Physical Test Methods Sub-Group

Technical Report

4th Proficiency Test (2020) on Diffusion Capacity of Cigarette Papers

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1. INTRODUCTION

1.1 Purpose and Scope

In 2014 the Physical Test Methods (PTM) Sub-Group of CORESTA published CRM No. 77 - Determination of Diffusion Capacity by Measurement of CO_2 Transfer Rate Through Materials Used as Cigarette Papers and Cigarette Papers having an Oriented Zone of Reduced Diffusion Capacity.

During and shortly after the development of this CRM the first and second proficiency tests were carried out and a further proficiency test was carried out in 2017. Improving laboratory proficiency for the measurement of diffusion capacity has turned out to be difficult, as no standard reference material for diffusion capacity is available despite an intensive search and numerous experiments and studies in the PTM Sub-Group. Thus, currently only the components of the measurement instruments can be calibrated and adjusted but not the measurement process as a whole. Consequently, the PTM Sub-Group decided to carry out this proficiency test to ensure that the laboratories using CRM No. 77 do not drift apart over time.

The results from this study serve as a check that laboratory procedures and their development over time did not lead to a substantial increase in between-laboratory variability and it further offers each laboratory the possibility to assess its performance in comparison to other laboratories and to derive actions for improvement.

All results are presented in anonymized form.

1.2 Study Protocol

The test protocol used for this proficiency test is given in Appendix A and is briefly summarized below.

The protocol contained information about the samples to be tested, the preparation of samples and the number of replicate determinations to be made, but otherwise asked laboratories to follow their internal routine procedures, in particular regarding certain instrument settings and the number of individual measurements for each replicate determination. Apart from the results, the laboratories were asked to provide information related to the configuration and the calibration of the measurement instrument and ambient conditions during measurement, particularly including the ambient air pressure, which is a known and uncontrolled factor that may influence the measurement result.

The measurement data were reported in an Excel sheet and sent to the study coordinator, who checked the results for plausibility. No corrections had to be made. Then the data set was anonymized and compiled into a single data sheet for evaluation and statistical analysis.

The distribution of samples started in June 2020 and the measured data were to be reported by August 31, 2020. By September 16, 2020, all data sets were received, but two laboratories, which initially registered for the proficiency test, informed the study coordinators before the deadline that they would not be able to provide results for at least one of the sample sets they had received.

1.3 Products and Measurements

For the proficiency test, two lower ignition propensity (LIP) cigarette papers were used. Both papers featured bands of low diffusion capacity to achieve self-extinguishment according to ISO 12863:2010. The laboratories were asked to make three determinations, on separate days, of the diffusion capacity of the LIP bands on the cigarette paper. The main characteristics of the two LIP cigarette papers are given in Table 1. The values in Table 1 do not represent actual measured values but just serve as an indication of the characteristics of the LIP cigarette paper.

As the upper and lower part of the measurement head exert a mechanical pressure on the paper, the paper may get damaged in the clamped areas. Thus a new sample strip was to be used for each measurement.

Table 1 – Characteristics of the LIP cigarette papers

Sample	Basis Weight g/m²	Air Permeability CU	D* (Band) cm/s
A	24	75	0,050
B	24	45	0,180

For the measurements the laboratories generally followed CRM No. 77 and mostly used either 10 or 20 individual measurements for one determination of diffusion capacity. One laboratory reported 16 individual measurements for a replicate and one laboratory provided only 9 individual measurements for sample B on day 1.

1.4 Study Participants

In total 6 laboratories participated in the study with the entire list of participants in alphabetical order given in Table 2. A code was assigned to each laboratory by the Study Coordinator, thus the order of laboratories in Table 2 does not agree with the order of the laboratories in other tables. Several laboratories participated with more than one instrument so that the number of instruments is also given in Table 2. In total 11 instruments from two different instrument manufacturers (Borgwaldt KC, Sodim) were used in the study.

The instruments used by the laboratories were distributed over the two instrument manufacturers by 8 and 3 instruments, which needs to be taken into account in the analysis, as past studies have shown differences between the results obtained on instruments of different manufacturers, but less so between instruments of the same manufacturer.

Table 2 – List of Participants

Participant	Country	No. of Instruments
British American Tobacco	Germany	1
Japan Tobacco International Germany GmbH	Germany	2
Papierfabrik Wattens GmbH & Co KG	Austria	3
SODIM	France	1
SWM International	France	3
China National Tobacco Supervision & Test Center	China	1

2. STATISTICAL EVALUATION

2.1 Raw Data Treatment

Data sets from all 6 laboratories were received covering in total 11 instruments. After a brief screening for inconsistencies, the data were prepared for statistical analysis.

Mean values (MV) over all instruments, within-laboratory standard deviation (SD-WL) and between-laboratory standard deviation (SD-BL) are provided in Table 3.

Table 3 – Mean values (MV), average within-laboratory standard deviations (SD-WL) and between-laboratory standard deviations (SD-BL) for diffusion capacity over all instruments, outliers included

ID	Band Diffusion Capacity			
	MV cm/s	SD-WL cm/s	SD-BL cm/s	N
A	0,048	0,0025	0,0056	11
B	0,175	0,0074	0,0199	11

The complete data set for all laboratories is given in Appendices B.1 to B.3.

2.2 Outlier Analysis and Removal

In order to evaluate laboratory proficiency in the form of z-scores, as described in ISO 13528:2015, a 'true' value and standard deviation need to be assigned to each measurement parameter, which form the basis for the calculation of z-scores. In contrast to other studies, where the 'true' value is known or can be easily assigned, such values are not available in this study. Consequently, the 'true' mean value and standard deviation were determined as a robust global average over all laboratories. To ensure that this global average represents the unknown 'true' value as closely as possible, outlier testing was performed to eliminate any laboratories, that provided unusual values. Outlier testing was only used to obtain a robust mean value and standard deviation, the z-scores were then calculated for all laboratories, which reported data, irrespective of whether their results were excluded in the calculation of the robust mean value.

The procedure follows ISO 13528:2015 by using Cochran's test to eliminate any laboratories with exceptionally high standard deviation and using Grubbs' test to detect whether the one or two highest or lowest values qualify as outlier.

As the individual values were available, Grubbs' test was first performed on each set of individual values per sample per day to check for any outlying values. In this step outliers were identified according to Table 4, all of them being too high, and excluded from further analysis.

Table 4 – Number of the individual data points detected as outlier by Grubbs' test on the individual measurement results per sample per day

Lab ID	Sample A			Sample B		
	Day 1	Day 2	Day 3	Day 1	Day 2	Day 3
4	1, 6		4			
5			8			7
9		11, 17				
10	8					

From the remaining data points the replicate values were calculated by averaging over the individual values so that the three replicate values per sample and laboratory were obtained. Mean and standard deviations of the replicates are provided in Appendices B.4 to B.6. These replicates were tested by Cochran's test to eliminate laboratories with high standard deviations. In this test the replicates of laboratory 2 for sample A were detected as outlier and eliminated from further calculations. Grubbs' test on the replicate values did not show any further outliers, so that laboratory 2, sample A, remained the only data set that was excluded. Mean values and standard deviations of the replicates after outlier elimination are provided in Appendix C.1. Mean values and confidence intervals of the replicates, after elimination of outliers according to Table 4 are shown in the diagrams in Appendix D.1, while the mean value and confidence interval for each of the replicates of days 1, 2 and 3, including all outliers, are shown in Appendices D.2 to D.4.

The remaining data sets were then used to calculate a robust mean and standard deviation.

2.3 Robust Mean Values and Standard Deviations

After the removal of outliers as explained above robust mean values, within-laboratory standard deviations and between-laboratory standard deviations were calculated using algorithm A of ISO 13528:2015. The results are given in Table 5. The number of laboratories is denoted by N.

Table 5 – Robust mean values (MV), average within-laboratory standard deviations (SD-WL), between-laboratory standard deviations (SD-BL) and coefficients of variation (CoV) for diffusion capacity, over all instruments, outliers excluded

ID	MV cm/s	SD-WL cm/s	Diffusion Capacity			
			CoV (WL) %	SD-BL cm/s	CoV (BL) %	N
A	0,048	0,0021	4,3	0,0056	12,3	10
B	0,174	0,0074	4,2	0,0199	11,9	11

These values were used for the calculation of z-scores.

For the further analysis it is of interest to investigate the differences between the instruments of various instrument manufacturers. For each of the two instrument manufacturers, robust global mean values and standard deviations were calculated separately and are given in Table 6.

Table 6 – Robust mean values (MV), average within-laboratory standard deviations (SD-WL) and between-laboratory standard deviations (SD-BL) for the diffusion capacity in the LIP bands of samples A and B. The data are provided separately for the two instrument manufacturers (Mfct) a and b. Outliers are excluded.

ID	Mfct	Diffusion Capacity			
		MV cm/s	SD-WL cm/s	SD-BL cm/s	N
A	a	0,046	0,0023	0,0048	7
	b	0,051	0,0014	0,0067	3
B	a	0,170	0,0082	0,0141	8
	b	0,176	0,0044	0,0313	3

2.4 Evaluation of Laboratory Performance (z-Scores)

Based on the robust mean value and the between-laboratory standard deviation z-scores were calculated as described in ISO 13528:2015. The results are given for samples A and B in Table 7. It has to be noted that individual data points detected as outliers by Grubbs' test according to Table 4 were removed before the calculation of z-scores. Inclusion of these outliers changes the absolute values of the z-scores by less than 0,05 and thus has no effect on the conclusions.

Table 7 – Z-Scores for all laboratories on the measurement of diffusion capacity of the LIP bands of samples A and B

Lab	Sample A	Sample B
1	1,39	0,83
2	0,97	0,85
3	-0,94	-0,15
4	-1,04	-0,97
5	-0,80	-0,56
6	-0,50	-0,34
7	1,45	1,62
8	-0,72	-1,20
9	0,19	-0,75
10	-0,23	-0,74
11	1,22	1,41

3. DATA INTERPRETATION

3.1 Conclusions from Outlier Testing

As has been observed in previous proficiency tests on diffusion capacity, the repeatability standard deviation of the measurement method, i.e. testing the same sample repeatedly on the same instrument by the same operator, is rather low. Even when day-to-day variability and product variability is included, as in this test, it remains low. In contrast the between-laboratory variability is substantially higher, mainly due to the inability to calibrate and adjust the measurement instruments by using a reference material.

Due to this large between-laboratory variability it does not come as a surprise that Grubbs' test did not detect any outliers between the laboratories, while within each laboratory, the individual data sets contained some outliers. With 8 outliers in 947 data points, however, their number is still rather low. Also the fact that while the measurement method itself is fairly sophisticated the actual operation of the instrument by the user is comparably simple. Thus, given proper conditioning of the samples and positioning of the LIP bands under the opening in the measurement head, there are few options where the operator can influence the measurement result.

In past studies a number of outlying measurements on LIP bands were found, as operators had difficulty positioning the LIP band under the opening in the measurement head. This leads to exceptionally high diffusion capacity values as areas of the much higher permeable base paper

contribute to the diffusion process. In the present study only a few such outliers were observed, which is an indication that laboratories have achieved a certain routine in dealing with the hardly visible LIP bands on the cigarette paper.

3.2 Conclusions from the Z-Scores

As described in ISO 13528:2015, in normal circumstances about 95 % of all z-scores will be in the range between -2 and 2. Occasionally, absolute z-scores equal to or greater than 2 may be expected at a rate of about 5 %, while absolute z-scores equal to or greater than 3 will occur only at a rate of about 0,3 %.

Thus for absolute z-scores between 2 and 3 it is up to the laboratory to decide if these exceptional values are of importance and require any corrective action or review of the laboratory procedures. For absolute z-scores of 3 or higher it is highly recommended that the laboratory investigates the reasons for the deviation and derives appropriate actions from these investigations.

In the present study no z-scores with absolute values above 2 were detected, which shows that the laboratories are able to reliably carry out the measurement method and that there are no large differences in mean values between the laboratories. However, z-scores compare deviations from the global mean to a standard deviation based on the between-laboratory variability, and therefore the generally high between-laboratory variability also helps to keep z-scores low, even when laboratories differ substantially in their mean values.

No statistically significant differences were observed between the instruments of the two manufacturers. A likely reason for this result is that laboratories using several instruments try to match the instruments to each other by measuring internal standard samples. Often instruments will allow adjustment factors to be set, such as gain and offset, to make the instruments match each other. Therefore, it may be expected that laboratories taking part with a higher number of instruments may obtain internally homogeneous results from instruments of different manufacturers. It has to be noted that two laboratories each using 3 instruments, make up more than half of all instruments in the study.

As can be seen in Table 5, the between-laboratory standard deviation is still a factor of about 3 higher than the average within-laboratory standard deviation. Despite a large number of instruments likely to be matched to each other, the differences between the laboratories and instruments remain a substantial factor in the overall variability. With a coefficient of variation, determined from the robust mean and the between-laboratory standard deviation, of about 10 %, the measurement of diffusion capacity still remains a less precise method than other similar, but admittedly less complex methods such as the measurement of air permeability.

3.3 Comparison with Historical Data

One of the purposes of a proficiency test is to assess laboratory performance so that over time a steady improvement can be achieved. The following historical assessment by comparing data from the previous three proficiency tests on diffusion capacity, carried out in 2014, 2015 and 2017, with the current test results are an attempt to investigate if such an improvement can be observed.

The results of this analysis have to be interpreted very cautiously as different laboratories have taken part in the four proficiency tests and as the test products differed in all tests. In contrast to the previous proficiency tests, in this test measurements were carried out only on LIP bands and not on the base paper as the diffusion capacity of the bands is of more practical relevance.

The LIP bands in all studies had a diffusion capacity between 0,02 cm/ and 0,20 cm/s, typically one high and one low value and the base papers in the first three proficiency tests had a diffusion capacity between 1,0 cm/s and 2,5 cm/s, thus at least the measurement ranges were similar.

To assess overall performance an average robust coefficient of variation is calculated over all samples by the ratio of the robust standard deviation and the robust global mean value and expressed as a percentage. As the robust standard deviation is calculated from the between-laboratory standard deviations it may be expected that the robust coefficient of variation decreases over time as the laboratories improve and the differences between the laboratories become smaller.

The results of this analysis are provided in Table 8 and are to be understood for information only and are not based on any specific statistical test or analysis.

Table 8 – Historical development of a robust coefficient of variation for diffusion capacity

Parameter	Robust Coefficient of Variation %			
	2014	2015	2017	2020
On LIP Bands	11,7	11,4	10,5	12,1
Between LIP Bands	5,3	5,6	12,0	---

The robust coefficient of variation for measurements on LIP bands did not change substantially over the four proficiency tests and has remained at about 10 % to 12 %. Also, this study did not show any significant difference in mean values and variability obtained with instruments from different manufacturers.

It is recommended to carry out further proficiency tests on a regular basis to at least ensure that laboratories do not drift apart and that the between-laboratory variability does not increase over time. Even though serious, but unsuccessful, attempts and experiments have been made to identify reference materials, it is also recommended to further try to identify such a reference material and to develop a 'gold-standard' method based on CRM 77 which can be used to assign values to calibration or reference standards.

4. REFERENCES

- CORESTA CRM No. 77:2014, Determination of Diffusion Capacity by Measurement of CO_2 Transfer Rate Through Materials Used as Cigarette Papers and Cigarette Papers having an Oriented Zone of Reduced Diffusion Capacity
- CORESTA Technical Report (2017), 3rd Proficiency Test (2017) on Diffusion Capacity of Cigarette Papers, PTM-123
- CORESTA Technical Report (2015), 2015 Proficiency Test Report - The Measurement of Diffusion Capacity of Cigarette Papers
- CORESTA Technical Report (2015), Collaborative Study to Evaluate a Paperboard Substitute for a Diffusion Capacity Standard
- CORESTA Technical Report (2015), 2014 Proficiency Test Report - The Measurement of Diffusion Capacity of Cigarette Papers
- ISO 12863:2010, Standard test method for assessing the ignition propensity of cigarettes

5. APPENDICES

APPENDIX A – Protocol

The protocol is reproduced in its original form. Minor typographical errors were corrected and e-mail addresses were removed.

4th Proficiency Test on Diffusion Capacity

NOTE: The purpose of a proficiency test is to allow laboratories to evaluate themselves in comparison to other laboratories. Therefore, it is important that you follow the procedure which is routinely used in your laboratory.

1. Measurement Plan

Measurements on each sample (and each instrument, if applicable) should take place on 3 consecutive days (e.g. Monday / Tuesday / Wednesday).

On each day determine the diffusion capacity of each of the samples A and B by performing the number of measurements, typically 10, you would normally use in routine determinations of diffusion capacity in your laboratory. On each day a new set of sample strips shall be used.

DAY 1 - Determination of Diffusion Capacity on Samples A1 and B1 (on bands only)

DAY 2 - Determination of Diffusion Capacity on Samples A2 and B2 (on bands only)

DAY 3 - Determination of Diffusion Capacity on Samples A3 and B3 (on bands only)

It is possible that different operators perform the measurements on different days, but only one instrument shall be used.

Please report individual measured values in the appropriate cells.

Make sure that consecutive measurements on bands are sufficiently spaced apart so that no damaged paper area is used for the measurement.

2. Measurement Method and Instrument Set-Up

According to CRM No. 77, calibration and instrument settings as used in your laboratory. Condition samples for at least 48 hours prior to the first series of measurements.

3. Recording of Results

It is not permitted to make any changes to the data recording sheets. Results, which are not reported correctly, may be disregarded in the proficiency test. For any additional information or remarks please use the comments column. You can also add an additional spreadsheet.

E-mail completed spreadsheets (as Excel file) to:

Bernhard Eitzinger

Philippe Le Men

Results must be submitted by August 31, 2020.

4. Sample Codes

A		Banded lower ignition propensity paper
	A1	for measurements on the band on day 1
	A2	for measurements on the band on day 2
	A3	for measurements on the band on day 3
B		Banded lower ignition propensity paper
	B1	for measurements on the band on day 1
	B2	for measurements on the band on day 2
	B3	for measurements on the band on day 3

APPENDIX B – Raw Data

Appendix B.1 – Determinations of diffusion capacity per laboratory and day on the LIP bands of samples A and B, including all outliers.

Lab	Sample A			Sample B		
	Day 1 cm/s	Day 2 cm/s	Day 3 cm/s	Day 1 cm/s	Day 2 cm/s	Day 3 cm/s
1	0,060	0,054	0,052	0,186	0,183	0,204
2	0,056	0,056	0,047	0,196	0,198	0,179
3	0,040	0,044	0,042	0,166	0,182	0,166
4	0,039	0,042	0,045	0,155	0,161	0,149
5	0,043	0,044	0,043	0,165	0,161	0,162
6	0,045	0,044	0,045	0,159	0,172	0,172
7	0,054	0,056	0,058	0,215	0,205	0,200
8	0,044	0,042	0,044	0,150	0,150	0,151
9	0,049	0,048	0,050	0,162	0,149	0,166
10	0,046	0,046	0,047	0,167	0,156	0,155
11	0,055	0,054	0,054	0,203	0,202	0,202

Appendix B.2 – Standard deviation of an individual measurement (i.e. not of a replicate) of diffusion capacity per laboratory and day on the LIP bands of samples A and B, including all outliers.

Lab	Sample A			Sample B		
	Day 1 cm/s	Day 2 cm/s	Day 3 cm/s	Day 1 cm/s	Day 2 cm/s	Day 3 cm/s
1	0,0070	0,0038	0,0070	0,0211	0,0247	0,0194
2	0,0069	0,0034	0,0024	0,0175	0,0112	0,0148
3	0,0035	0,0069	0,0040	0,0180	0,0180	0,0131
4	0,0023	0,0040	0,0027	0,0096	0,0099	0,0093
5	0,0043	0,0036	0,0040	0,0127	0,0114	0,0104
6	0,0030	0,0034	0,0047	0,0135	0,0162	0,0128
7	0,0059	0,0064	0,0071	0,0155	0,0197	0,0139
8	0,0031	0,0044	0,0047	0,0091	0,0088	0,0105
9	0,0030	0,0050	0,0043	0,0125	0,0067	0,0127
10	0,0036	0,0030	0,0052	0,0138	0,0122	0,0112
11	0,0070	0,0041	0,0041	0,0116	0,0063	0,0045

Appendix B.3 – Number of individual measurements used for a determination of diffusion capacity per laboratory and day on the LIP bands of samples A and B, including all outliers.

	Sample A			Sample B		
Lab	**Day 1**	**Day 2**	**Day 3**	**Day 1**	**Day 2**	**Day 3**
1	10	10	10	10	10	10
2	10	10	10	10	10	10
3	10	10	10	10	10	10
4	20	20	20	20	20	20
5	20	20	20	20	20	20
6	20	20	20	20	20	20
7	10	10	10	10	10	10
8	10	10	10	10	10	10
9	20	20	20	16	16	16
10	20	20	20	20	20	20
11	10	10	10	9	10	10

Appendix B.4 – Determinations of diffusion capacity per laboratory and day on the LIP bands of samples A and B, excluding outliers eliminated by Grubbs' test applied to the individual data points. Laboratories that had outliers are highlighted.

Lab	**Sample A**			**Sample B**		
	Day 1 cm/s	**Day 2 cm/s**	**Day 3 cm/s**	**Day 1 cm/s**	**Day 2 cm/s**	**Day 3 cm/s**
1	0,060	0,054	0,052	0,186	0,183	0,204
2	0,056	0,056	0,047	0,196	0,198	0,179
3	0,040	0,044	0,042	0,166	0,182	0,166
4	0,038	0,042	0,044	0,155	0,161	0,149
5	0,043	0,044	0,042	0,165	0,161	0,164
6	0,045	0,044	0,045	0,159	0,172	0,172
7	0,054	0,056	0,058	0,215	0,205	0,200
8	0,044	0,042	0,044	0,150	0,150	0,151
9	0,049	0,047	0,050	0,162	0,149	0,166
10	0,045	0,046	0,047	0,167	0,156	0,155
11	0,055	0,054	0,054	0,203	0,202	0,202

Appendix B.5 – Standard deviation of an individual measurement (i.e. not of a replicate) of diffusion capacity per laboratory and day on the LIP bands of samples A and B, excluding outliers eliminated by Grubbs' test applied to the individual data points. Laboratories that had outliers are highlighted.

Lab	Sample A			Sample B		
	Day 1 cm/s	Day 2 cm/s	Day 3 cm/s	Day 1 cm/s	Day 2 cm/s	Day 3 cm/s
1	0,0070	0,0038	0,0070	0,0211	0,0247	0,0194
2	0,0069	0,0026	0,0024	0,0175	0,0112	0,0148
3	0,0035	0,0063	0,0040	0,0180	0,0180	0,0131
4	0,0013	0,0033	0,0020	0,0096	0,0099	0,0093
5	0,0043	0,0028	0,0032	0,0127	0,0114	0,0082
6	0,0030	0,0021	0,0047	0,0135	0,0162	0,0128
7	0,0059	0,0055	0,0071	0,0155	0,0197	0,0139
8	0,0031	0,0037	0,0047	0,0091	0,0088	0,0105
9	0,0030	0,0016	0,0043	0,0125	0,0067	0,0127
10	0,0029	0,0038	0,0052	0,0138	0,0122	0,0112
11	0,0070	0,0030	0,0041	0,0116	0,0063	0,0045

Appendix B.6 – Number of individual measurements used for a determination of diffusion capacity per laboratory and day on the LIP bands of samples A and B, excluding outliers eliminated by Grubbs' test applied to the individual data points. Laboratories that had outliers are highlighted.

Lab	Sample A			Sample B		
	Day 1	Day 2	Day 3	Day 1	Day 2	Day 3
1	10	10	10	10	10	10
2	10	10	10	10	10	10
3	10	10	10	10	10	10
4	18	20	19	20	20	20
5	20	20	19	20	20	19
6	20	20	20	20	20	20
7	10	10	10	10	10	10
8	10	10	10	10	10	10
9	20	18	20	16	16	16
10	19	20	20	20	20	20
11	10	10	10	9	10	10

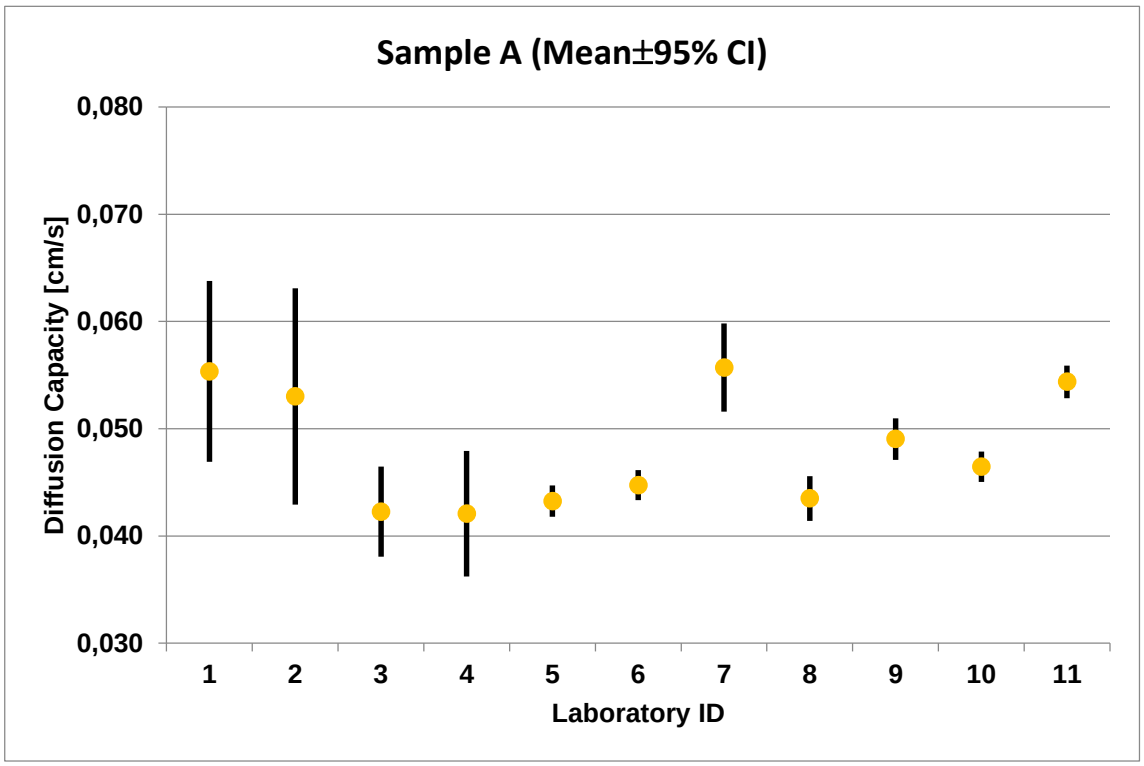
APPENDIX C – Measurement Results

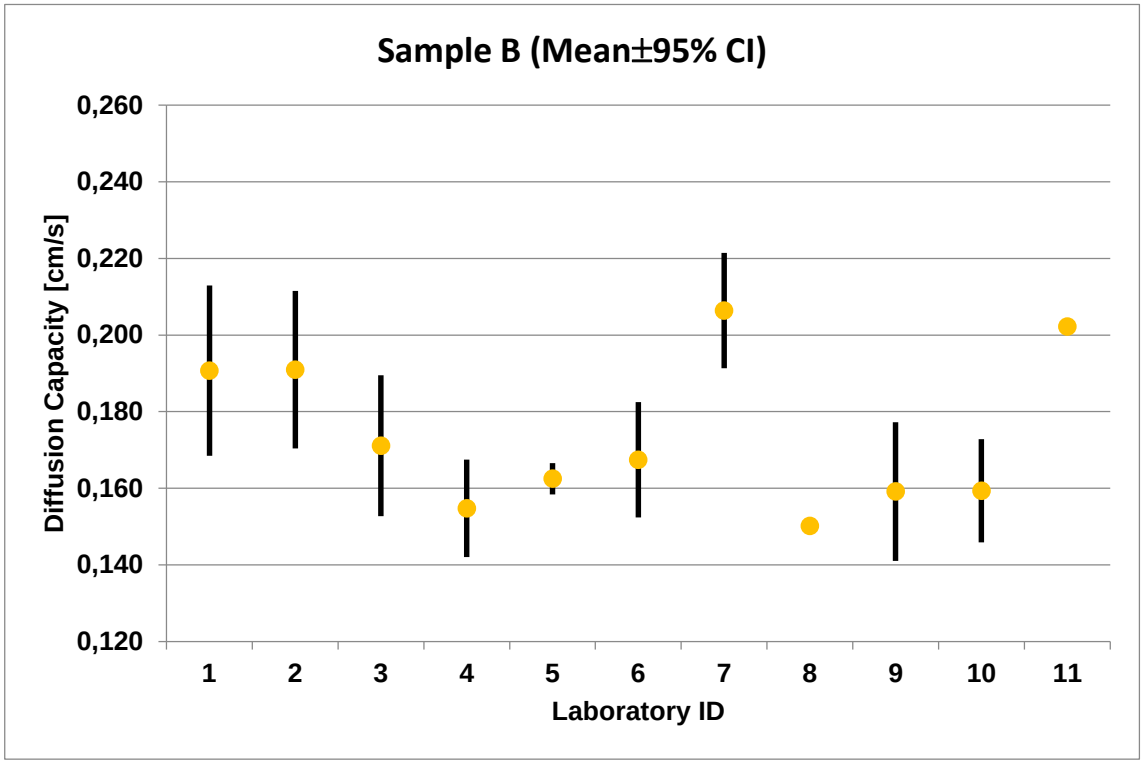
Appendix C – Mean value (MV) and standard deviation (SD) of the three determinations of diffusion capacity per laboratory on the LIP bands of samples A and B. The data set excluded as outlier based on Cochran's test is highlighted.

Lab	Sample A		Sample B	
	MV cm/s	SD cm/s	MV cm/s	SD cm/s
1	0,055	0,0042	0,191	0,0111
2	0,053	0,0050	0,191	0,0103
3	0,042	0,0021	0,171	0,0092
4	0,042	0,0030	0,155	0,0064
5	0,043	0,0010	0,163	0,0022
6	0,045	0,0007	0,167	0,0075
7	0,056	0,0021	0,206	0,0075
8	0,044	0,0010	0,150	0,0004
9	0,049	0,0016	0,159	0,0091
10	0,046	0,0009	0,159	0,0067
11	0,054	0,0008	0,202	0,0004

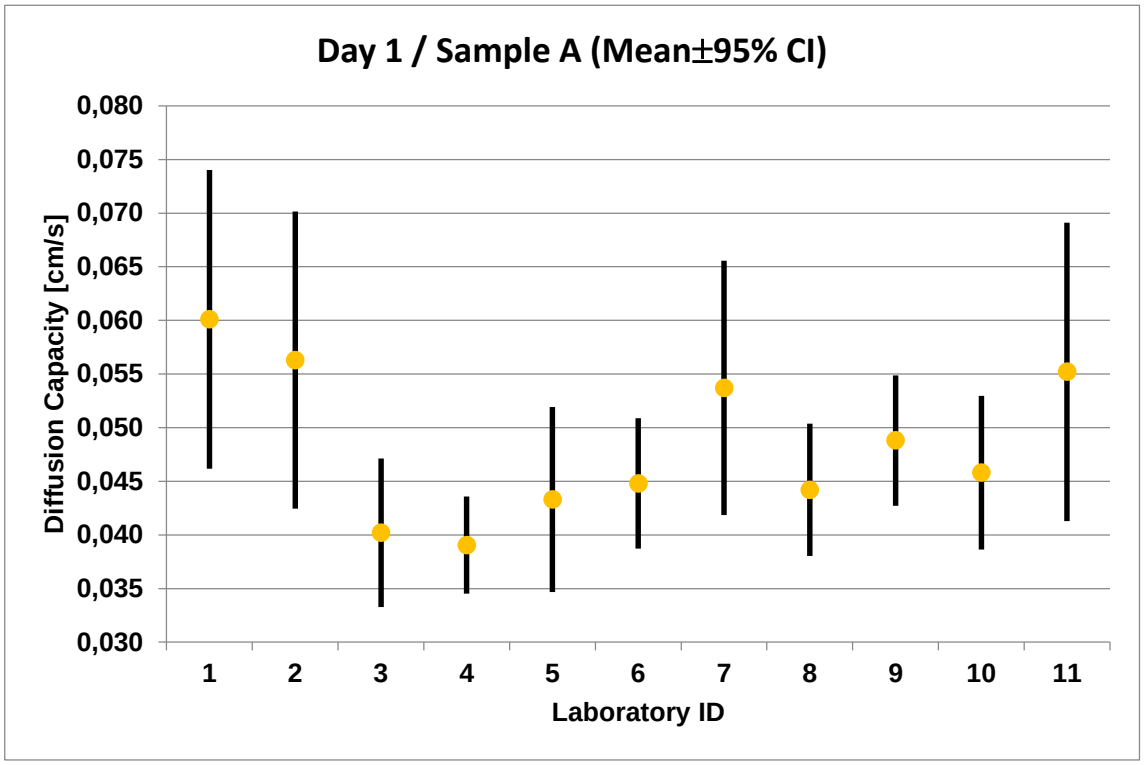
APPENDIX D – Laboratory Results (Diagrams)

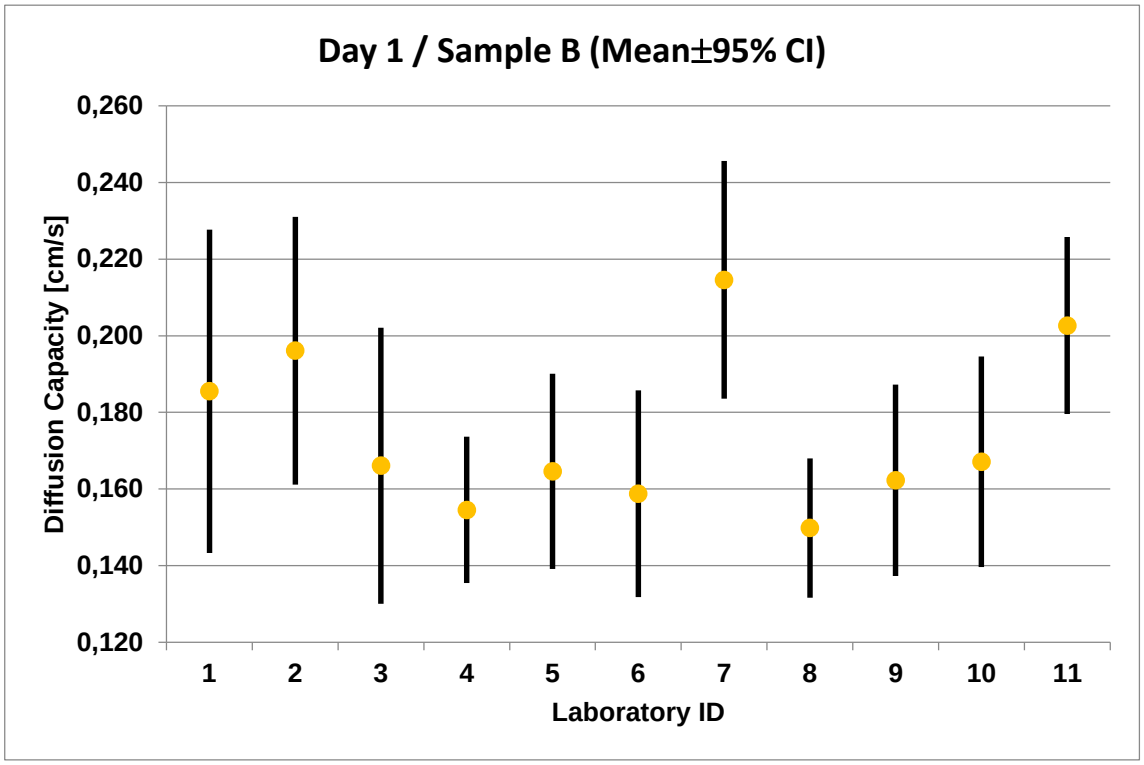
Appendix D.1 – Average and range of the three replicate determinations of diffusion capacity of LIP bands for samples A and B for all laboratories. Outliers identified by Grubbs' test according to Table 4 are excluded. The error bars are ±1.96 times the standard deviation of the three replicates of each laboratory.



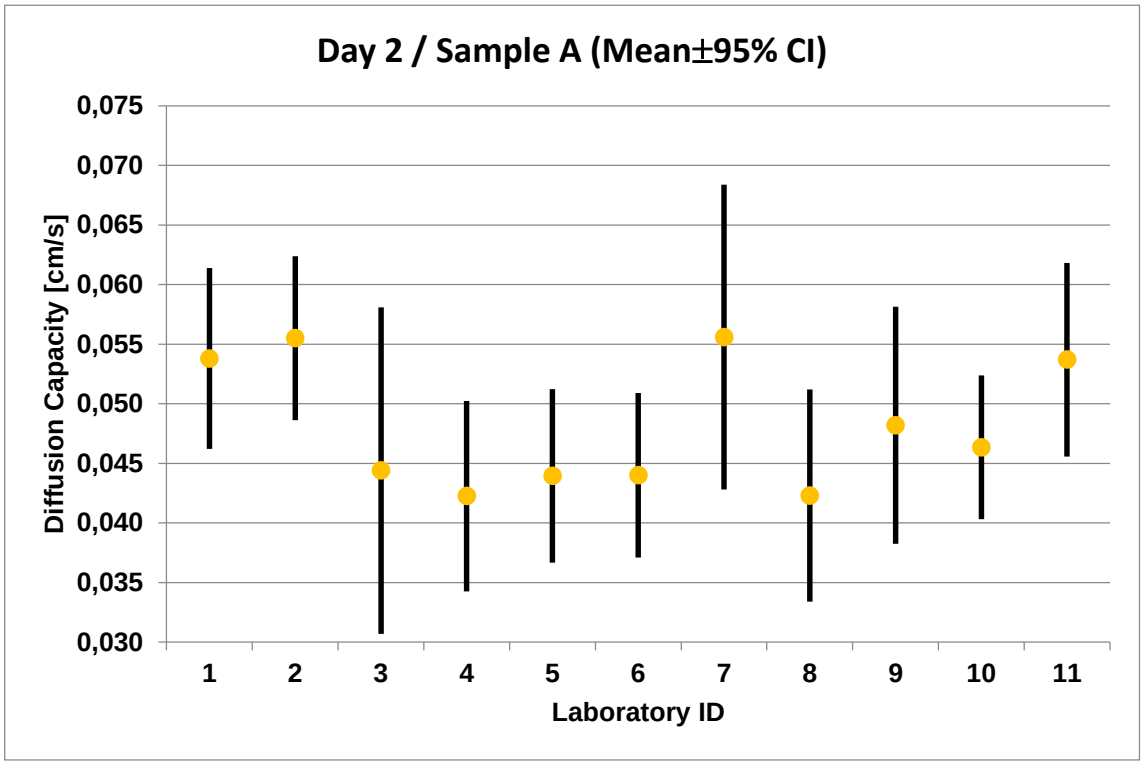


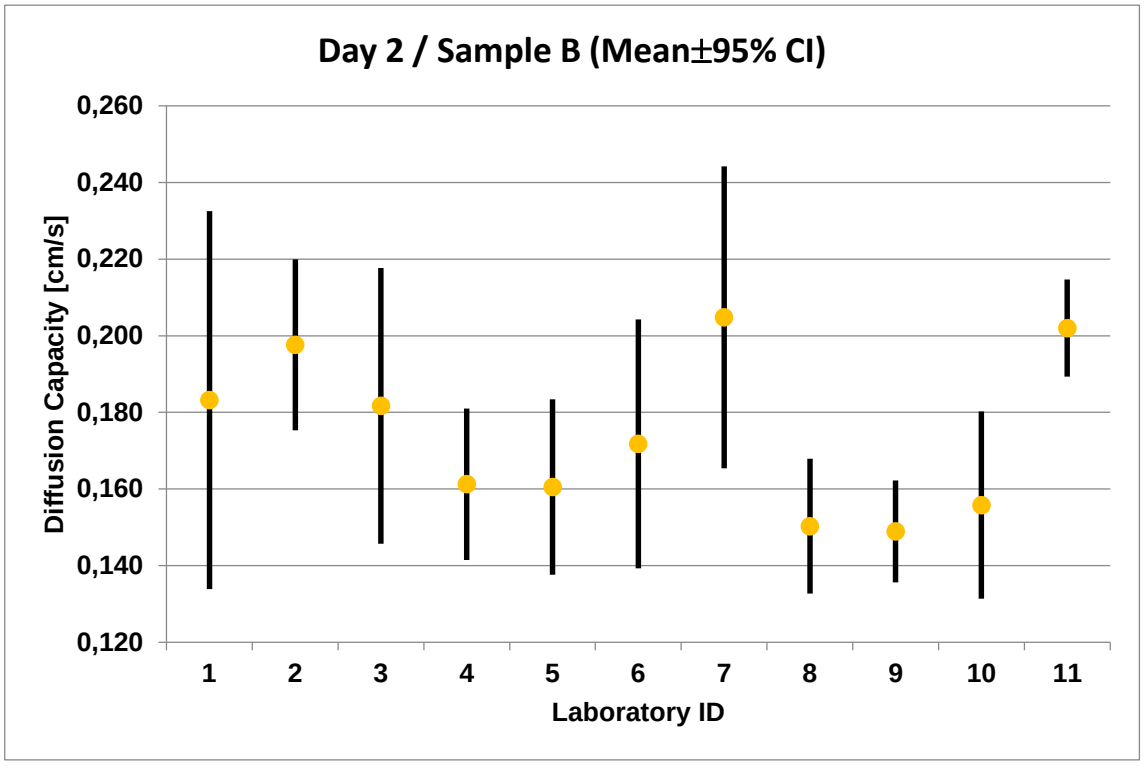
Appendix D.2 – Average and range of the replicate determination of diffusion capacity of LIP bands for samples A and B on day 1 for all laboratories. Outliers are included. The error bars are ±1.96 times the standard deviation of the individual measurements per sample and day obtained by each laboratory.





Appendix D.3 – Average and range of the replicate determination of diffusion capacity of LIP bands for samples A and B on day 2 for all laboratories. Outliers are included. The error bars are ±1.96 times the standard deviation of the individual measurements per sample and day obtained by each laboratory.





Appendix D.3 – Average and range of the replicate determination of diffusion capacity of LIP bands for samples A and B on day 3 for all laboratories. Outliers are included. The error bars are ±1.96 times the standard deviation of the individual measurements per sample and day obtained by each laboratory.