Physical Test Methods Sub-Group

Technical Report

Collaborative Study on Sealing Strength of Pouch Materials

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Statistical Evaluation and Author:
Bernhard Eitzinger, delfortgroup AG, Austria

Project Leader and Study Coordinator:
Danny Gentilcore, Nonwovenn, U.K.

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1. INTRODUCTION

1.1 Purpose and Scope

The Physical Test Methods (PTM) Sub-Group of CORESTA developed a CRM on the Determination of Dry and Wet Sealing Strength of Pouches for Smokeless Tobacco and Derivative Products. This collaborative study was carried out in order to determine repeatability and reproducibility statistics of the newly developed method.

All results will be presented in anonymized form.

1.2 Study Protocol

The test protocol used for this collaborative study is given in Appendix A and will be briefly summarized below.

The protocol contained information on the sample material, the preparation of samples and the procedure for carrying out the measurements. The study participants were required to provide data to identify their laboratory and report all measurement results obtained.

A single type of pouch was distributed to the laboratories and the laboratories were asked to perform ten measurements on the pouches to determine the dry and wet sealing strength in cross direction and in longitudinal direction as specified in the CRM. Thus, each laboratory had to make forty measurements.

The data were collected in an Excel sheet and sent to the study coordinator for further processing and evaluation.

The distribution of sample materials, the protocol and the data collection sheet started in July 2018 and laboratories were asked to report the results until end of August 2018.

1.3 Products and Measurements

The pouches were manufactured using Nonwovenn Snus paper, PT7106 (Bridgwater, UK) by Nordic Snus AB (Vargarda, Sweden) and the resultant pouches were distributed to the participants by Nonwovenn.

The pouch format was taken from Nordic Snus' regular commercial product known as LD Vit, which contains 44 % moisture and was manufactured using a G.D. horizontal flat pouching machine.

The main characteristics of the pouches and the pouch material are listed in Table 1.

Table 1 – Characteristics of the pouch and pouch material

	Parameter	Unit	Value
Pouch LD Original Vit 16g SNUS	Pouch Length	mm	32,3 ± 1
	Pouch Width	mm	17 ± 1
Pouch Material Nonwovenn Snus Paper PT7106	Basis Weight	g/m^2	29,0 ± 2,9
	Thickness	µm	200 ± 20

1.4 Study Participants

In total seven laboratories participated in the study with the list of participants in alphabetical order given in Table 2. A code was assigned to each laboratory and the order of laboratories in Table 2 does not agree with the order of the laboratories in other tables. An eighth laboratory intended to participate in the study, but reported the results too late for being included in this report. ISO 5725-2, which was followed in this study, recommends that at least eight laboratories should take part in a study to determine repeatability and reproducibility statistics. However, even though every effort was made by the study coordinator to recruit a sufficient number of laboratories, only eight participants could be found and only seven submitted data.

Table 2 – List of Participants

Participant Name	Country
British American Tobacco	Germany
Nonwovenn	United Kingdom
Nordic Snus AB	Sweden
Reemtsma Cigarettenfabriken	Germany
Swedish Match AB	Sweden
Tenowo GmbH	Germany
Zhengzhou Tobacco Research Institute	China

2. STATISTICAL EVALUATION

2.1 Raw Data Treatment

In total seven data sets were received and used for statistical analysis. The mean values (MV), the standard deviation (StD) and the number of replicate measurements (N) for each laboratory are reported for the dry sealing strength in cross and longitudinal direction in Table 3 and for the wet sealing strength again in cross and longitudinal direction in Table 4. The full data set containing all individual measured values is given in tabulated form in Appendix B and in graphical form in Appendix C.

Table 3 – Summary data for dry sealing strength per laboratory, outliers included

	Cross Direction (Dry)			Longitudinal Direction (Dry)		
Lab ID	MV	StD	N	MV	StD	N
	N/mm	N/mm		N/mm	N/mm	
1	0,0728	0,0056	10	0,1723	0,0168	10
2	0,1121	0,0102	10	0,2276	0,0270	10
3	0,1240	0,0207	10	0,2570	0,0215	10
4	0,1147	0,0163	10	0,2454	0,0280	10
5	0,1276	0,0224	10	0,1849	0,0258	10
6	0,1541	0,0172	10	0,2661	0,0426	10
7	0,1480	0,0230	10	0,2430	0,0231	10

Table 4 – Summary data for wet sealing strength per laboratory, outliers included

	Cross Direction (Wet)			Longitudinal Direction (Wet)		
Lab ID	MV	StD	N	MV	StD	N
	N/mm	N/mm		N/mm	N/mm	
1	0,0407	0,0072	10	0,1095	0,0341	10
2	0,0468	0,0033	10	0,0848	0,0104	10
3	0,0559	0,0065	10	0,1033	0,0087	10
4	0,0547	0,0117	10	0,0778	0,0039	10
5	0,0392	0,0070	10	0,1094	0,0438	10
6	0,0678	0,0118	10	0,0559	0,0087	10
7	0,0560	0,0070	10	0,0810	0,0074	10

2.2 Outlier Analysis and Removal

Repeatability and reproducibility data were determined following ISO 5725-2, whereby outlier testing according to Cochran's test and Grubbs' test was used. First for each laboratory the maximum and the minimum of the ten replicates was checked for being an outlier according to Grubbs' test ("within-laboratory Grubbs' test"). Any outliers that were detected were removed from the data set, but the other replicates remained for further calculations.

Second the standard deviation of each laboratory's results were compared to the total standard deviation by Cochran's test to detect any laboratories that had an unusually high standard deviation. If an outlier was detected this data set was removed from further analysis.

In a third step Grubbs' test ("between-laboratory Grubbs' test") was used to check if the mean value of a laboratory was exceptionally high or low compared to the other laboratories. All outliers were removed. These two outlier tests were repeated as often as was necessary until no further outliers appeared, however in this study a single round of outlier elimination was sufficient. Generally the number of outliers was low so that there was no danger of removing too many data sets.

After elimination of outliers global statistics, in particular mean values and standard deviations, were calculated and the repeatability and reproducibility statistics were determined.

For dry sealing strength in cross direction and for wet sealing strength in longitudinal direction the maximum value obtained by laboratory 1 qualified as an outlier based on within-laboratory Grubbs' test. Laboratory 5 had an unusually high standard deviation when measuring wet sealing strength in longitudinal direction. The minimum value in this data set qualified as a straggler under within-laboratory Grubbs' test, but even after elimination of this straggler the standard deviation remained too high and thus the entire data set was excluded based on Cochran's test.

No outliers were excluded based on between-laboratory Grubbs' test, so that in total two individual data points and one data set had to be excluded from the analysis of repeatability and reproducibility.

2.3 Robust Mean Values and Standard Deviations

After the removal of outliers robust mean values and between-laboratory standard deviations were calculated using ISO 5725-2. The results are given in Table 5. For comparison results obtained with outliers are also shown in Table 5.

Table 5 – Robust mean values (MV), between-laboratory standard deviations (SDb) and within-laboratory standard deviation (SDw) of pouch sealing strength

	Outliers included				Outliers excluded			
	MV	SDb	SDw	N	MV	SDb	SDw	N
	N/mm	N/mm	N/mm		N/mm	N/mm	N/mm	
Dry, Cross Direction	0,1219	0,0268	0,0175	7	0,1217	0,0273	0,0174	7
Dry, Longitudinal Direction	0,2280	0,0360	0,0274	7	0,2280	0,0360	0,0274	7
Wet, Cross Direction	0,0516	0,0100	0,0083	7	0,0516	0,0100	0,0083	7
Wet, Longitudinal Direction	0,0888	0,0198	0,0221	7	0,0837	0,0170	0,0092	6

2.4 Evaluation of Repeatability and Reproducibility

Based on the robust mean value and the between-laboratory and within-laboratory standard deviations repeatability and reproducibility statistics were calculated according to ISO 5725-2. The results are given in Table 6. The Table shows the standard deviation (SD), the limit and the coefficient of variation (CoV) relative to the global mean value for repeatability and reproducibility.

Table 6 – Repeatability and reproducibility statistics for pouch sealing strength

	Sealing Strength					
	Repeatability			Reproducibility		
	StD	Limit	CoV	StD	Limit	CoV
	N/mm	N/mm	%	N/mm	N/mm	%
Dry, Cross Direction	0,0174	0,0493	14,33	0,0319	0,0903	26,22
Dry, Longitudinal Direction	0,0274	0,0776	12,04	0,0444	0,1256	19,48
Wet, Cross Direction	0,0083	0,0234	16,03	0,0127	0,0360	24,69
Wet, Longitudinal Direction	0,0092	0,0260	10,96	0,0191	0,0541	22,83

3. CONCLUSIONS

The number of outliers observed in this collaborative study is rather low, which shows that laboratories are in general able to reliably perform the measurement of sealing strength as described in the CRM. The coefficient of variation for repeatability is between about 11 % and 16 %, which is sufficient for practical purposes. The coefficient of variation for reproducibility is between about 20 % and 26 % and is thus by a factor between 1,5 and 2,0 higher than for the repeatability, which is not unusual compared with other physical test methods.

It has to be noted that the testing according to the CRM is destructive, thus the repeatability and reproducibility statistics contain the sample-to-sample variability. Experience from inter-laboratory studies carried out during development of the test method have shown that the sample-to-sample variability is a substantial contribution to the overall variability of the method. Also the sample preparation is an important contributor to variability, particularly for the measurement of wet sealing strength. The instruments used for measuring the sealing strength in this study are commercially available instruments and are known to have an accuracy which is an order of magnitude less than the repeatability determined in this study.

The repeatability and reproducibility statistics are provided for an individual measured value. As recommended in the CRM, normally ten measurements should be carried out to obtain the sealing strength. It can be expected that the variability of an average of ten measured values is by a factor of $1/\sqrt{10}$ lower than that of an individual value, thus the repeatability coefficient of variation of an average of ten values may be expected to be 3,5 % to 5,0 % and the reproducibility coefficient of variation of the average of ten values will be in the range from 6 % to 8 %.

4. REFERENCES

- CORESTA Recommended Method N° 90, Determination of Dry and Wet Sealing Strength of Pouches for Smokeless Tobacco and Derivative Products Definitions and Measurement Principles

5. APPENDICES

APPENDIX A – Protocol

The following protocol was used in this collaborative study based on the details described in the CRM.

The contents of each pouch sample must be removed prior to cutting the sample to 10 mm ±1 mm for the long seam and as close to the edges as is feasible for the cross seam. Record the widths of each sample.

Condition the samples at 22 °C ± 1 °C and 60 % ± 3 % RH for 24 hours prior to testing. This conditioning step is not required when conducting the 'wet' measurements.

The jaw separation must be set at 15 mm ± 0,1 mm with a pull speed of 20 mm/min. Record both values and use a pre-load of 0,1 N if possible.

Insert the sample to be tested into the jaws and measure the average load (if software allows) as well as the maximum load when testing the cross seam. When testing for the long seam then only measure the maximum load. Record values.

Sealing strength is measured in N/mm.

In the case of a wet measurement, dip the pouch sample in demineralised water for 60 minutes prior to testing either the long or cross seams. Conduct the test as per the dry measurements.

Measure 10 replicates.

APPENDIX B – Individual Raw Data

Appendix B.1: Dry sealing strength in cross direction, the outlier (Grubbs' test) is marked in yellow.

	Dry Sealing Strength in Cross Direction									
	N/mm									
Lab ID	1	2	3	4	5	6	7	8	9	10
1	0,0718	0,0871	0,0735	0,0729	0,0712	0,0724	0,0724	0,0700	0,0647	0,0724
2	0,1080	0,1133	0,0920	0,1047	0,1200	0,1160	0,1233	0,1040	0,1147	0,1253
3	0,1073	0,1369	0,0787	0,1353	0,1543	0,1333	0,1358	0,1218	0,1176	0,1187
4	0,1367	0,1217	0,1122	0,1067	0,1344	0,1100	0,0944	0,1028	0,1344	0,0939
5	0,1380	0,1013	0,0833	0,1373	0,1427	0,1340	0,1120	0,1367	0,1600	0,1307
6	0,1320	0,1430	0,1680	0,1390	0,1650	0,1620	0,1430	0,1820	0,1370	0,1700
7	0,1600	0,1500	0,1600	0,1400	0,1100	0,1400	0,1300	0,1700	0,1300	0,1900

Appendix B.2: Dry sealing strength in longitudinal direction

	Dry Sealing Strength in Longitudinal Direction									
	N/mm									
Lab ID	1	2	3	4	5	6	7	8	9	10
1	0,1723	0,1786	0,1632	0,1883	0,1344	0,1855	0,1741	0,1647	0,1939	0,1685
2	0,2120	0,2280	0,1840	0,2540	0,2790	0,2500	0,2240	0,2130	0,2090	0,2230
3	0,2557	0,2590	0,2820	0,2580	0,2790	0,2820	0,2660	0,2330	0,2300	0,2250
4	0,2072	0,2838	0,2264	0,2408	0,2592	0,2016	0,2404	0,2472	0,2775	0,2696
5	0,2230	0,1960	0,1540	0,1940	0,1390	0,2020	0,2010	0,1640	0,1770	0,1990
6	0,2710	0,3360	0,2180	0,2480	0,2990	0,2330	0,2050	0,3100	0,2490	0,2920
7	0,2500	0,2300	0,2700	0,2200	0,2600	0,2600	0,2600	0,2600	0,2100	0,2100

Appendix B.3: Wet sealing strength in cross direction

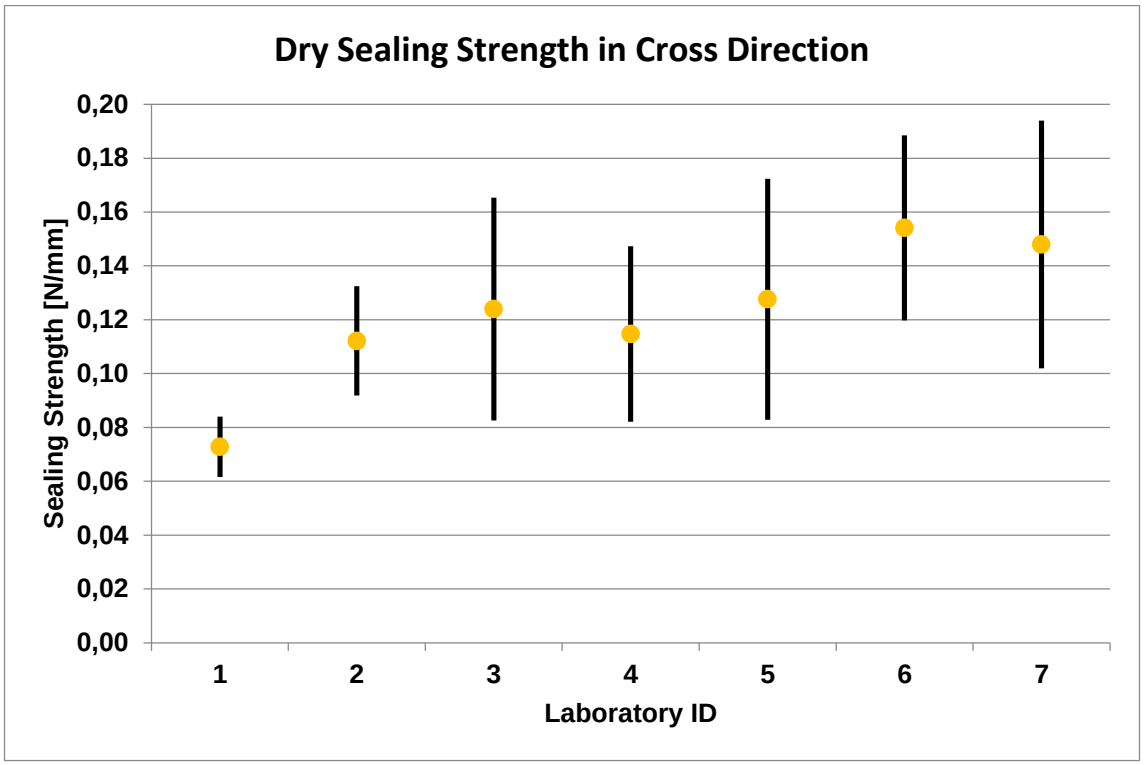
	Wet Sealing Strength in Cross Direction									
	N/mm									
Lab ID	1	2	3	4	5	6	7	8	9	10
1	0,0506	0,0388	0,0312	0,0300	0,0412	0,0441	0,0524	0,0376	0,0418	0,0394
2	0,0427	0,0493	0,0500	0,0460	0,0500	0,0487	0,0507	0,0440	0,0420	0,0447
3	0,0515	0,0620	0,0493	0,0587	0,0513	0,0520	0,0633	0,0465	0,0649	0,0590
4	0,0661	0,0606	0,0756	0,0445	0,0533	0,0497	0,0622	0,0387	0,0412	0,0553
5	0,0375	0,0352	0,0235	0,0480	0,0449	0,0439	0,0439	0,0402	0,0346	0,0407
6	0,0446	0,0593	0,0780	0,0650	0,0738	0,0627	0,0584	0,0816	0,0755	0,0793
7	0,0400	0,0600	0,0600	0,0600	0,0500	0,0600	0,0500	0,0600	0,0600	0,0600

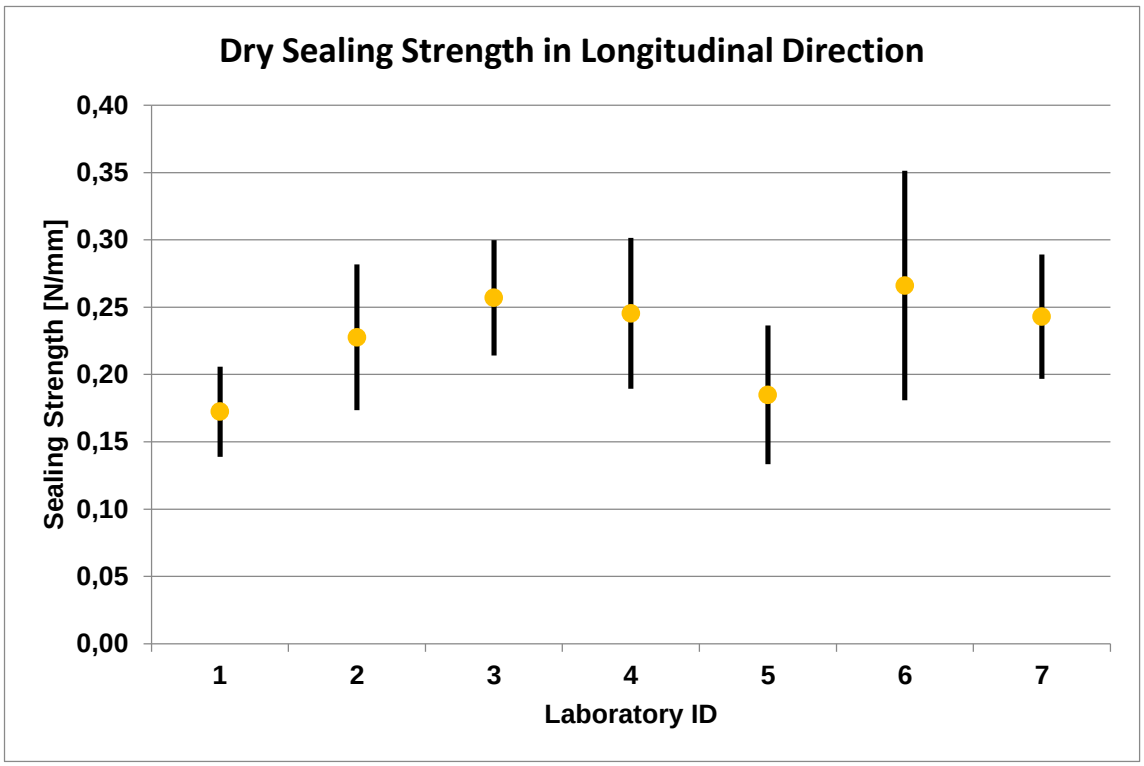
Appendix B.4: Wet sealing strength in longitudinal direction, the outliers are marked in yellow.

Lab ID	Wet Sealing Strength in Longitudinal Direction N/mm									
	1	2	3	4	5	6	7	8	9	10
1	0,1126	0,1021	0,1999	0,0905	0,0946	0,0932	0,1202	0,0758	0,1088	0,0971
2	0,0740	0,0900	0,0960	0,0770	0,0820	0,0910	0,0820	0,0780	0,0730	0,1050
3	0,0960	0,0950	0,0940	0,0970	0,1070	0,1090	0,1080	0,1220	0,1000	0,1050
4	0,0804	0,0848	0,0764	0,0800	0,0736	0,0796	0,0720	0,0736	0,0784	0,0796
5	0,1010	0,1201	0,0901	0,1110	0,1020	0,0078	0,1023	0,1680	0,1380	0,1540
6	0,0380	0,0450	0,0630	0,0560	0,0590	0,0620	0,0640	0,0550	0,0640	0,0530
7	0,0900	0,0800	0,0900	0,0800	0,0900	0,0800	0,0800	0,0700	0,0700	0,0800

APPENDIX C – Sealing Strength (Diagrams)

Appendix C.1: Dry Sealing Strength





Appendix C.2: Wet Sealing Strength