlaboratory Proficiency Test

Cigarette and Filter Individual Weight, Diameter, Pressure Drop and Ventilation

Issued by:

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2010 CORESTA PTM 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, Diameter, 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.

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Data Analysis

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

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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Summary of Results

Filters

Laboratory A did not measure all filter samples.

Filter Individual Weights

Laboratory E reported significantly higher results for samples F3 and F4. No laboratory reported exceptional variability in results.

Filter Diameter

Laboratories B and D reported low and high results respectively for sample F2, but they did not exceed the hcrit value. No laboratory reported exceptional variability in results.

Filter Pressure Drop

Laboratory H did not measure any samples. Laboratory N did not measure sample F2. There were no exceptional results.

Cigarettes

Cigarette Individual Weights

Laboratory L did not measure cigarettes. Laboratory E reported significantly higher results for all cigarette samples. Laboratory F reported significantly higher results for sample C5.

No laboratory reported exceptional variability in results.

Cigarette Diameter

Laboratory D reported significantly higher results for cigarette C4. Laboratory A reported higher variability for samples C4 and C5.

Results for Laboratory E, cigarette C1, and Laboratory D, cigarettes C2, C3 and C4 gave questionable z-scores.

Cigarette Pressure Drop (Open)

Laboratory N reported significantly lower results for cigarettes C3, C4 and C5, and laboratory P reported significantly lower results for cigarette C1. The z-scores for these labs/ samples were questionable.

There were no outliers identified by the k-statistic plot or Cochran's test.

Cigarette Ventilation

Laboratory N reported significantly lower results for cigarette C2 and significantly higher results for cigarette C4. The z-scores for Laboratory N, cigarettes C2, C4 and C5 are questionable. The value for Lab P Cig C1 was extremely high (see Fig 25) and removed from the analysis.

The k-statistic plot shows exceptional variation for Laboratory N cigarette C2, which is reported as an outlier with Cochran's test.

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FILTER DATA ANALYSIS

Table 2. Filter Summary Statistics

| Variable | Filter_ID | N | Mean | StDev | CoefVar | Minimum | Maximum | Range |
|---------------------------|-----------|----|--------|-------|---------|---------|---------|-------|
| Individual Weight Mean mg | F2 | 39 | 462.8 | 16.31 | 3.52 | 454.2 | 561.0 | 106.8 |
| | F1 | 39 | 513.2 | 3.31 | 0.64 | 507.7 | 518.7 | 11.0 |
| | F4 | 39 | 797.0 | 3.75 | 0.47 | 789.0 | 805.0 | 16.0 |
| | F5 | 39 | 894.1 | 3.73 | 0.42 | 884.3 | 903.9 | 19.6 |
| | F3 | 39 | 1047.2 | 4.16 | 0.40 | 1041.5 | 1059.8 | 18.3 |
| Diameter Mean mm | F2 | 39 | 5.73 | 0.007 | 0.120 | 5.71 | 5.74 | 0.03 |
| | F1 | 39 | 6.76 | 0.006 | 0.090 | 6.75 | 6.77 | 0.02 |
| | F4 | 39 | 7.77 | 0.006 | 0.070 | 7.76 | 7.78 | 0.02 |
| | F5 | 39 | 7.78 | 0.006 | 0.080 | 7.77 | 7.80 | 0.03 |
| | F3 | 39 | 7.79 | 0.009 | 0.110 | 7.77 | 7.81 | 0.04 |
| Pressure Drop Mean mmWg | F4 | 36 | 242.7 | 2.72 | 1.12 | 238.0 | 249.0 | 11.0 |
| | F1 | 36 | 397.9 | 6.93 | 1.74 | 385.0 | 416.0 | 31.0 |
| | F2 | 33 | 416.2 | 4.37 | 1.05 | 409.0 | 425.9 | 16.9 |
| | F5 | 36 | 545.2 | 6.03 | 1.11 | 536.0 | 562.9 | 26.4 |
| | F3 | 36 | 799.3 | 7.57 | 0.95 | 786.0 | 820.0 | 34.0 |

Filter Rod Individual Weights

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 ID | MEAN | sr | sR | r | R |
|-----------|--------|------|------|------|------|
| F2 | 460.2 | 1.77 | 2.27 | 4.9 | 6.4 |
| F1 | 513.2 | 2.89 | 3.29 | 8.1 | 9.2 |
| F4 | 797.0 | 3.74 | 3.89 | 10.5 | 10.9 |
| F5 | 894.1 | 3.71 | 3.84 | 10.5 | 10.7 |
| F3 | 1047.2 | 3.13 | 3.98 | 8.8 | 11.1 |

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

Mandel Plots

The h-statistic Mandel Plots (Fig. 2) show that laboratory E is significantly different for samples F3 and F4.

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)

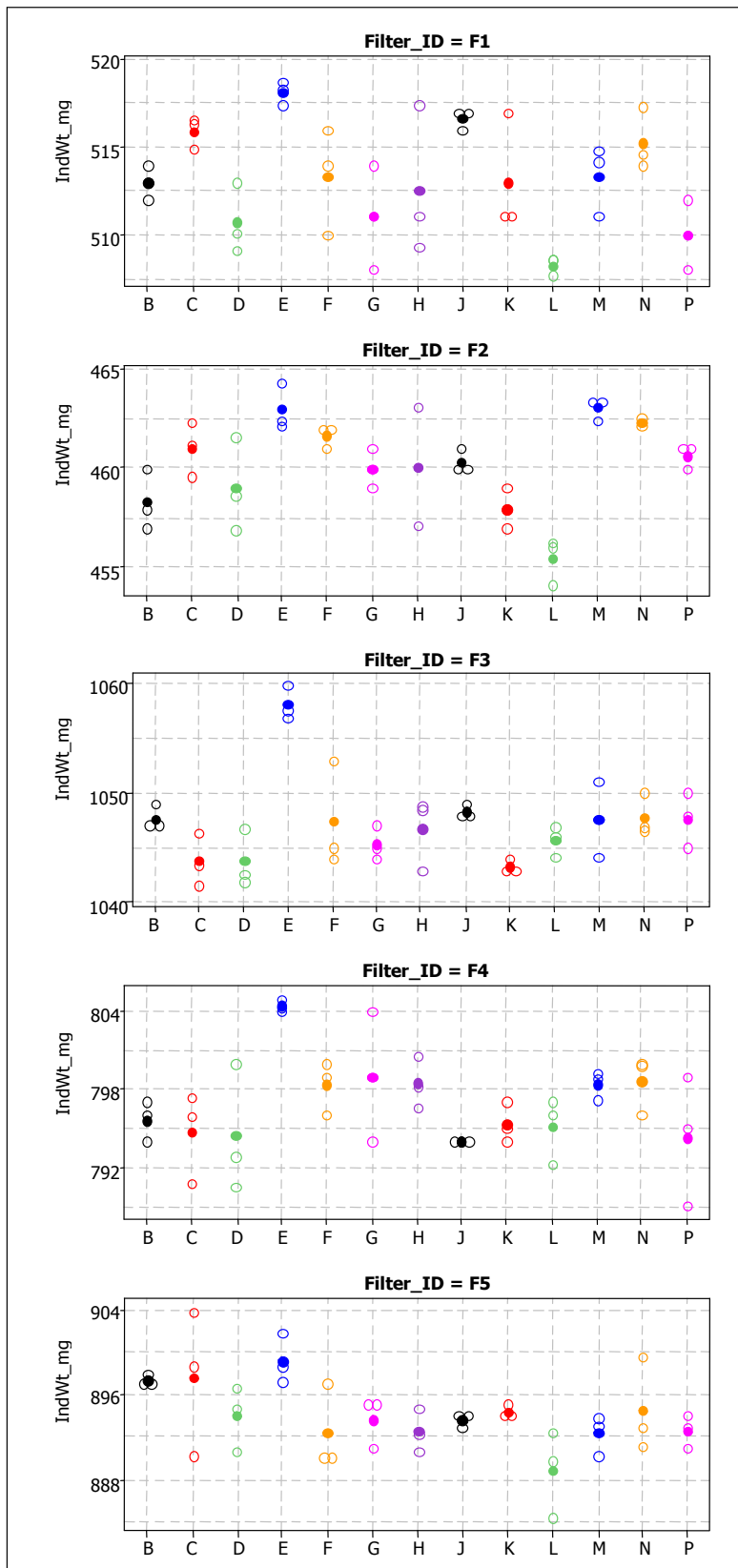
Results for Laboratory E, filters F3 and F4, and Laboratory L Filter F2 were questionable.

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Figure 1. Filter Rod Individual Weights – Individual Value Plots

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



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

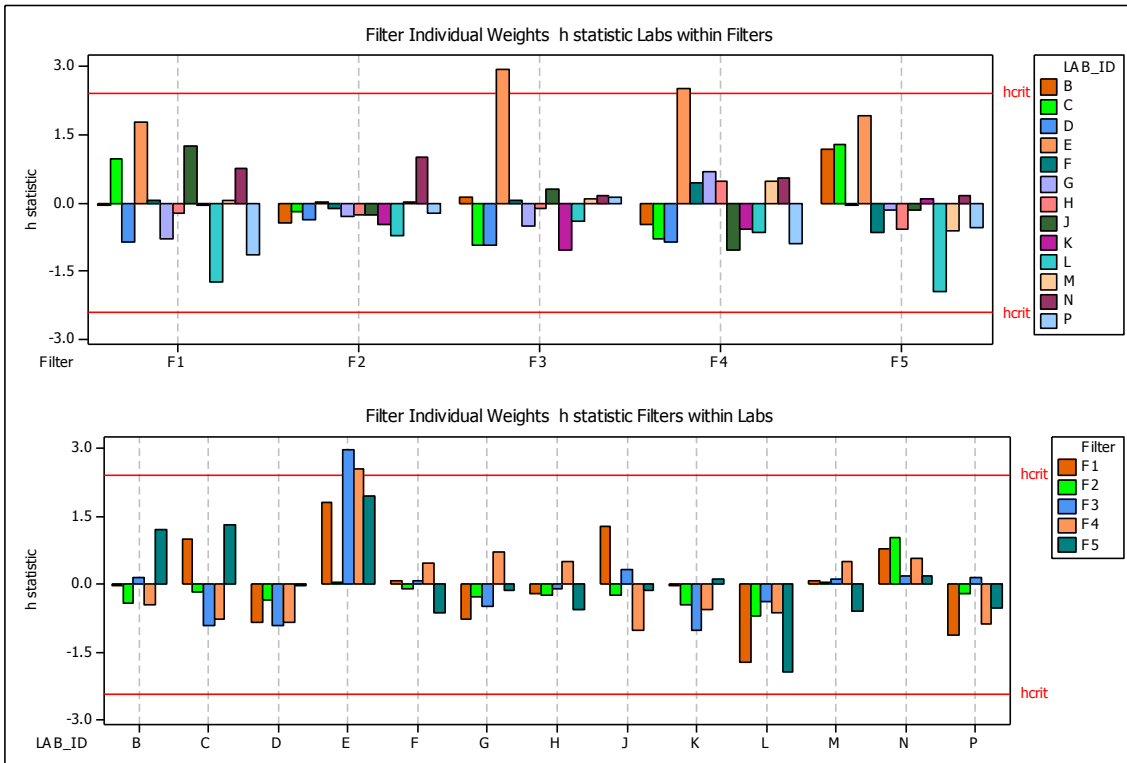
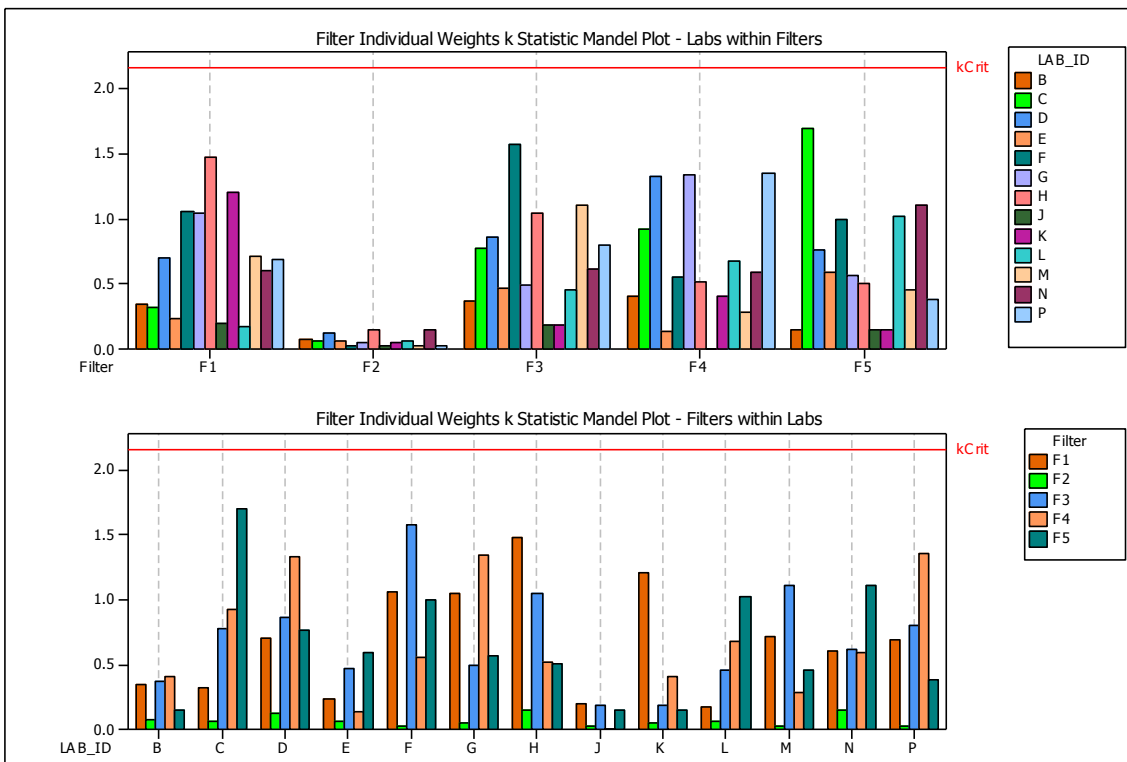


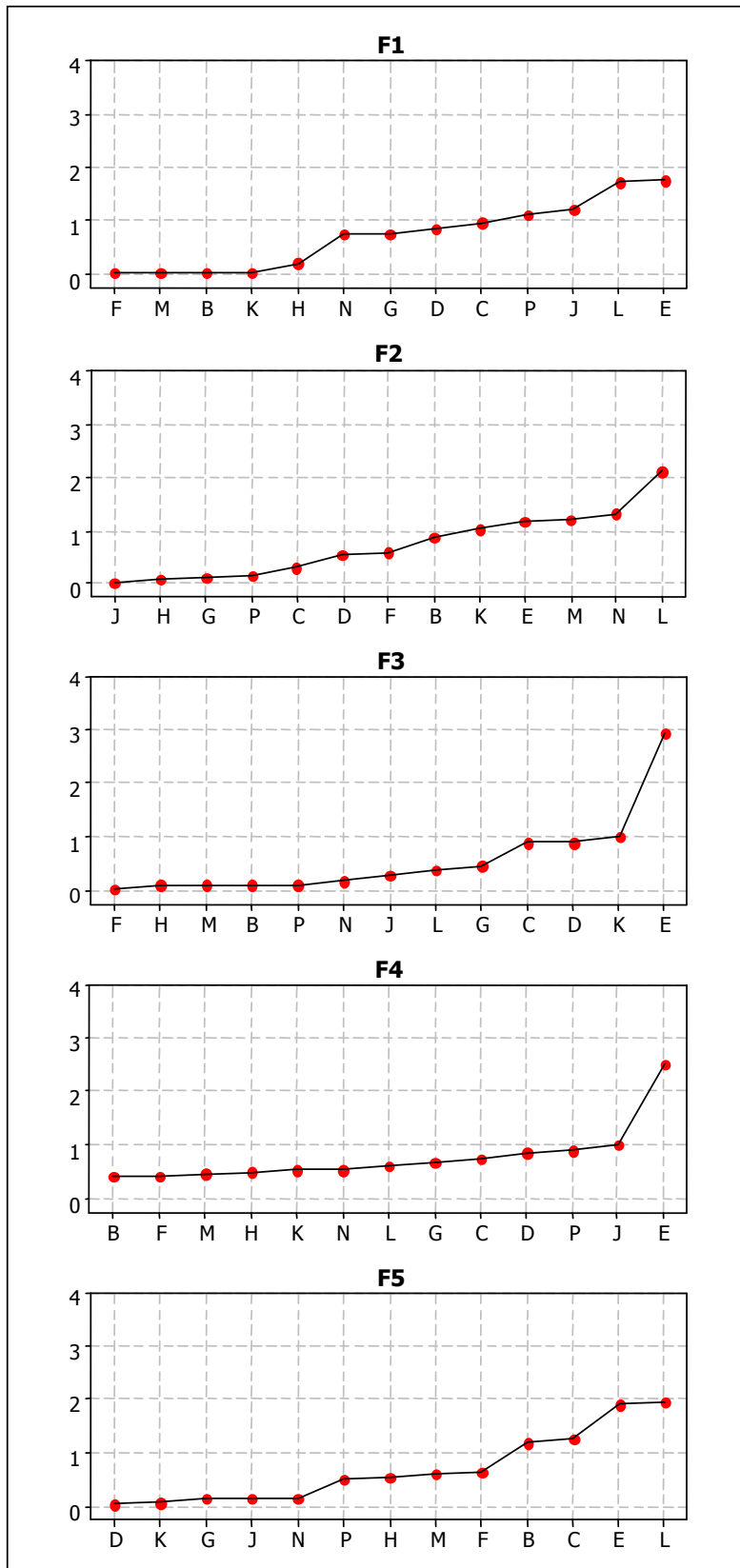
Figure 3. Filter Rod Individual Weights – k-statistic Mandel Plots



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



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Filter Diameter

Table 4. Filter Diameter Precision Statistics

Filter diameter data were screened for outliers and precision statistics calculated for each filter sample.

| FILTER ID | MEAN | sr | sR | r | R |
|------------------|-------------|-----------|-----------|----------|----------|
| F2 | 5.73 | 0.006 | 0.007 | 0.016 | 0.019 |
| F1 | 6.76 | 0.005 | 0.006 | 0.014 | 0.016 |
| F4 | 7.72 | 0.006 | 0.006 | 0.017 | 0.017 |
| F3 | 7.78 | 0.007 | 0.007 | 0.018 | 0.019 |
| F5 | 7.79 | 0.008 | 0.009 | 0.023 | 0.024 |

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

Mandel Plots

Mandel plots do not show any laboratories beyond critical values.

Repeatability Variation

Cochran's test did not identify any outliers.

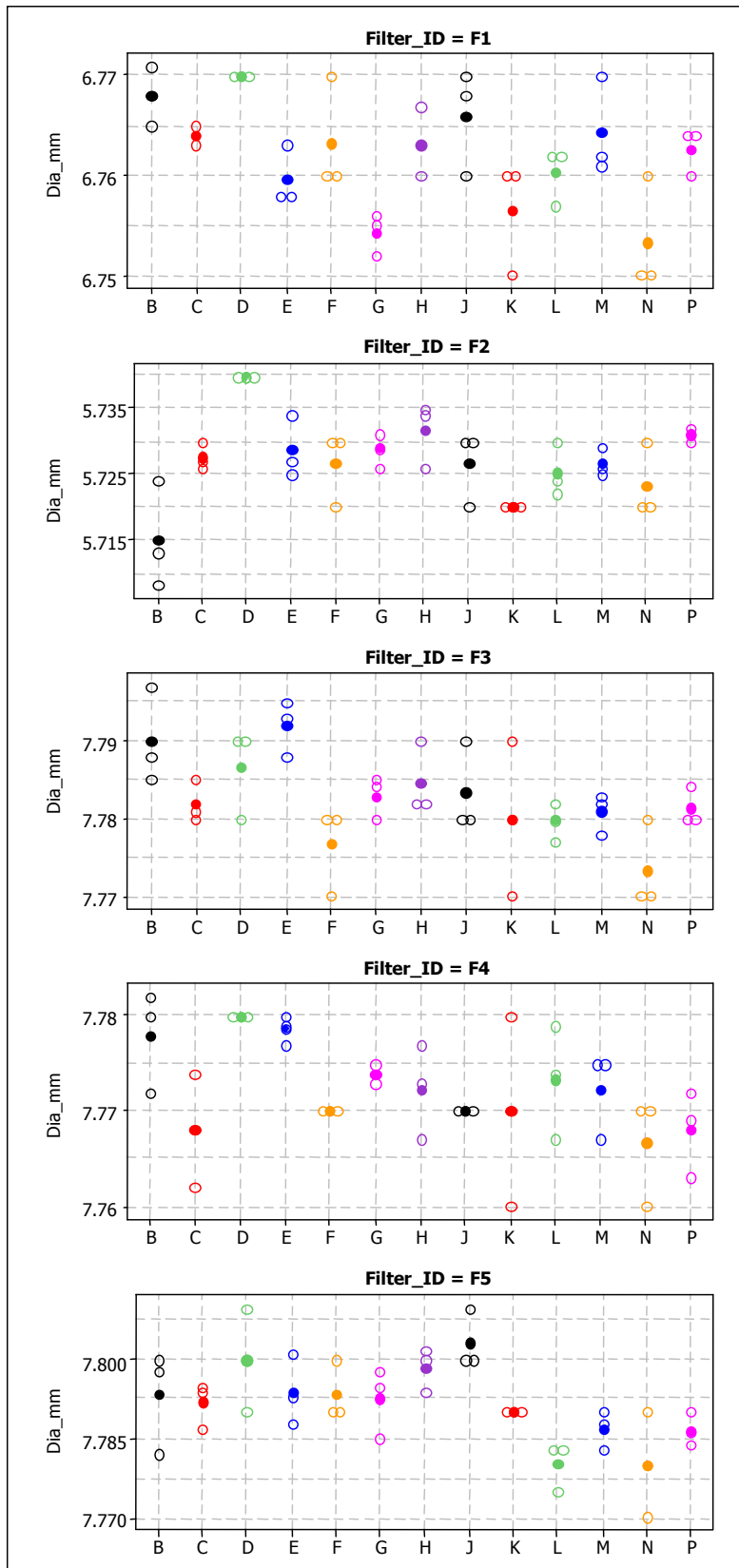
Z-scores

Laboratories B and D were questionable for sample F2, but were not beyond the h_{crit} values on the Mandel plot.

Laboratory A did not measure the samples.

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Figure 5. Filter Rod Diameter – Individual Value Plots



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Figure 6. Filter Rod Diameter: *h*-statistic Mandel Plots

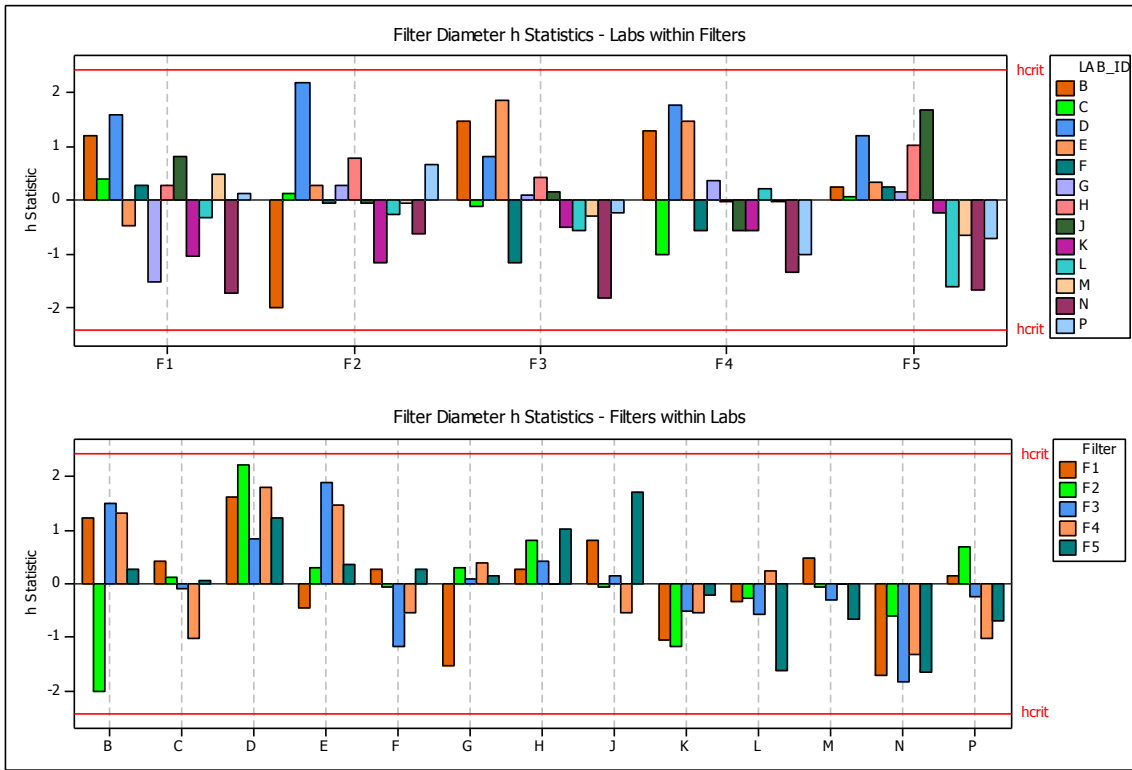
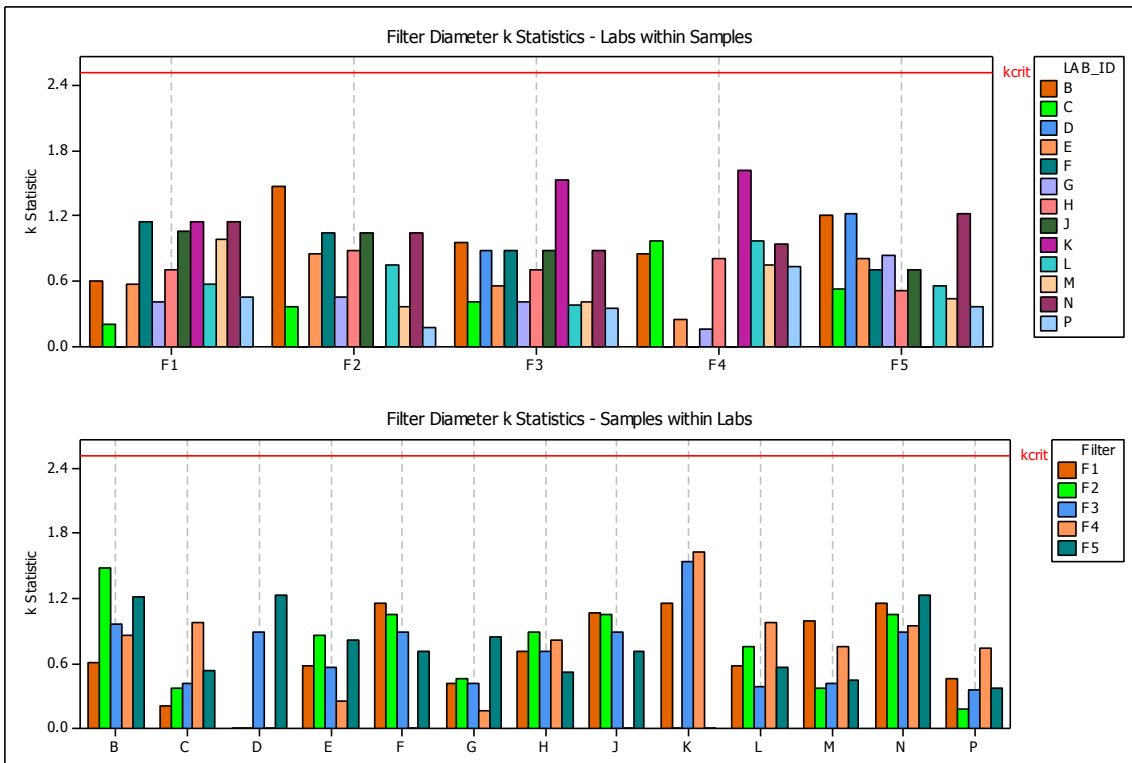


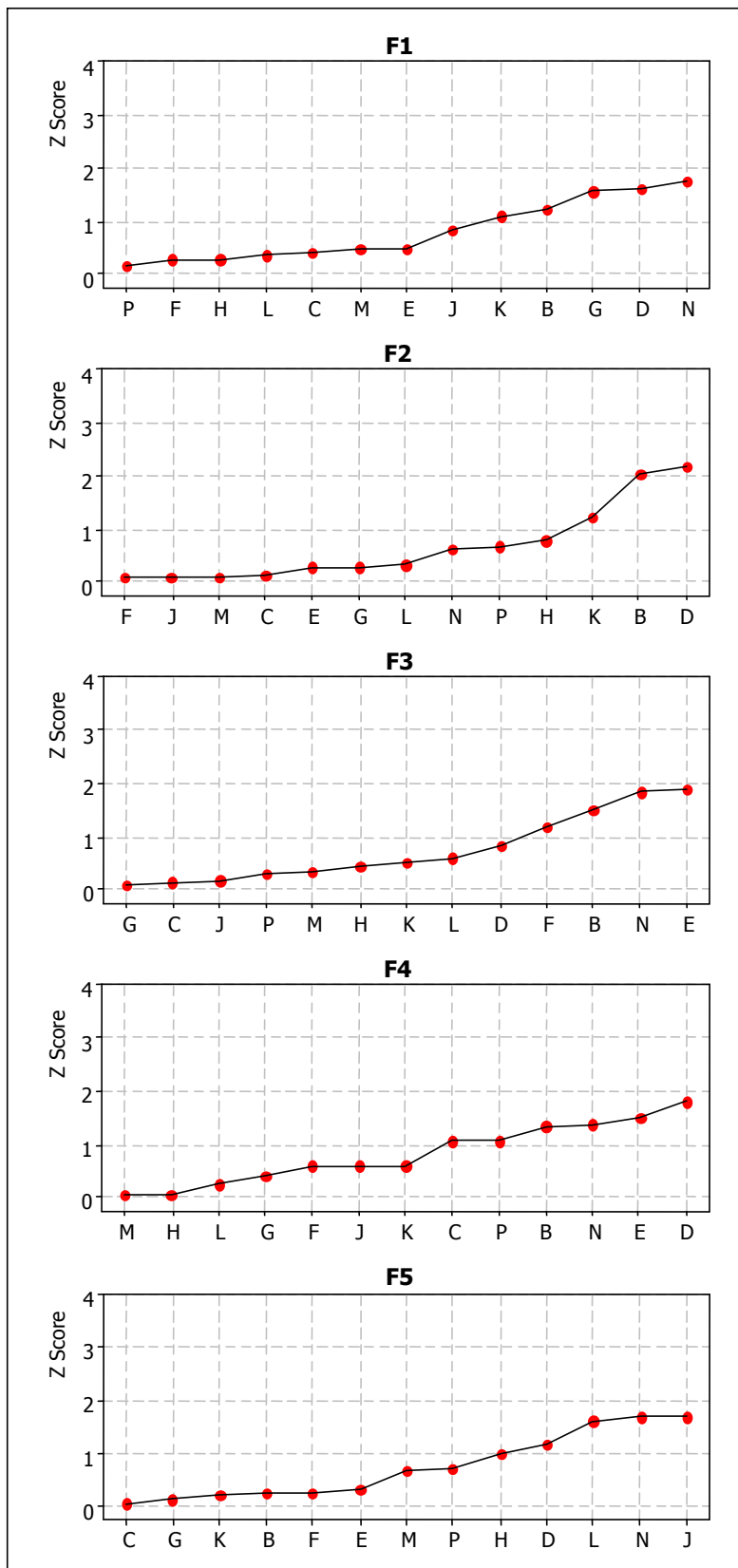
Figure 7. Filter Rod Diameter: *k*-statistic Mandel Plots



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Figure 8. Filter Rod Diameter – Z-Score Plots



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FILTER ROD PRESSURE DROP

Table 5. Filter Rod Pressure Drop Precision Statistics

Laboratory H did not measure any samples, Laboratory N did not measure sample F2.

| FILTER ID | MEAN | Sr | sR | r | R |
|-----------|-------|------|------|------|------|
| F4 | 242.7 | 2.57 | 2.74 | 7.2 | 7.7 |
| F1 | 397.9 | 2.96 | 6.08 | 8.3 | 17.0 |
| F2 | 416.2 | 3.22 | 4.10 | 9.0 | 11.5 |
| F5 | 545.2 | 4.89 | 5.82 | 13.7 | 16.3 |
| F3 | 799.3 | 4.06 | 6.80 | 11.4 | 19.0 |

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

Mandel Plots

There were no results outside the h_{crit} or k_{crit} limits.

Repeatability Variation

Cochran's test did not identify any outliers.

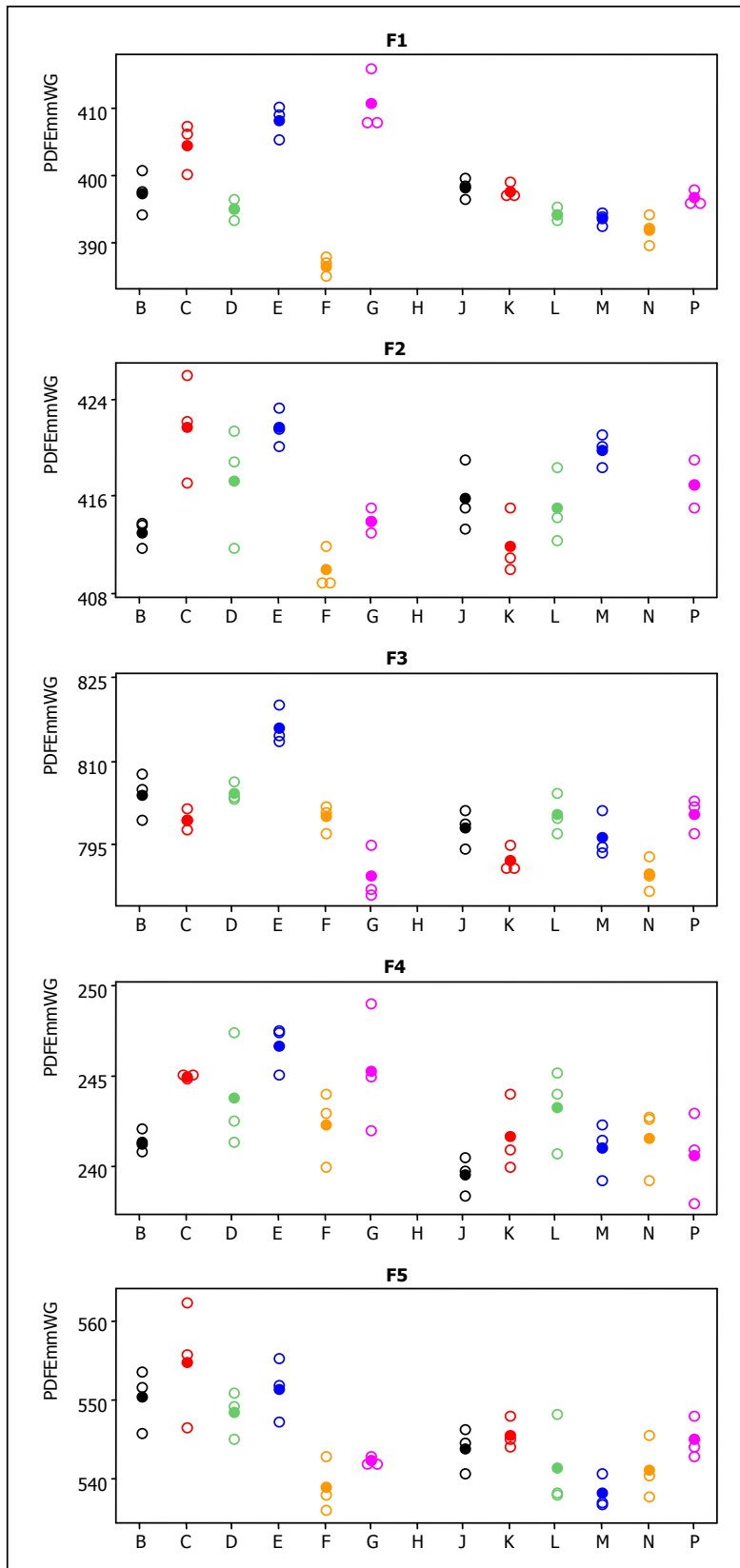
Z-scores

Laboratories with missing data points did not, or were unable to, measure the samples. Laboratory E, sample F3 was identified as questionable, but did not exceed h_{crit} .

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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

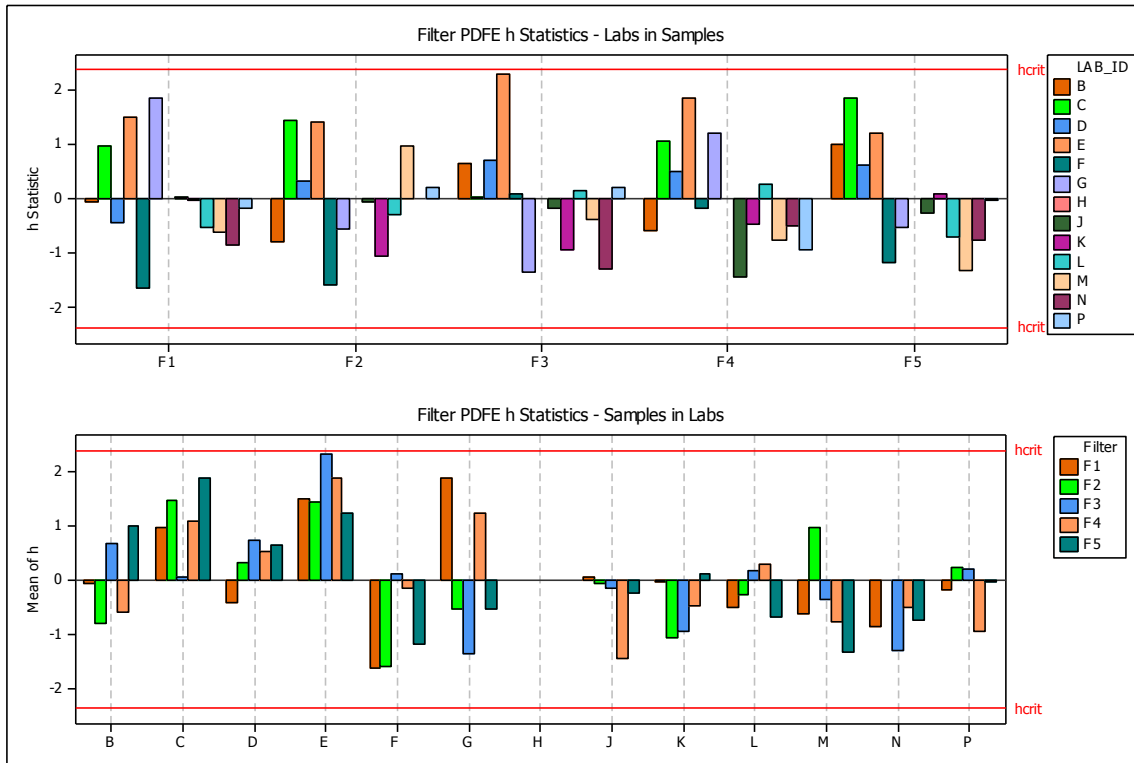
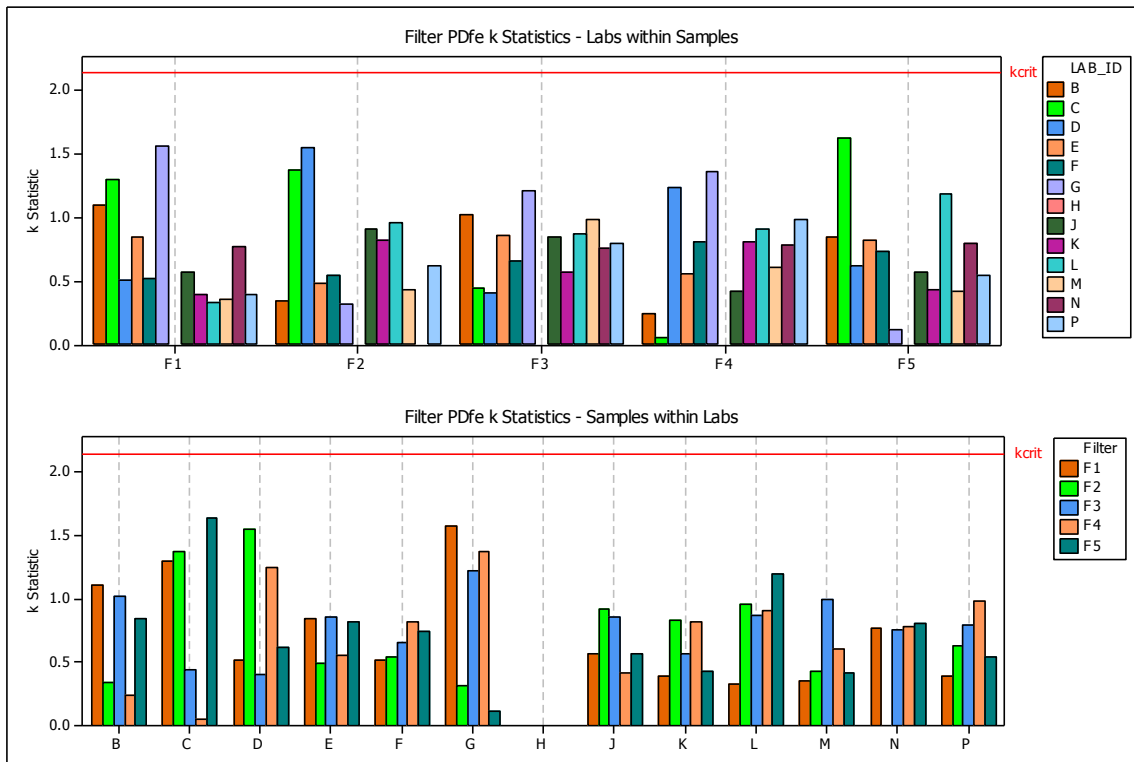


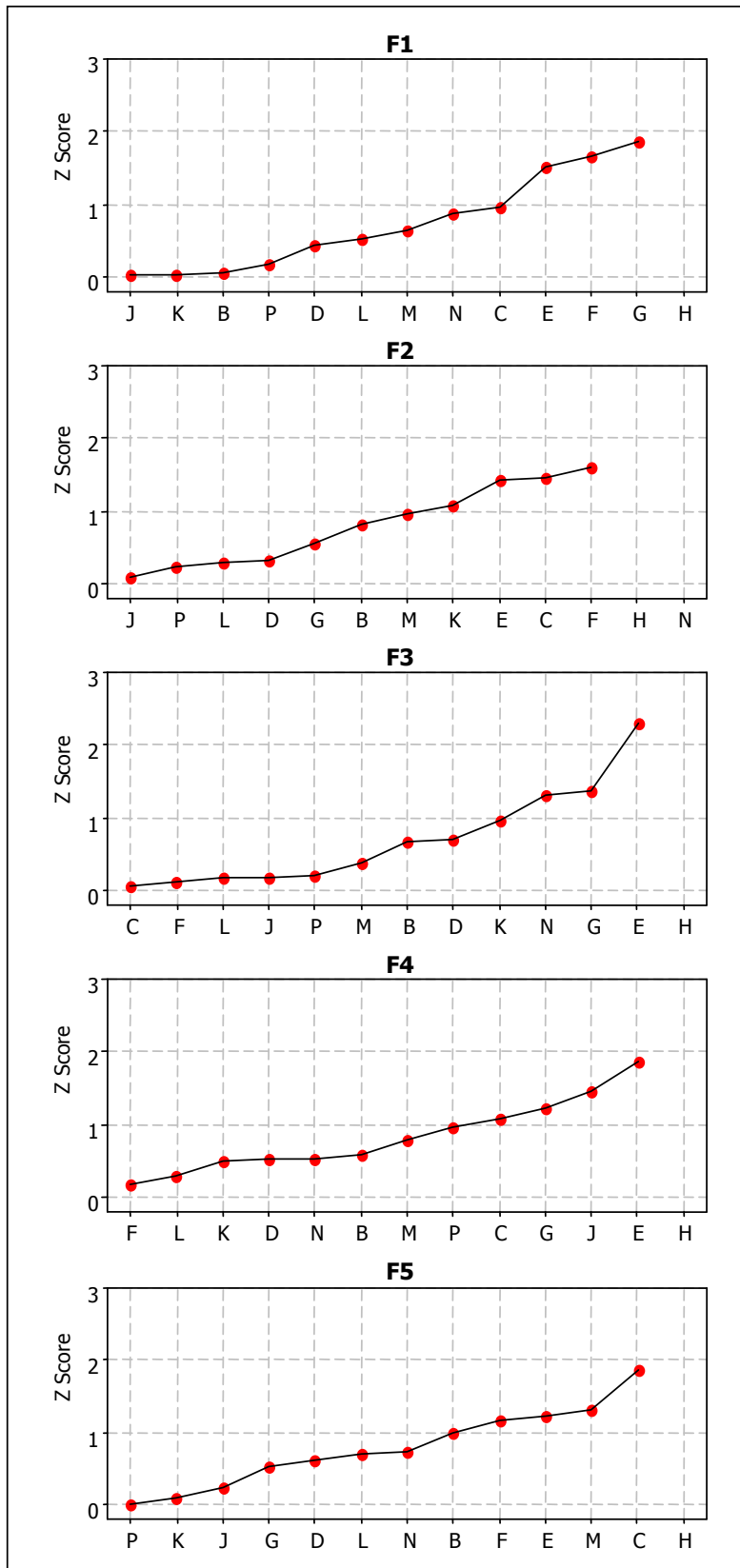
Figure 11. Filter Rod Pressure Drop: *k*-statistic Mandel Plots



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Figure 12. Filter Rod Pressure Drop: Z-Score Plots



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CIGARETTE DATA ANALYSIS

Table 6. Cigarette Summary Statistics

| Variable | Cig_ID | N | N* | Mean | StDev | CoefVar | Minimum | Maximum |
|------------------------------|--------|----|----|--------|-------|---------|---------|---------|
| Individual Weight Mean mg | C1 | 39 | 3 | 951.5 | 15.63 | 1.64 | 924.0 | 1005.0 |
| | C2 | 39 | 3 | 954.1 | 13.88 | 1.45 | 927.0 | 998.0 |
| | C3 | 39 | 3 | 716.8 | 9.40 | 1.31 | 702.9 | 744.0 |
| | C4 | 39 | 3 | 663.6 | 9.66 | 1.46 | 651.0 | 694.0 |
| | C5 | 39 | 3 | 661.1 | 10.83 | 1.64 | 645.8 | 695.0 |
| Diameter Mean mm | C1 | 39 | 3 | 7.93 | 0.019 | 0.250 | 7.88 | 7.98 |
| | C2 | 39 | 3 | 7.94 | 0.017 | 0.210 | 7.88 | 7.97 |
| | C3 | 39 | 3 | 6.84 | 0.019 | 0.270 | 6.79 | 6.87 |
| | C4 | 39 | 3 | 5.74 | 0.021 | 0.360 | 5.67 | 5.71 |
| | C5 | 39 | 3 | 5.73 | 0.025 | 0.430 | 5.65 | 5.83 |
| Pressure Drop open Mean mmWG | C1 | 39 | 3 | 117.12 | 3.180 | 2.710 | 105.00 | 123.60 |
| | C2 | 39 | 3 | 108.10 | 1.940 | 1.790 | 104.90 | 114.60 |
| | C3 | 39 | 3 | 117.63 | 2.060 | 1.750 | 110.90 | 121.00 |
| | C4 | 39 | 3 | 176.45 | 6.160 | 3.490 | 153.00 | 189.00 |
| | C5 | 39 | 3 | 87.35 | 2.607 | 2.980 | 78.60 | 93.20 |
| Vent Mean % | C1 | 39 | 3 | 19.92 | 3.815 | 19.150 | 17.70 | 35.90 |
| | C2 | 39 | 3 | 27.54 | 1.550 | 5.630 | 18.99 | 29.25 |
| | C3 | 39 | 3 | 41.80 | 0.893 | 2.140 | 39.80 | 43.14 |
| | C4 | 39 | 3 | 45.13 | 1.852 | 4.100 | 42.60 | 50.50 |
| | C5 | 39 | 3 | 81.61 | 1.015 | 1.240 | 79.90 | 84.51 |

CIGARETTE INDIVIDUAL WEIGHTS

Table 7. Cigarette Individual Weights – Precision Statistics

Laboratory L did not measure cigarettes. Lab E is excluded.

| CIGARETTE ID | MEAN | sr | sR | r | R |
|--------------|-------|------|------|------|------|
| C5 | 658.6 | 6.68 | 6.68 | 18.7 | 18.7 |
| C4 | 661.4 | 5.96 | 6.00 | 16.7 | 16.8 |
| C3 | 714.6 | 6.23 | 6.23 | 17.4 | 17.4 |
| C1 | 947.6 | 8.52 | 8.52 | 23.9 | 23.9 |
| C2 | 950.9 | 9.44 | 9.44 | 26.2 | 26.4 |

Note R values for 4 of the samples are the same as r, this is due to the within lab variation being much greater than the between lab variation.

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) show that Laboratory E is significantly different for all cigarettes, and distorts the appearance of the plot.

Fig 14b is the Mandel Plot redrawn, without Laboratory E. Laboratory F Sample C5 is above the hcrit value.

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

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Repeatability Variation

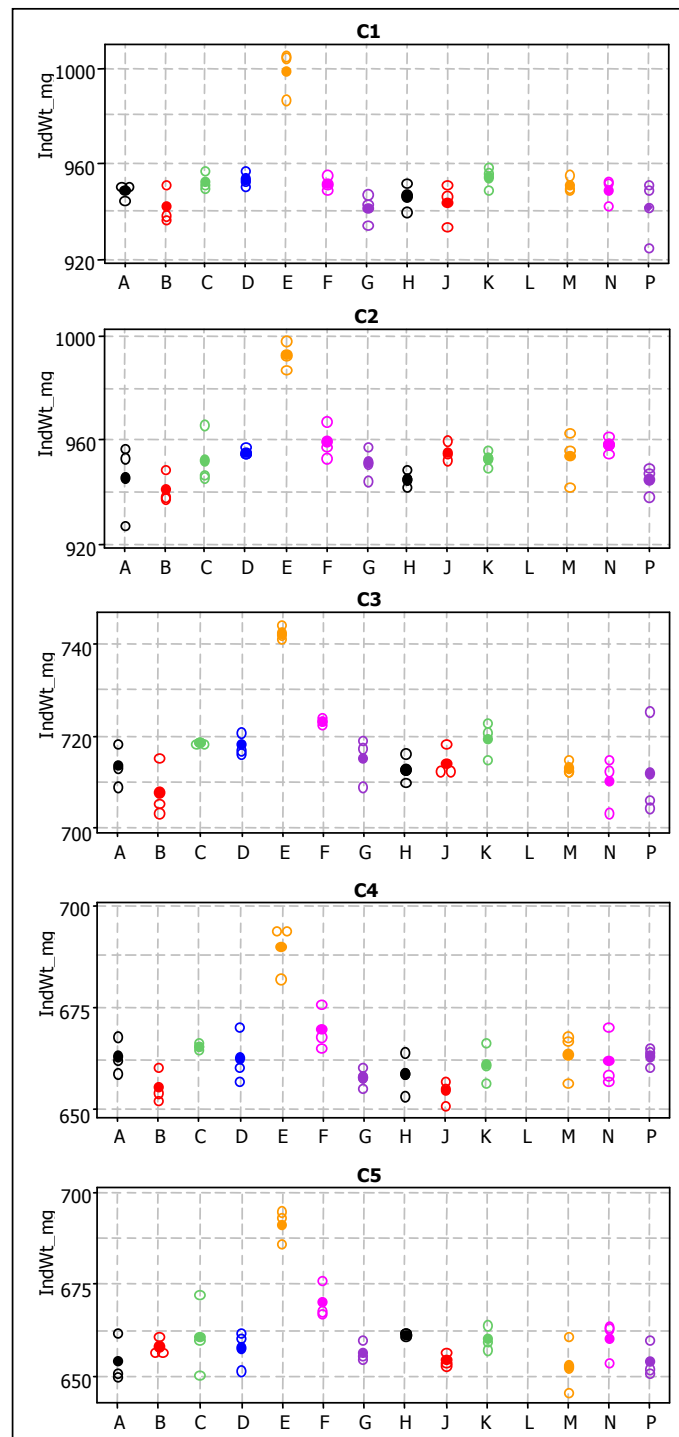
Cochran's test shows repeatability variation is acceptable for all laboratories.

Z-scores

Results for Laboratory E distort the z-score plot (Fig. 16), The plot is redrawn (Fig 16a) without Laboratory E and shows Laboratory F, sample C5 as questionable.

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

Figure 13. Cigarette Individual Weights – Individual Value Plot



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Figure 14. Cigarette Individual Weights: *h*-statistic Mandel Plots

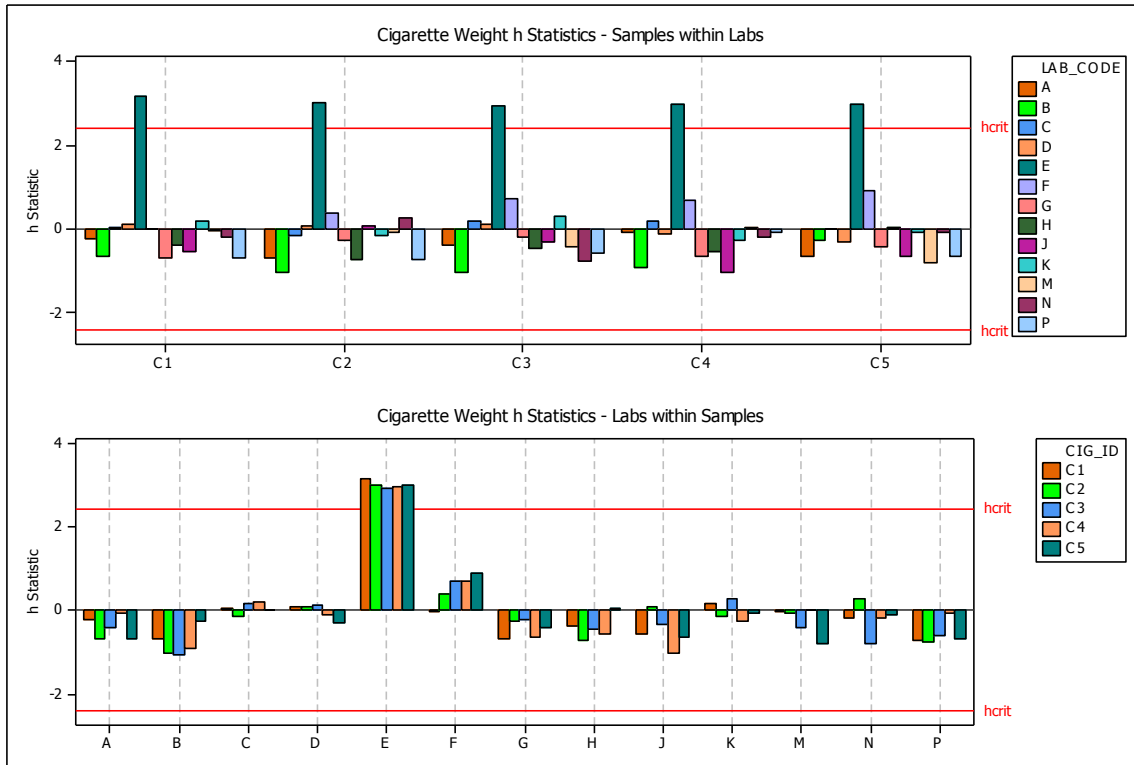
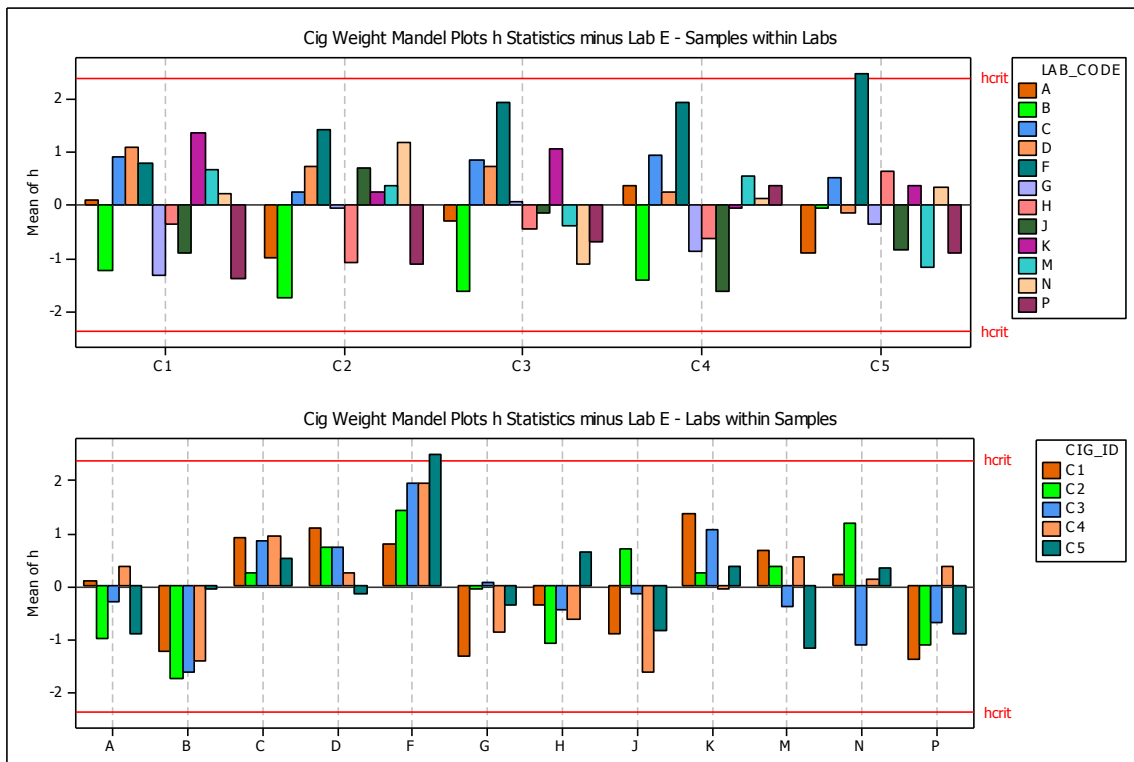
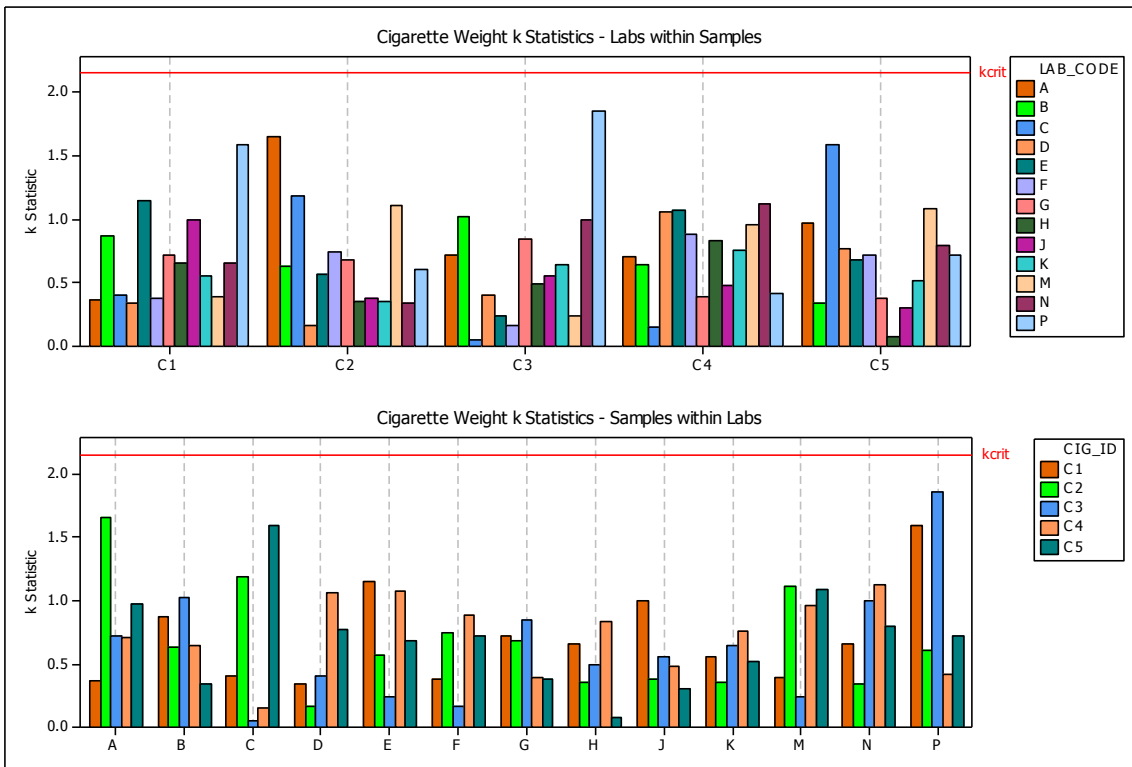


Figure 14b. Cigarette Individual Weights: *h*-statistic Mandel Plots with Laboratory E excluded



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



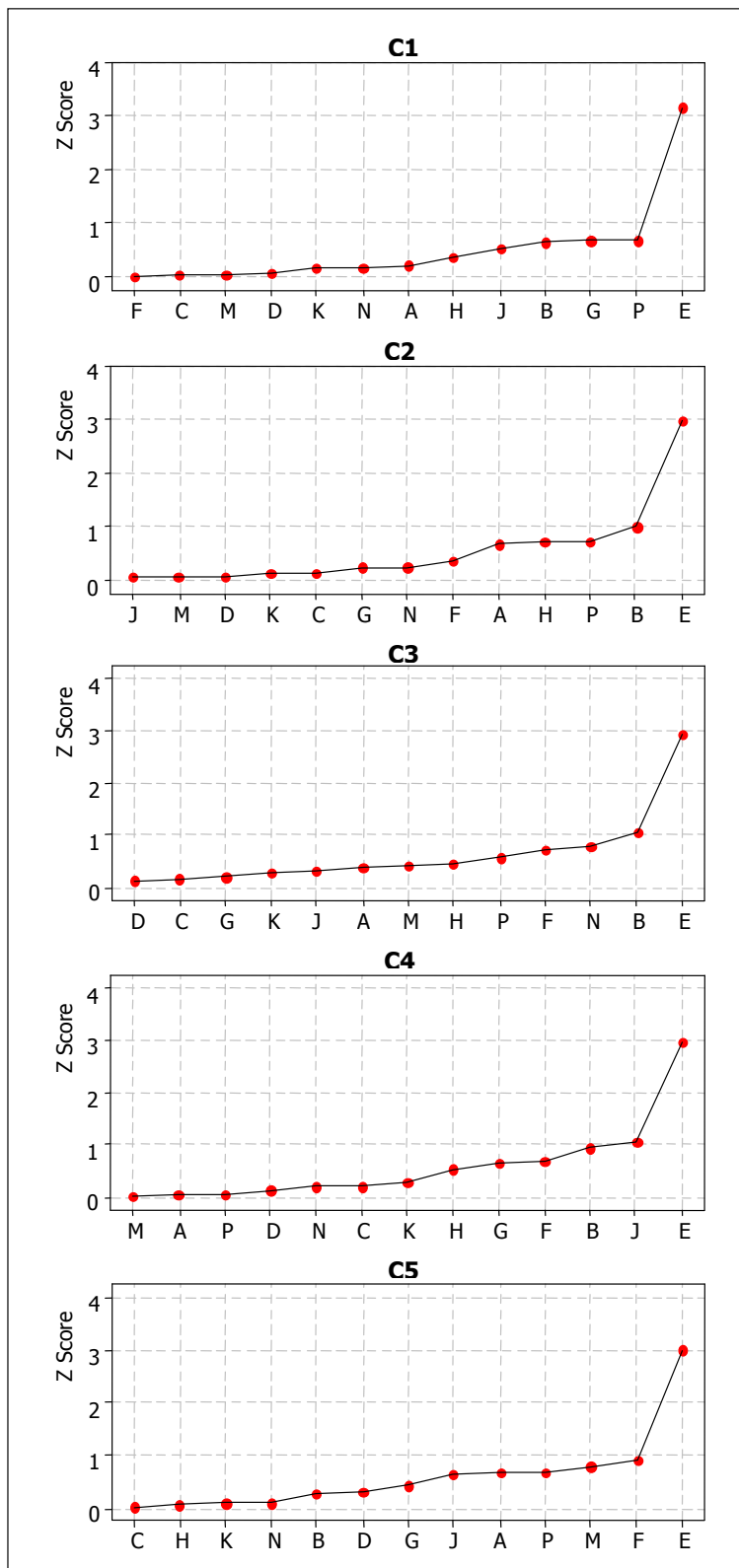
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Cigarette Individual Weights; Repeatability Variation

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

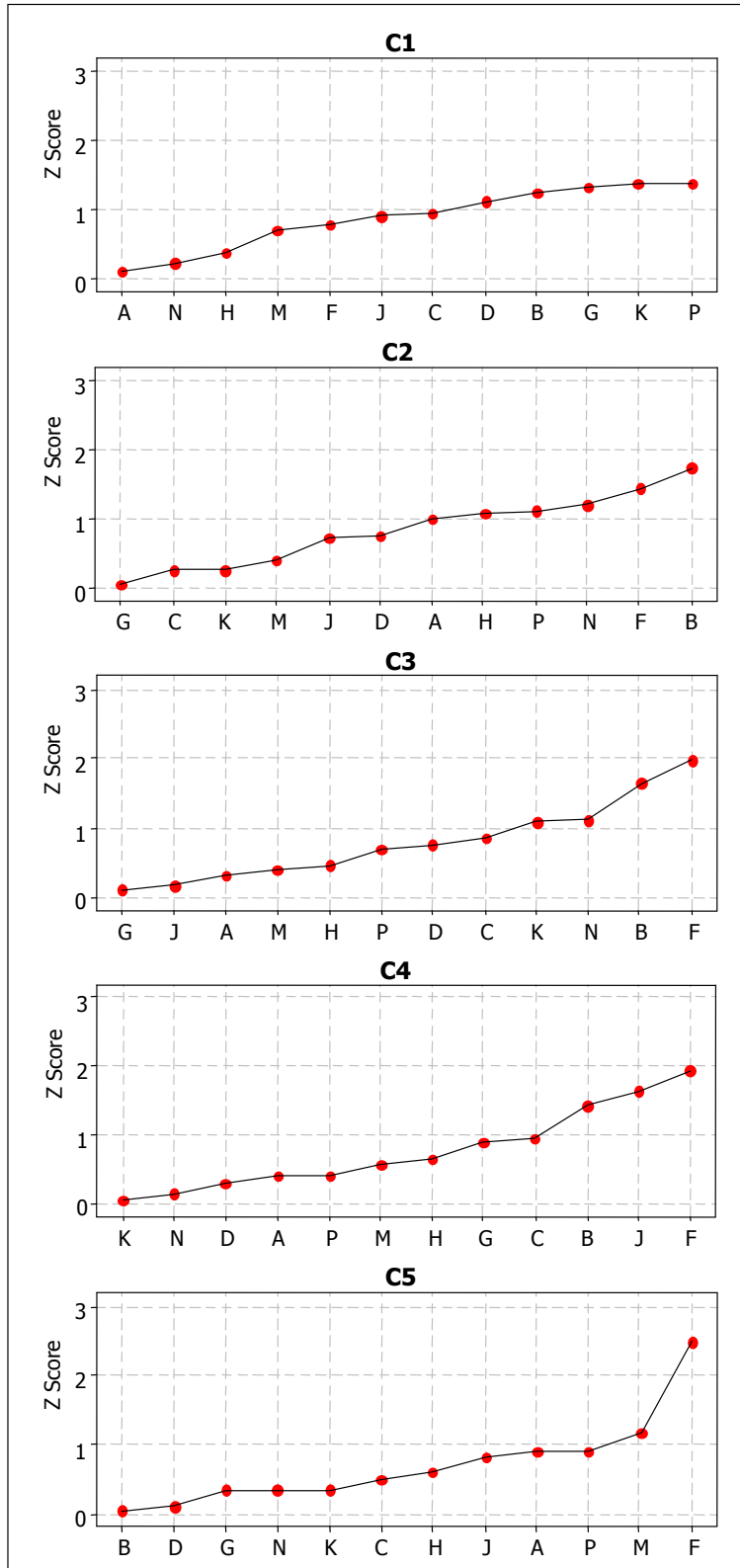
Figure 16. Cigarette Individual Weights: Z-Score Plots



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Figure 16a. Cigarette Individual Weights: Z-Score Plots – Lab E excluded



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CIGARETTE DIAMETER

Table 8. Cigarette Diameter Precision Statistics

| CIGARETTE ID | MEAN | sr | sR | r | R |
|--------------|------|-------|-------|-------|-------|
| C5 | 5.73 | 0.036 | 0.036 | 0.101 | 0.101 |
| C4 | 5.74 | 0.018 | 0.020 | 0.051 | 0.057 |
| C3 | 6.84 | 0.010 | 0.017 | 0.028 | 0.047 |
| C1 | 7.93 | 0.017 | 0.019 | 0.048 | 0.054 |
| C2 | 7.94 | 0.014 | 0.017 | 0.040 | 0.047 |

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

Laboratory D reported significantly higher results for sample C4. Laboratory A reported higher variability for samples C4 and C5.

Repeatability Variation

Cochran's test shows Laboratory A cigarette C4 and C5 and Laboratory D cigarette C2 as outliers.

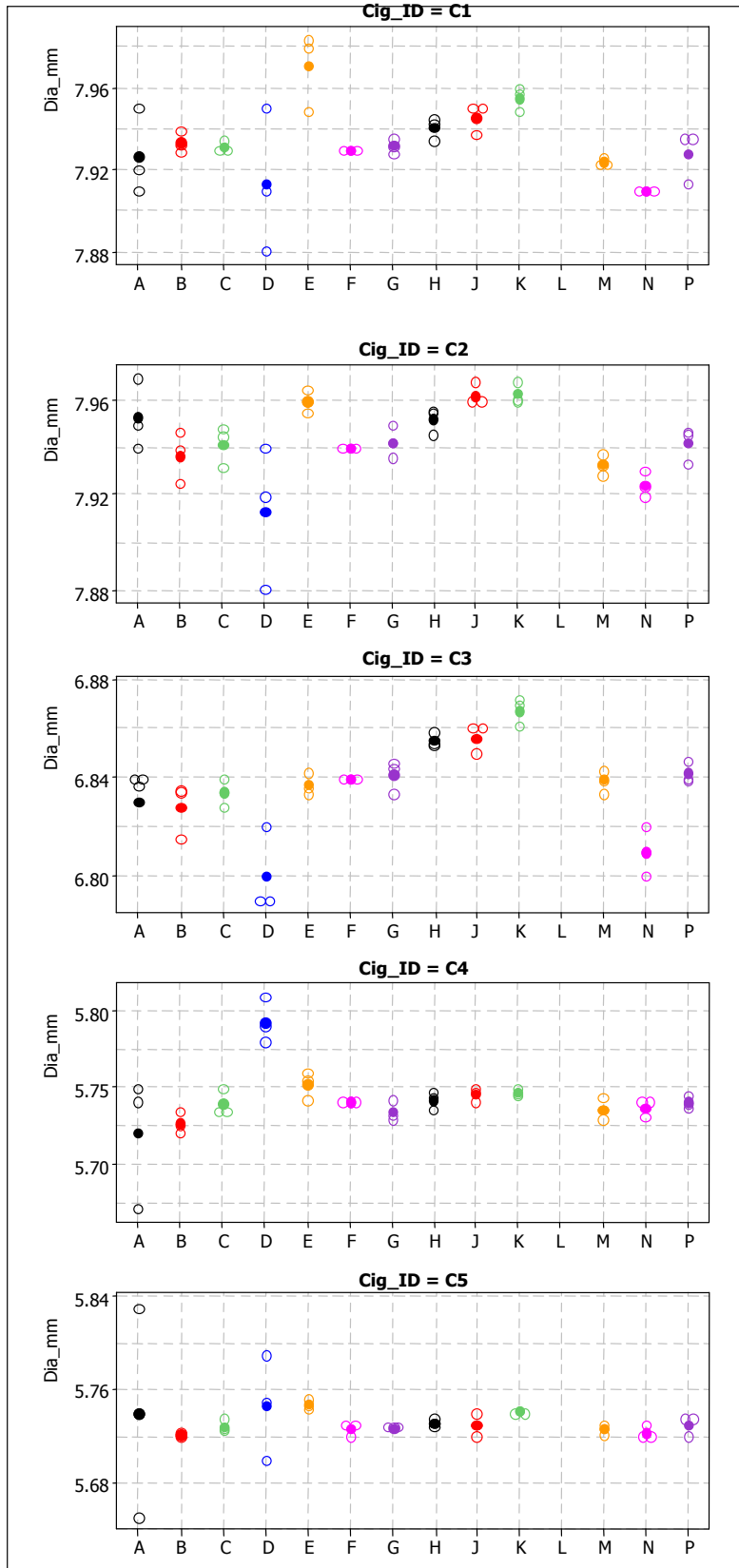
Z-scores

Results for Laboratory E, Cigarettes C1 (Fig. 20) are questionable as are results for Laboratory D, cigarettes C2, C3 and C4.

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

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Figure 17. Cigarette Diameter Individual Value Plots



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Figure 18. Cigarette Diameter: *h*-statistics Mandel Plots

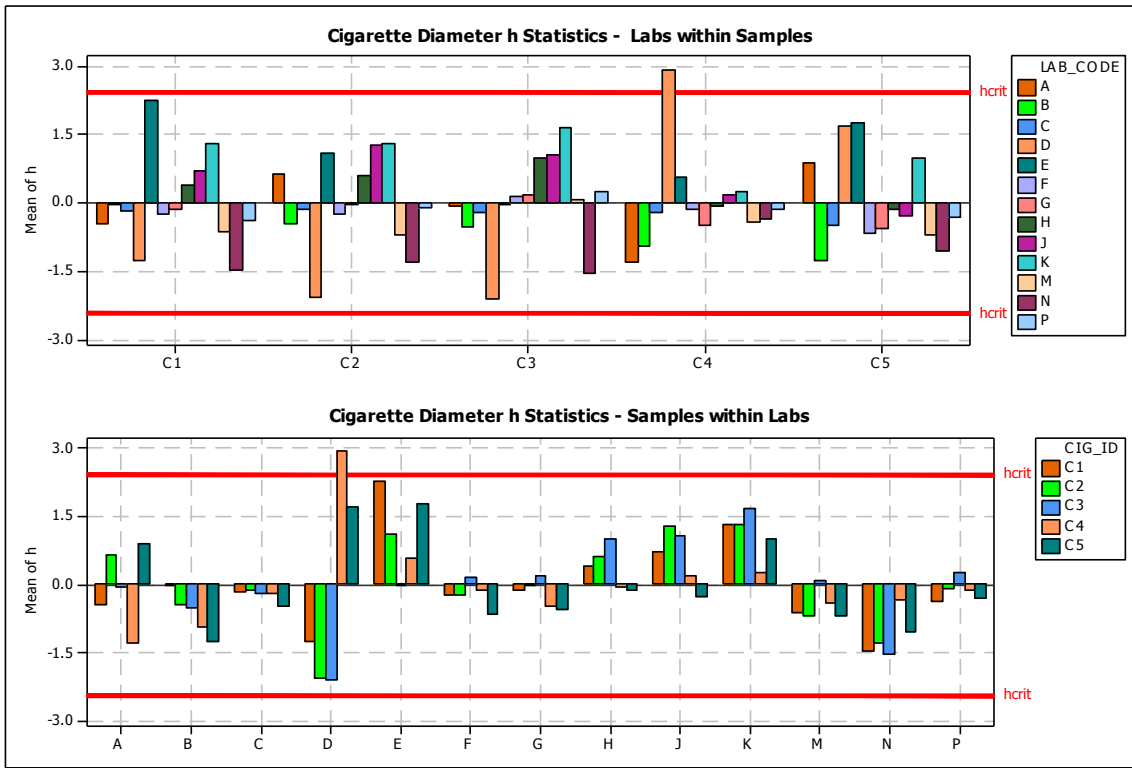
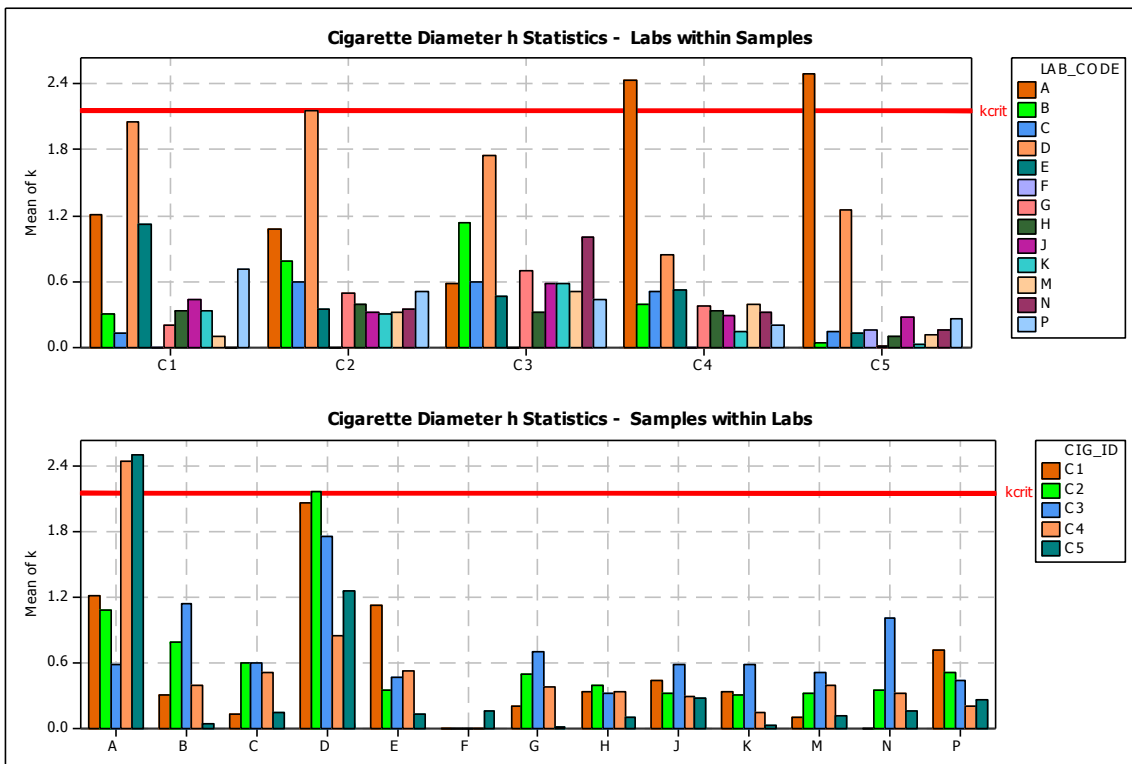


Figure 19. Cigarette Diameter: *k*-statistics Mandel Plots



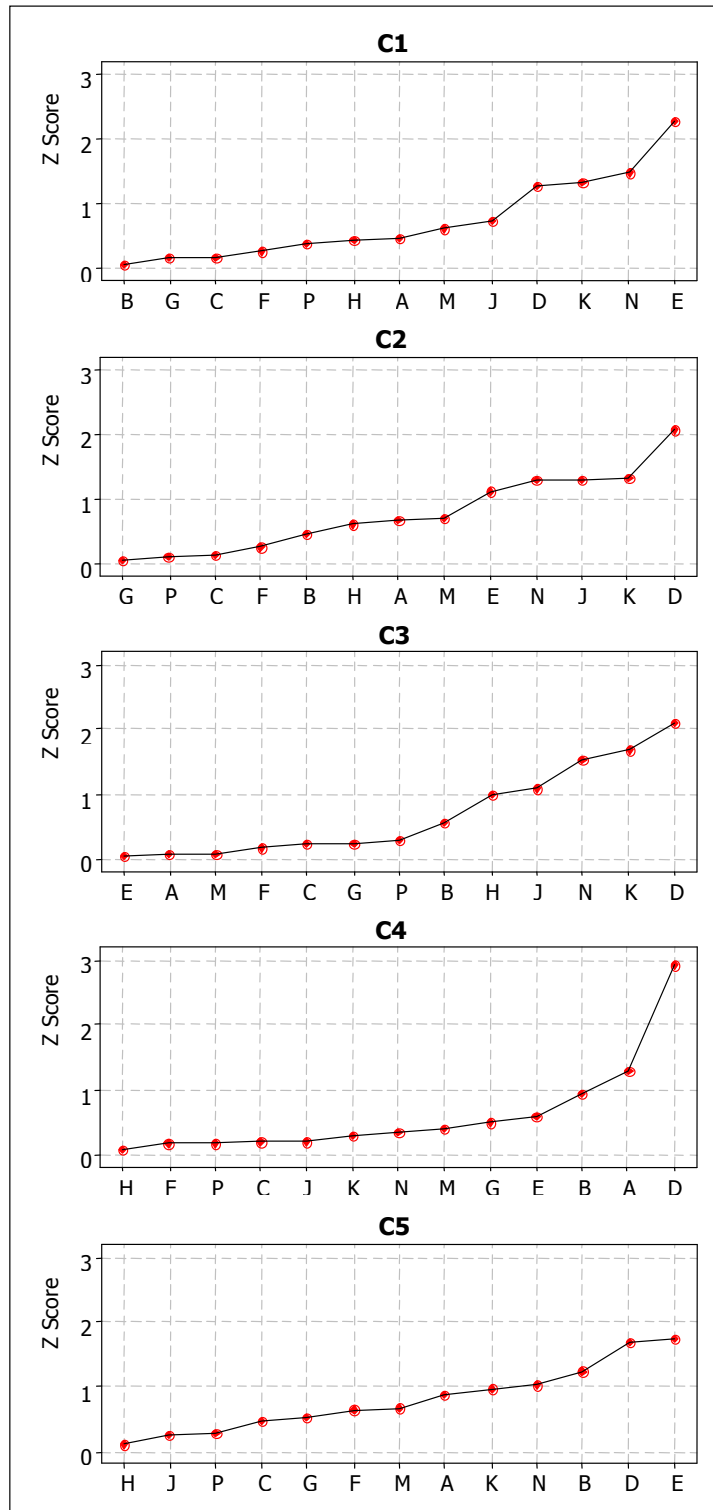
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Cigarette Diameter – Repeatability Variation

Cochran's test highlighted Lab A samples C4 and C5 as outliers and Lab D sample C2 as a straggler.

Figure 20. Cigarette Diameter: Z-Score Plots Plots



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CIGARETTE PRESSURE DROP (OPEN)

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

| CIGARETTE ID | MEAN | sr | sR | r | R |
|--------------|-------|------|------|-------|-------|
| C5 | 87.4 | 1.78 | 2.44 | 4.98 | 6.83 |
| C2 | 101.8 | 2.69 | 2.69 | 7.52 | 7.52 |
| C1 | 117.2 | 2.02 | 2.94 | 5.66 | 8.22 |
| C3 | 117.6 | 2.16 | 2.17 | 6.04 | 6.07 |
| C4 | 176.5 | 5.46 | 6.15 | 15.29 | 17.21 |

Individual value plots for each sample are shown in Fig. 21. Individual data points are represented by open circles, and laboratory means by solid circles.

Mandel Plots

The h-statistic Mandel Plots (Fig. 22) shows that Laboratory N is significantly different from the reference value for cigarettes C3, C4 and C5. Laboratory P, cigarette C1 is significantly different from the reference value.

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

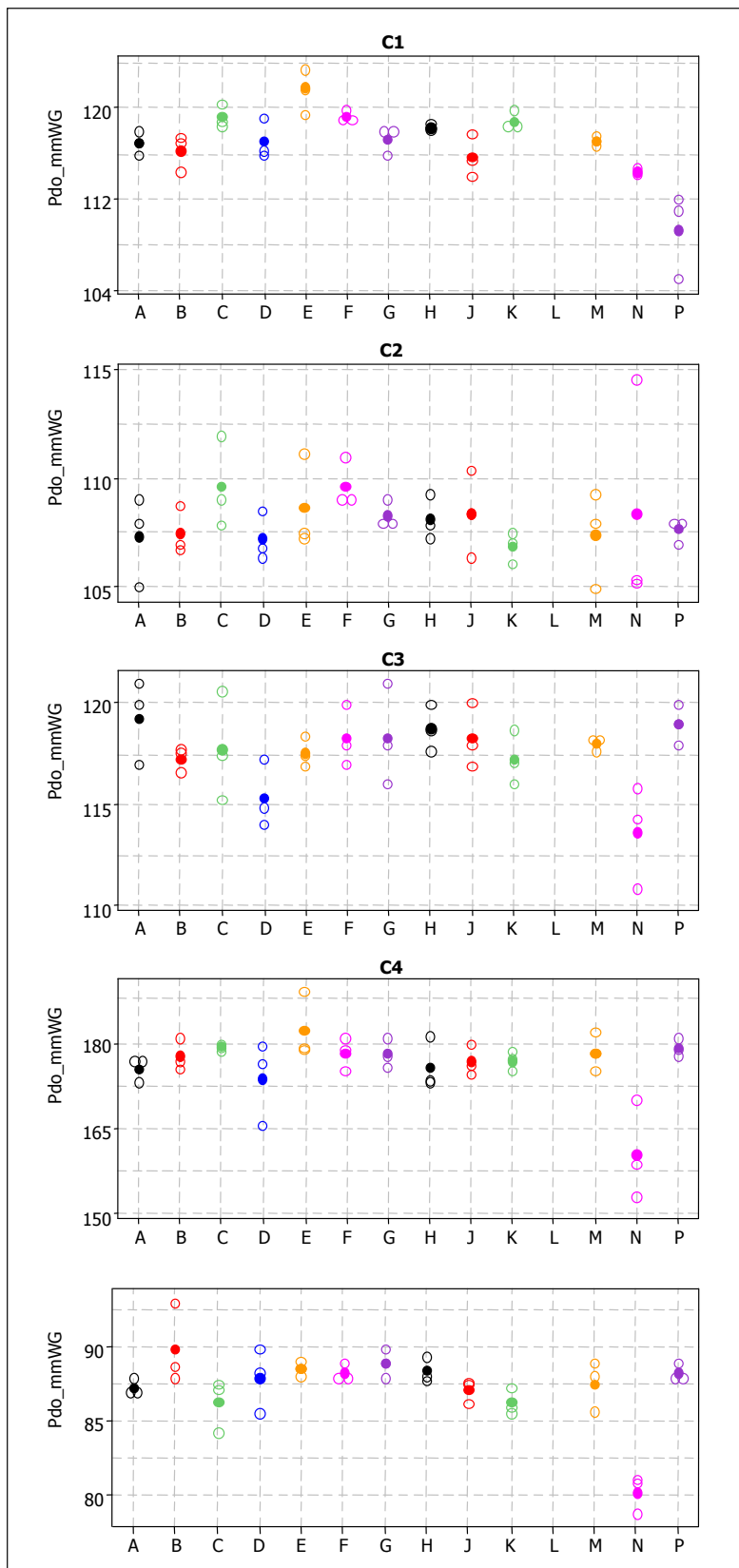
Laboratory N gave questionable results for cigarettes C3, C4 and C5 (Fig. 24). Laboratory P gave questionable results for cigarette C1.

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



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

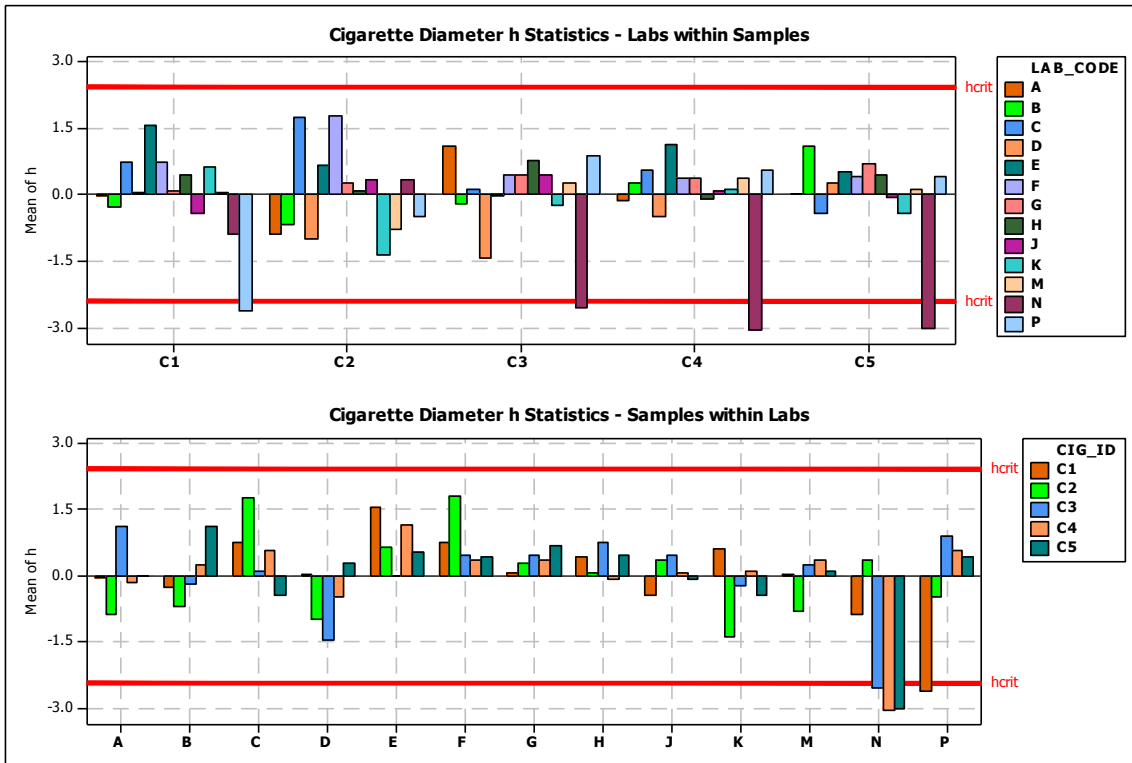
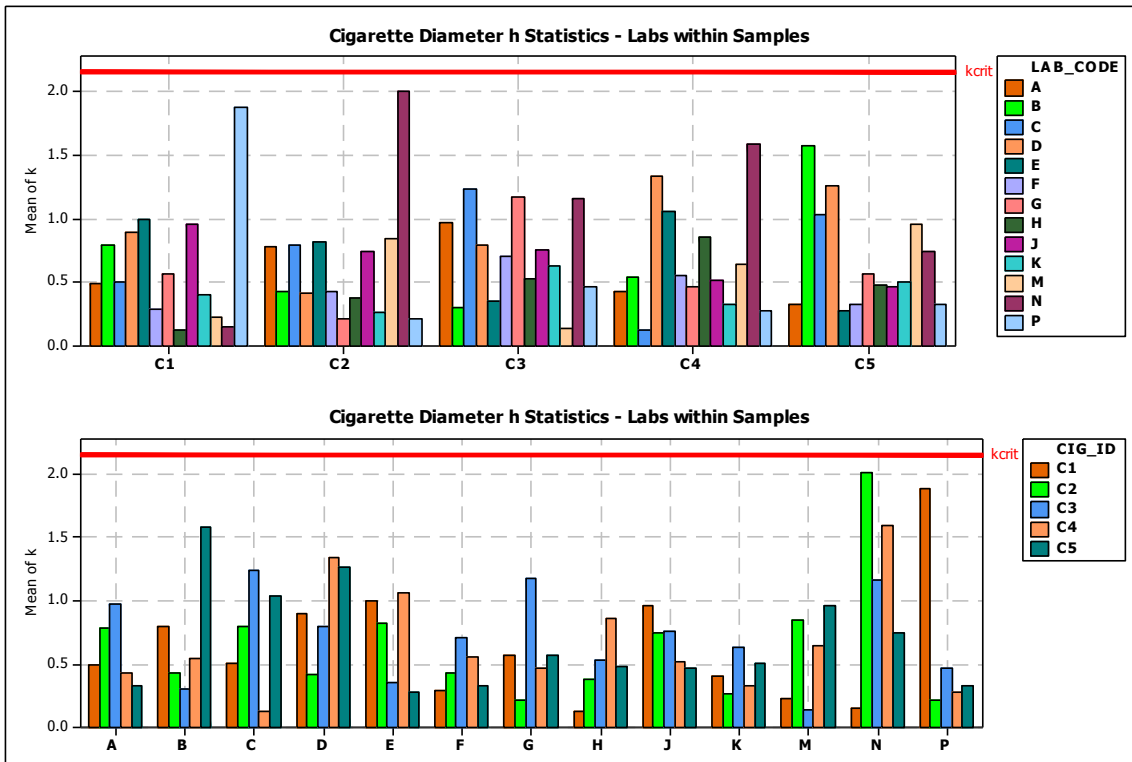


Figure 23. Cigarette Pressure Drop: *k*-statistics Mandel Plots



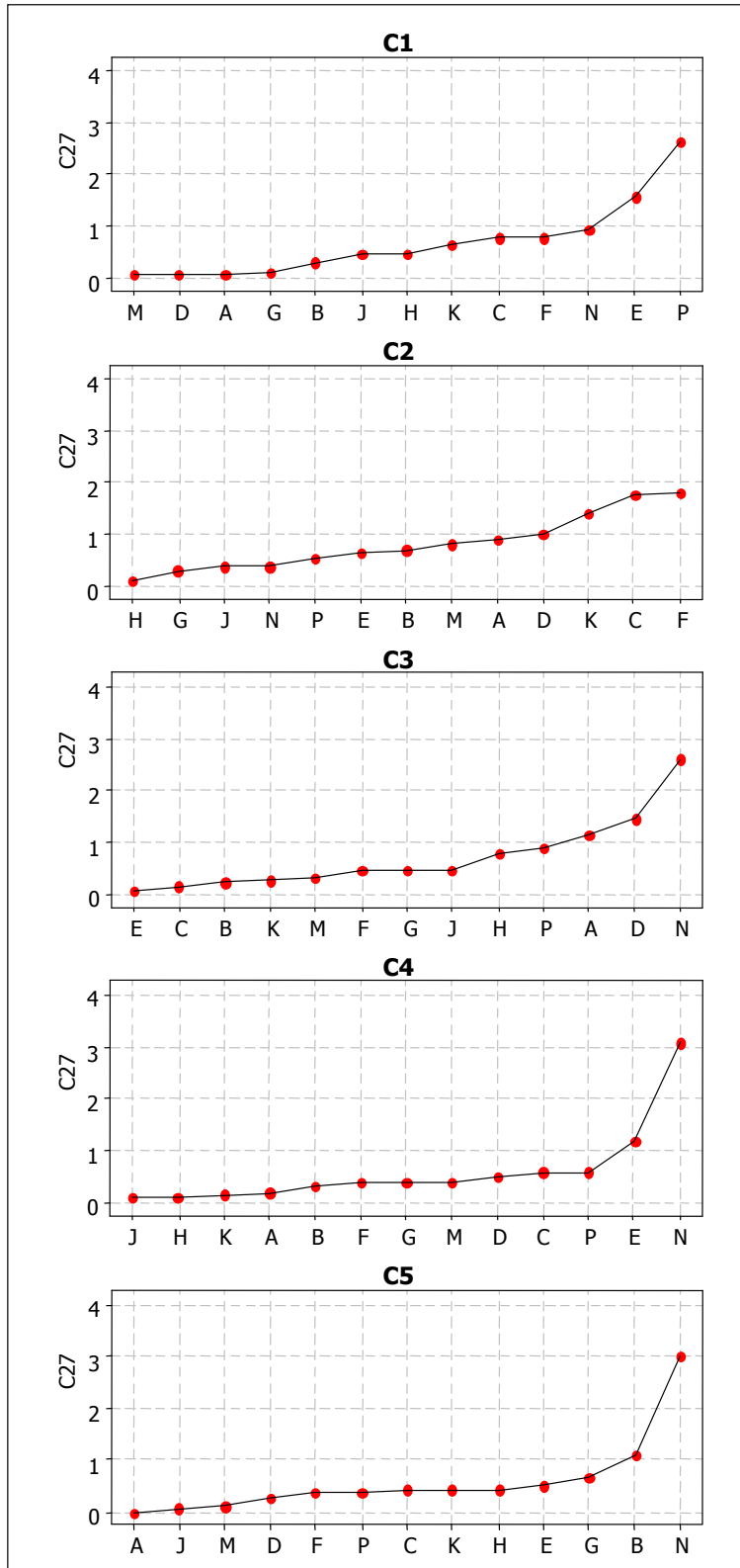
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Cigarette Pressure Drop (open) – Repeatability Variation: Cochran's Test

There were no unusual results for Cochran's test.

Figure 24. Cigarette Pressure Drop: Z-Score Plots



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CIGARETTE VENTILATION

Table 10. Cigarette Ventilation: Precision Statistics

| CIGARETTE ID | MEAN | sr | sR | r | R |
|--------------|------|------|------|------|------|
| C1 | 18.9 | 0.51 | 0.59 | 1.43 | 1.64 |
| C2 | 27.5 | 0.80 | 0.80 | 2.25 | 2.25 |
| C3 | 41.8 | 0.66 | 0.85 | 1.85 | 2.38 |
| C4 | 45.1 | 0.97 | 1.66 | 2.72 | 4.65 |
| C5 | 81.6 | 0.62 | 0.93 | 1.74 | 2.61 |

The Individual values plots for cigarette ventilation identified Laboratory P Cigarette C1 as different enough to distort the scale on the graphs (Fig 25), there was also one extreme value for Lab N Cigarette 2. The plots were redrawn without these values (Fig. 25a).

Mandel Plots

The Mandel Plot shows that Laboratory N is significantly different from the mean value for cigarettes C2 and C4. For cigarette C2 the value is significantly lower, and for cigarette C4, it is significantly higher.

The k-statistic plot (Fig. 27) shows exceptional variation for Laboratory N, cigarette C2.

Repeatability Variation

Cochran's test suggests exceptional variation for Laboratory N cigarette C2.

Z-scores

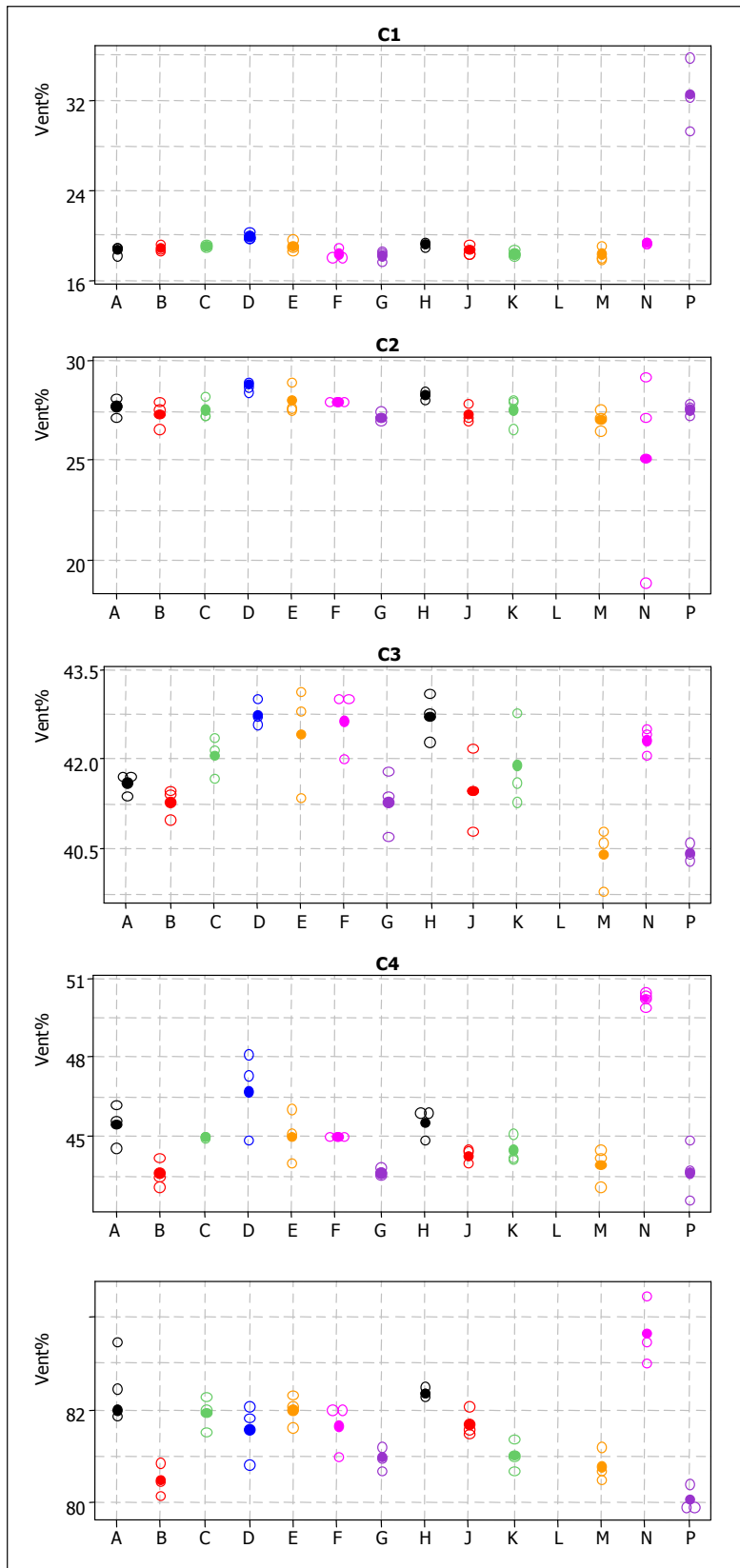
Results for Laboratory N, cigarettes C2, C4 and C5 are questionable.

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

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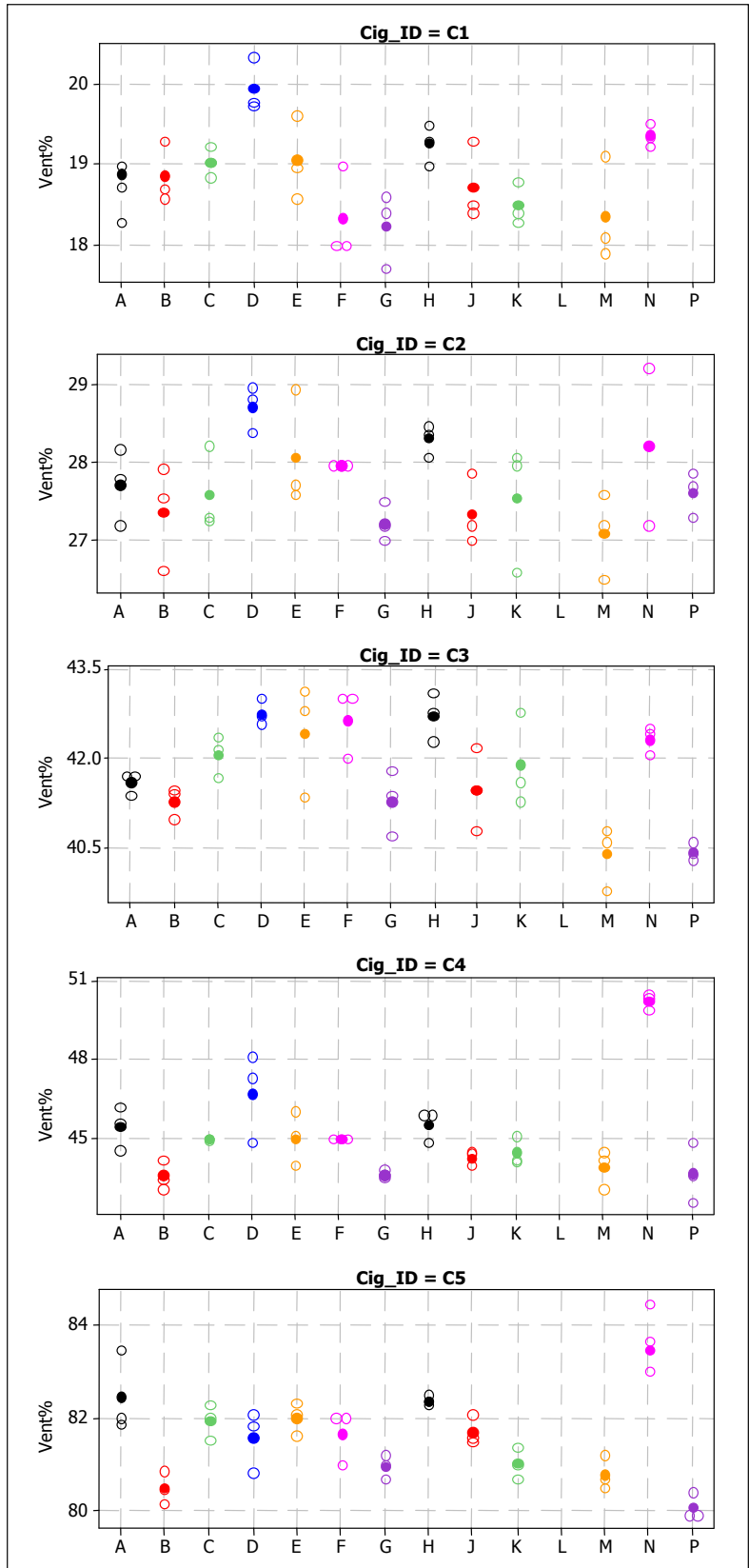
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Figure 25. Cigarette Ventilation: Individual Value Plots



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Figure 25a. Cigarette Ventilation: Individual Value Plots – Extreme Values Removed



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Figure 26. Cigarette Ventilation: *h*-statistics Mandel Plots

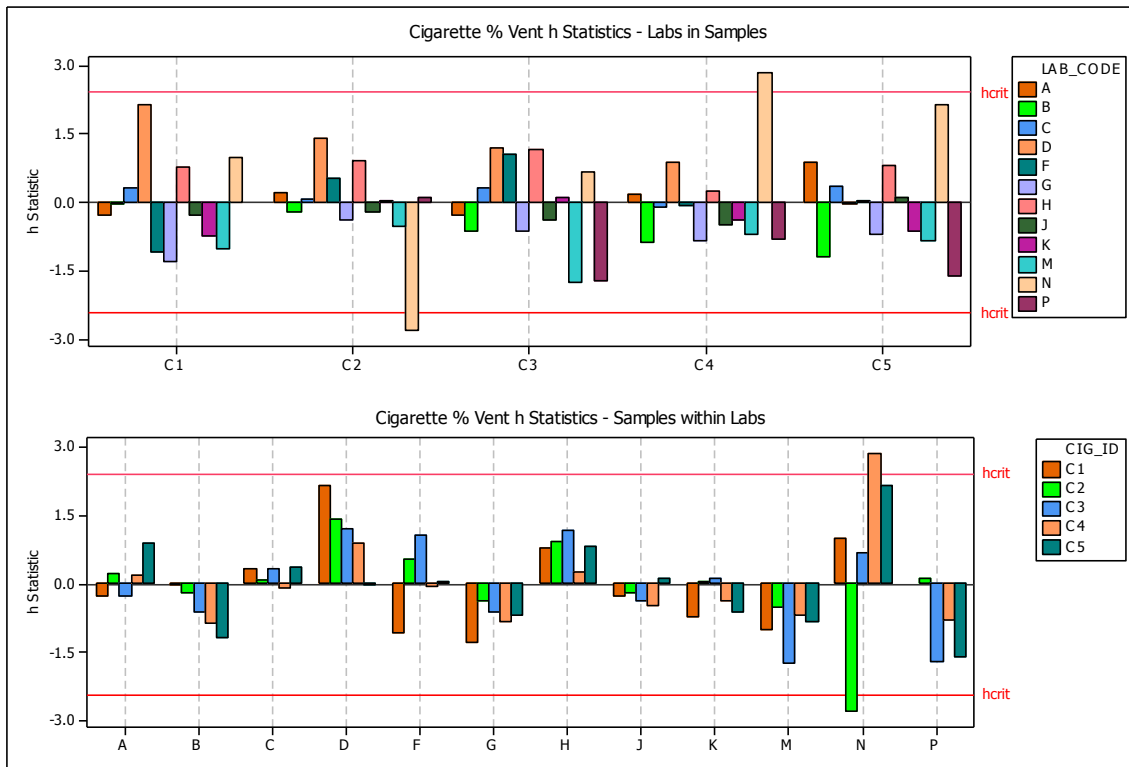
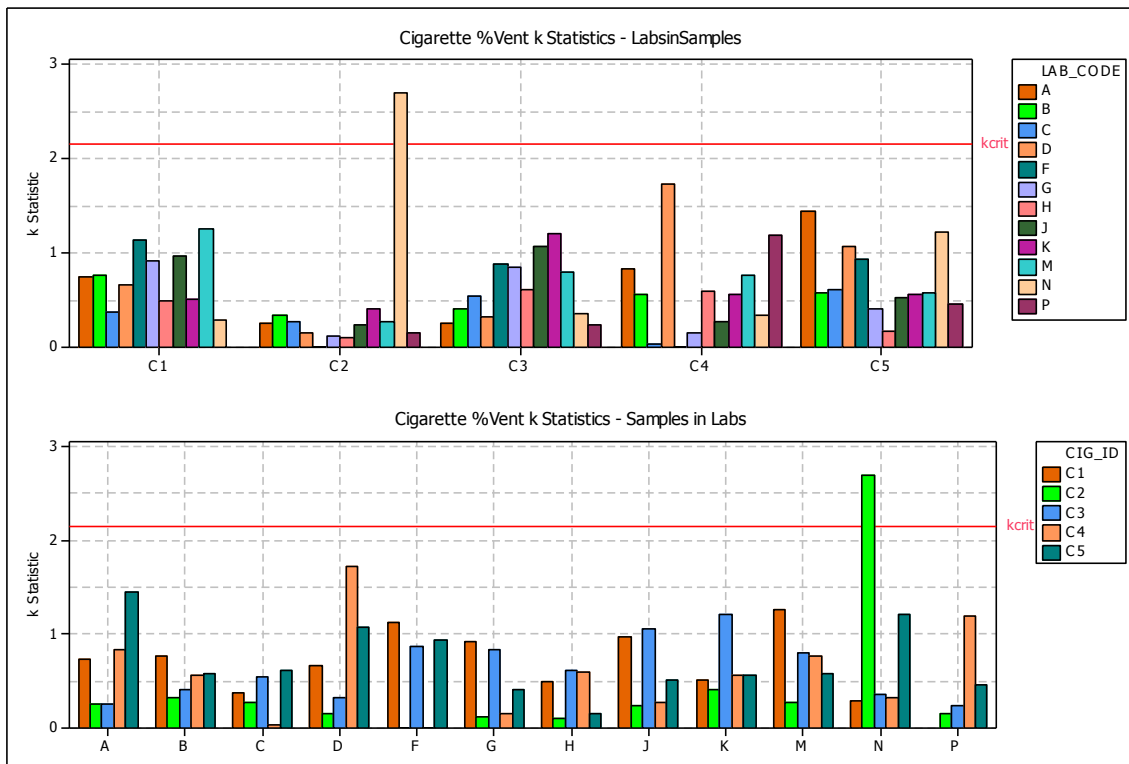


Figure 27. Cigarette Ventilation: *k*-statistics Mandel Plots

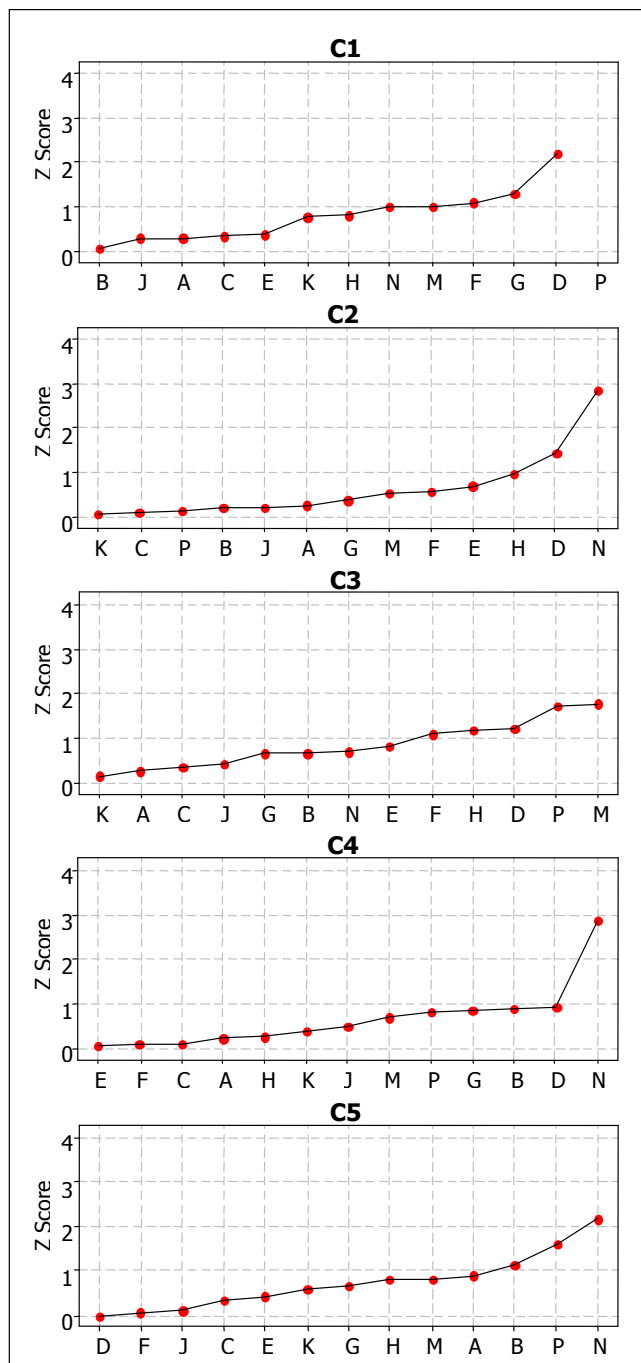


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Cigarette Ventilation: Repeatability Variation

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.*