STICKINESS TESTING METHOD FOR CONTEST INSTRUMENTS RECOGNITION BY ICCTM

1. Instrument

- **Instrument / Detector :**
  Stickiness Testing Method in Contest Instruments, Mesdan, S.p.A., Italy
- **Target type of recognition:**
  Full Method Recognition for stickiness grade measurement
- **Area of recognition:**
  Testing for spinning mills, trading and research purposes

2. General description

CONTEST - Stickiness Tester is a fully automatic high speed thermodetector designed to measure and classify the sticky points in cotton fiber material for large mass testing in compliance with the international standard **UNI EN 14278-3**.

It is embedded in two different testing equipment[1], such as.

- ***Mesdan* CONTEST-F**
- ***Mesdan* CONTEST-S**

Figure 1: *Pictures of CONTEST-F (left) and CONTEST-S (right).*

[1] The **Stickiness Tester** has been supplied in other instruments over the years, such as *Mesdan* CONTEST and *Loepfe* FIBERMAP.

3. Target group

CONTEST - Stickiness Tester has been developed to assist spinners to achieve consistent yarn quality standards whenever cotton lots are affected by stickiness contamination. In addition, it can further be of interest for cotton traders and suppliers in order to monitor and level out the stickiness in the season crop. Concerning research laboratories, instead, the **Stickiness Tester** can be used to grade and compare the stickiness content among different reference samples.

4. Principle of Operation

The function principle to detect sticky points follows the international standard **UNI EN 14278-3**.

For each specimen, the tester is fully automatic and easy to use: the operator prepares a 3.5 ± 0.2 g sample by using an external balance[2] and places the sample on the conveyor belt, which feeds a micro card system which produces a fine fiber web of about 10 meter in length. The web is then delivered to the **Stickiness Tester** and pressed in between two metal drums revolving in opposite direction and constantly heated at 35.0 ± 1.0°C (after a warming up period). The heating of the drums is achieved by a special (patented) friction system of two moveable and adjustable brushes, which allows the drums to rapidly reach the correct starting temperature, and remain stable during the test, thus ensuring measurement reliability and accuracy.

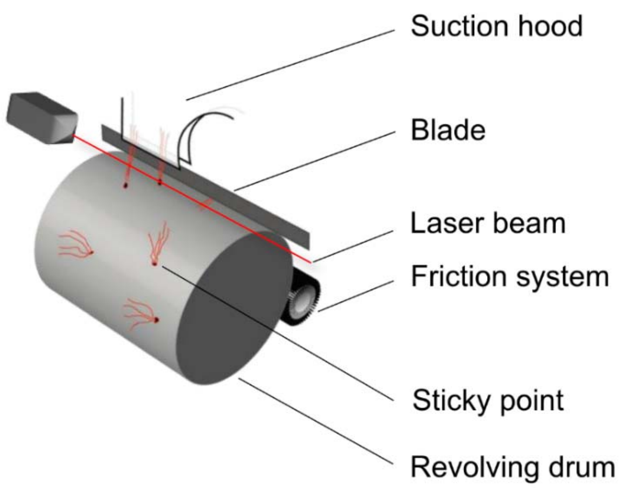


Figure 2: *Schematic picture for the principle of operation of the Stickiness Tester.*

[2] The external balance is provided with the instrument.

The setting of 35.0°C allows the system to be close to the typical temperature of the machines in the spinning mill[3], in order to provide an evaluation of the "effective stickiness", as defined as "*the tendency of cotton fibers to stick to textile working surfaces*"[4]. The drum temperature is monitored in real time by an infrared sensor for non-contact measurements.

While no sticky fibers (i.e., the residual web) are sucked away, the sticky deposits adhering to the drum surface are optically inspected by a laser beam, as well as counted and divided into 5 count classes, which are related to the number of fibers attached to the sticky point (more information in chapter 7.2).

During the test, the surface of the drums are automatically cleaned by a system of rotating brushes, as well as a blade mechanism, which prevents double counting and/or contamination between subsequent measurements. At the end of each test, a check of the stability of the laser signals ensures the proper cleaning of the drum surfaces and warns the user in case of need.

5. Usefulness/Benefits

CONTEST – Stickiness Tester is a fully automatic high-speed laboratory tester which is very useful to the whole cotton industry from cotton production to processing. Indeed, stickiness is a variable contaminant which has economic impacts both in the form of discount that is applied to sticky cotton (supplier/trader), as well as additional processing costs (spinning mill).

It combines together sample preparation, which is conventional for spinning processes, like a cotton web formation produced by a carding unit, with a thermo-detection principle based on fiber adhering on metal surfaces, which are heated at temperatures typical of machines in the spinning mill[3]. In this way, the **Stickiness Tester** provides an effective measurement of stickiness on the basis of what the European Committee for Standardization defined in 2004 as "*the tendency of cotton fibers to stick to textile working surfaces*".

In spite of this method rules out any determination of sugar contents, which are at the origin of the stickiness phenomenon (chemical approach), the **CONTEST** instruments provide a high speed detector for large mass testing, which allows a cotton bale characterization before entering into the spinning process, thus offering the possibility to handle the stickiness contamination in due time.

[3] E. F. Hequet and N. Abidi, "Sticky Cotton: Measurements and Fiber Processing", Texas Tech University Press (2006), p.137, Table 11.
[4] European Committee for Standardization, 2004: UNI EN 14278-1, UNI EN 14278-2, UNI EN 14278-3
"Textiles - Determination of cotton fibre stickiness - Part 1-3"

In fact, cotton bales, which are affected by high levels of stickiness, may seriously compromise the spinning process, increasing the production costs due to excessive wear of machinery parts, additional cleaning and maintenance operations and even causing machinery blockages. Moreover, it is well known that the presence of stickiness in cotton can reduce the quality of the yarn (i.e., decreasing evenness and increasing nep content and hairiness).

As the stickiness phenomenon is generally unpredictable from season to season, the **Stickiness Tester** provides a tool to manage the bale lots according to the stickiness measurements, thus reducing the risk of issues during the spinning process by properly setting up the bale blend between sticky cotton with non-sticky cotton.

Since it is really unlikely that a single critical threshold for stickiness may be found among the spinning mills all over the world[5], it is clear that every potential customer of **CONTEST – Stickiness Tester** should determine their own critical threshold for stickiness which is typical for their mill, combined together with the equipment, experience and ability of the staff, as well as the fibers to be processed.

The **Stickiness Tester** is embedded with two instruments:

- ***Mesdan*** **CONTEST-S**, i.e. measuring only stickiness;
- ***Mesdan*** **CONTEST-F**, i.e. a high volume cotton testing equipment, which integrates the stickiness evaluation with the other measurements useful for the classification of cotton: UHML, UI, Strength, Elongation, SFI, Moisture, Rd, +b, Color Grade, Trash Count, Trash Area, Leaf, Micronaire, Maturity Ratio, Fineness.

If the sample is properly prepared (in weight, length and shape) as per the instructions, the **Stickiness Tester** is almost operator-independent by virtue of its card unit, which converts each operator-prepared sample into a fiber web (refer to Chapter 16.3 for a detailed analysis of the operator influence).

6. Application range of testing

CONTEST - Stickiness Tester is specifically designed to process (web formation) and guarantee the reliability of results (stickiness detection) for 100% raw cotton fibers. If required, it is also suitable for measuring slivers of 100% cotton fibers, even although the carding performance/efficiency of the instrument is not guaranteed at the same level as with raw material.

[5] J. P. Gourlot and R. Frydrych, "Improvement of the Marketability of Cotton Produced in Zones Affected by Stickiness. Final report of the Project CFC / ICAC / 11", Technical Paper N.17, Common Fund for Commodities, (2001).

The range of application for sample processing (web formation) is from short staple to extra-long staple fibers (i.e., 20 mm < UHML < 40 mm). The testing range of application, instead, cannot be precisely defined because of the lack of a recognized standard unit of reference: stickiness can be detected from zero level (non-sticky cotton) up to high level (sticky cotton).

The preparation of samples from raw cotton is manual as per the instructions of the weight (3.5 ± 0.2 g) and length (30 ± 3 cm). The shape of the sample has to be sliver-like, as well as homogeneous along the length.

6.1 Range of recognition

In the recognition only 100% raw cotton fibers are used within the staple range of 20 mm < UHML < 40 mm to ensure a good carding performance. The testing parameter, which is considered, is the Stickiness Grade (St grade) with the maximum stickiness range of detectability (0 < Stickiness grade < Max ever[6]).

6.2 Additional range

Other parameters, which are not considered in the recognition, are available with the **Stickiness Tester**, such as the Total Stickiness Count (St Cnt), the Stickiness Average Size (St Size), and the Stickiness Count per Class 1-5 (see Chapter 7.2).

7. Result parameters and definitions

7.1 Recognized parameters

The parameter under recognition is the Stickiness Grade (St Grade), which is an evaluation of stickiness involving the sticky point count/g and their classes.

7.2 Other parameters

Additional parameters provided by the **Stickiness Tester**:

- Total Stickiness Count (St Cnt) [1/g] : the total number of sticky points;
- Stickiness Average Size (St Size) [a.u.] : the mean class of stickiness count;
- Stickiness Count 1 [1/g] : the number of sticky points of class 1;
- Stickiness Count 2 [1/g] : the number of sticky points of class 2;
- Stickiness Count 3 [1/g] : the number of sticky points of class 3;
- Stickiness Count 4 [1/g] : the number of sticky points of class 4;
- Stickiness Count 5 [1/g] : the number of sticky points of class 5;

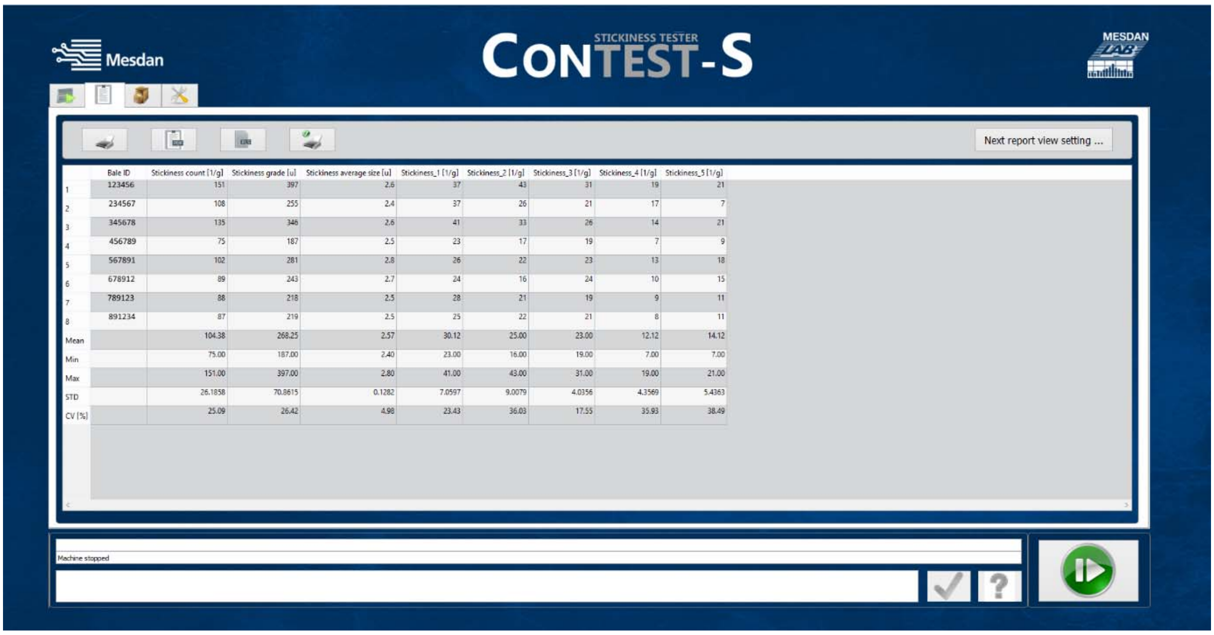
[6] The highest result of stickiness grade (750) so far recorded among all the Stickiness Testers participating in the International Round Test of ICCTM-ITMF from 2017-1 up to 2019-2.

The count classes 1-5 are related to the number of fibers attached to the sticky point. Depending on the decrease of the laser signal, the sticky point is classified in class 1, if the number of fibers are very few (approximately less than 3 fibers), up to class 5, if the number of fibers is quite large (approximately greater than 30-40 fibers). Thereby, this classification of sticky points is a sort of indirect evaluation of size, since it is reasonable to argue that the number of fibers sticked on an honeydew deposit are increasing with the point size[7].

The choice to use 5 classes is not based on the fiber count, but rather it is determined by the equal division of the whole detecting range of the optical sensor (i.e., laser + photodiode) in such a way that each class has an amplitude of detection (i.e. voltage gap) sufficiently larger than the signal noise. Then, it is not claimed to discriminate single fibers nor to count the exact number of fibers nor to directly quantify the real dimension of sticky points, but simply to effectively classify the sticky point within certain discretized classes (i.e., range of fiber number) on the basis of the sensitivity of the optical sensor provided, thus exploiting a technique (i.e. the combination of a light source with a photodiode) which is commonly used in several fiber detectors.

7.3 Printout example

An example of stickiness results, which are displayed on the screen at the end of the test session or printed out in exportable files.



	Bale ID	Stickiness count [1/g]	Stickiness grade [u]	Stickiness average size [u]	Stickiness_1 [1/g]	Stickiness_2 [1/g]	Stickiness_3 [1/g]	Stickiness_4 [1/g]	Stickiness_5 [1/g]
1	123456	151	397	2.6	37	43	31	19	21
2	234567	108	255	2.4	37	26	21	17	7
3	345678	135	346	2.6	41	33	26	14	21
4	456789	75	187	2.5	23	17	19	7	9
5	567891	102	281	2.8	26	22	23	13	18
6	678912	89	243	2.7	24	16	24	10	15
7	789123	88	218	2.5	28	21	19	9	11
8	891234	87	219	2.5	25	22	21	8	11
Mean		104.38	268.25	2.57	30.12	25.00	23.00	12.12	14.12
Min		75.00	187.00	2.40	23.00	16.00	19.00	7.00	7.00
Max		151.00	397.00	2.80	41.00	43.00	31.00	19.00	21.00
STD		26.1858	70.8615	0.1282	7.0597	9.0079	4.0356	4.3569	5.4363
CV [%]		25.09	26.42	4.98	23.43	36.03	17.55	35.93	38.49

Figure 3: *An example of results.*

[7] E. Hequet, R. Frydrych, and M. Watson, "The use of the High Speed Stickiness Detector on a large range of cotton coming from different countries", *In* Proc. Beltwide Cotton Prod. Res. Conf., Memphis, TN. 4-8 Jan. 1997.

MESDAN LAB CONTEST

Sample ID	SUDANESE	Gin ID	Lot ID	Origin	Producer	Operator
SUDANESE		12345	AB123	SUDAN	SUPPLIER 1	OPERATOR 1

Test data and time : 2019/12/17 11:00:56

Bale ID	Stickiness count [1/g]	Stickiness grade [u]	Stickiness average size [u	Stickiness_1 [1/g]	Stickiness_2 [1/g]	Stickiness_3 [1/g]	Stickiness_4 [1/g]	Stickiness_5 [1/g]
123456	151	397	2.6	37	43	31	19	21
234567	108	255	2.4	37	26	21	17	7
345678	135	346	2.6	41	33	26	14	21
456789	75	187	2.5	23	17	19	7	9
567890	102	281	2.8	26	22	23	13	18
678912	89	243	2.7	24	16	24	10	15
789123	88	218	2.5	28	21	19	9	11
891234	87	219	2.5	25	22	21	8	11
Mean	104.38	268.25	2.57	30.12	25.00	23.00	12.12	14.12
Min	75.00	187.00	2.40	23.00	16.00	19.00	7.00	7.00
Max	151.00	397.00	2.80	41.00	43.00	31.00	19.00	21.00
STD	26.1858	70.8615	0.1282	7.0597	9.0079	4.0356	4.3569	5.4363
CV [%]	25.09	26.42	4.98	23.43	36.03	17.55	35.93	38.49

Figure 4: *An example of an exportable report.*

7.4 Parameter calculation

The Stickiness Grade (St Grade) parameter is calculated by means of the formula:

$$St\ Grade = \sum_{i=1}^{5} (i \cdot S_i) \tag{1}$$

with S_i is the number of sticky points of class i. In this way, the St Grade combines together two different information about stickiness: counts and their indirect size evaluation, so called class[8] for CONTEST. Thereby, St Grade is a sum of counts weighted on the classes.

This approach takes into account the possibility that the sticky point size may significantly affect during the spinning process, thus leading to different evaluation of stickiness with respect to the simple count of sticky deposits[7]. Moreover, the interest to follow this approach is confirmed by the empirical observation of increasing counts in higher dimension classes along with the total count of sticky points in CONTEST, as well as similar outcome by using H2SD about the size variability independently of the count[7].

8. Testing procedure

8.1 Number of tests

Since the stickiness phenomenon is highly affected by variability[9], it is unlikely to achieve a reasonable precise evaluation on the basis of a single test or few samples,

[8] Refer to paragraph 7.2 for a detailed description of classes related to size.

[9] E. Hequet, T. J. Henneberry, and R. L. Nichols, "Sticky Cotton: Causes, Effects, and Prevention", USDA Technical Bulletin n.1915 (2007)

especially when the level of stickiness is not clearly high. In this situation, the problem of the number of tests to be carried out is thoroughly connected with the sampling, as well as the within-variability of the material to be tested, which requires a certain own experience by the user's side.

However, if a limited sample (< 200 g) is considered, the minimum number of tests per sample, which is recommended, is between 3 and 6, depending on the level of stickiness[10]. All the statistical parameters (Average, Standard Deviation, Coefficient of Variation) should be considered to properly evaluate the sample.

8.2 Description of sample preparation

The preparation of samples from raw cotton is manual, as per the following instructions:

1. manually remove before testing only large foreign particles, such as large pieces of bark and entire seeds, in addition to fluff the fibers of the test specimen to eliminate dense clumps of fibers or knotty balls;
2. weight a portion of 3.5 g with an acceptable tolerance of ±0.2 g;
3. manually stretch the fibers, as to distribute them and to reach the required sample length (30 ± 3 cm);
4. gently roll the material in order to obtain a compact cylindrical shape (as shown Figure 5);
5. insert the sample onto the conveyor belt and start the test.

Estimated time for sample preparation: 30 seconds

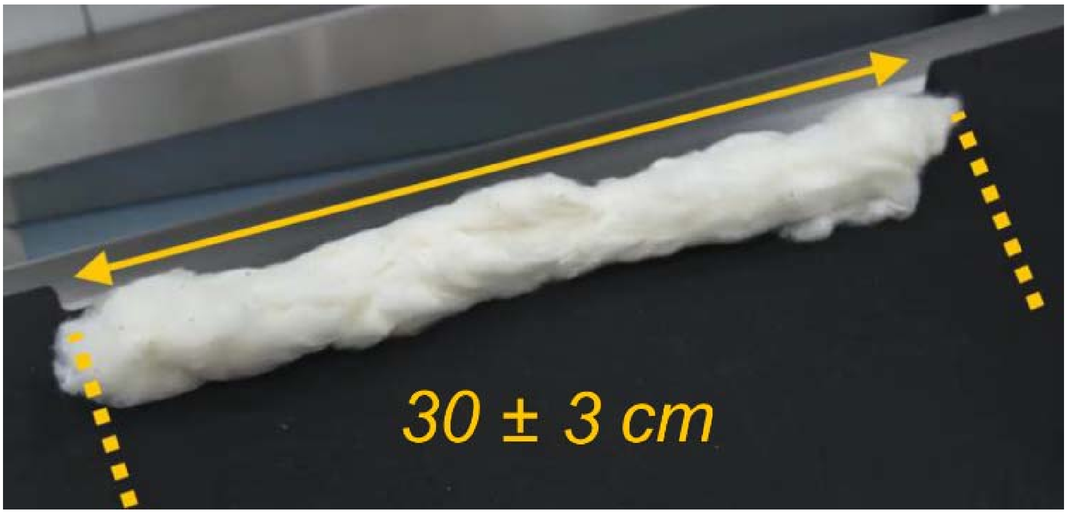


Figure 5: *An example of sample, which is ready to be tested, with the proper length (30 ± 3 cm), weight (3.5 ± 0.2 g) and shape.*

[10] Higher level of stickiness, lower the number of tests and vice versa.

8.3 Necessary surrounding

Atmospheric conditions

As the stickiness measurement is strongly influenced by moisture (i.e., lower relative humidity, equals lower stickiness detection), it is essential to pre-conditioning and test the samples, in a standard atmosphere in compliance with ASTM D1776 (21 ± 1°C, 65% ± 2% RH) for at least 24 hours.

9. Testing preparation time, sample preparation time and testing time

In order to reach the proper drum temperature (35°C), a warming up of about 10 minutes is required. The time to perform a test changes depending on the instrument type, in which the **Stickiness Tester** is provided:

CONTEST-S:

- Sample preparation: 30 sec.
- Sample transfer by the conveyor belt : 20 sec
- Testing time: 30 sec

CONTEST-F:

- Sample preparation: 30 sec.
- Sample transfer by the conveyor belt : 30 sec
- Testing time: 60 sec

Total time (sample preparation + testing) required for a single test:

- **CONTEST-S:** 80 seconds
- **CONTEST-F:** 2 minutes

Total time (preparation of samples + testing) required for 6 consecutive tests:

- **CONTEST-S:** 5 minutes (avg time per sample: 50 sec)
- **CONTEST-F:** 9 minutes (avg time per sample: 1 min 30 sec)

10. Reference Methods and Reference Materials

Today several techniques are available for stickiness detection, based on different approaches (i.e., chemical, physical and mechanical). Nevertheless, according to the latest outcomes[11], it is unlikely that a single reference method will be identified in the short term due to the large variability of results between different devices and/or

[11] Stickiness Round Test conducted by the Stickiness Task Force of the ITMF-ICCTM in 2014-2019.

different laboratories. In addition, it has still to be determined whether all methods are able to detect the same stickiness, interpreted as "*the tendency of cotton fibers to stick to textile working surfaces*". Finally, the efforts still in progress[12] for preparing and producing reference materials (which should be certified in the stickiness level on a common scale and within a certain expiration time) have not yet provided calibration cottons which would make the search for a reference method less complex.

10.1 Reference Methods

Considering the above mentioned, the only techniques, which may be reasonably taken as reference for the **CONTEST- Stickiness Tester**, should be all the techniques that share a similar principle of functioning, such the H2SD and SCT (thermodetectors), as well as the MINICARD (mechanical detector).

10.2 Calibration Materials

The HarCoStiC project[12] has been the only task force involved in the research of reference materials for stickiness. Nevertheless, certified cottons are not available until now, on the basis of which it should be possible to calibrate every stickiness detector with a single common scale.

In this situation, it is strongly recommended to customers to identify some production cottons, which are significant for stickiness in their own spinning process, and to keep them as *"reference materials"* to periodically check the stability of the obtained results. Before using such *reference materials*, they should establish reference values for these materials by accurate sampling of different portions of material and a sufficient number of tests per portion in order to determine a reliable mean stickiness value for the material, as well as its level of variability.

10.3 How to calibrate

Due to all the aforementioned points, there is currently no calibration procedure for the **CONTEST – Stickiness Tester** by using reference materials, which are related to a common reference scale for stickiness. In this condition, the **Stickiness Tester** is set by the manufacturer on its own scale (CONTEST St Grade) and the reliability of instruments during time is monitored and guaranteed by the manufacturer's after sales service.

[12] The HarCoStiC project, which started in 2016 under the conduction of J.P. Gourlot, was designed for the creation of universal reference materials for harmonization of cotton stickiness characterization. It is still active and in progress.

Nevertheless, it is possible to monitor significant deviations of the calibration by using the *reference materials*, described in the previous paragraph, and possibly contact the assistance service for a more accurate investigation or a retuning of the tester.

11. Applicable Standard test method

UNI EN 14278-3 : Textiles - Determination of cotton fibre stickiness - Part 3: Method using an automatic thermodetection rotating drum device

12. Test result Repeatability / Reproducibility

12.1 Repeatability of the basic system / basic test

The reliability of the system is real time checked by monitoring the temperature of the drums by means of infrared sensors for non-contact measurements, as well as the stability of the optical signals between subsequent tests.

12.2 Repeatability on same specimen

Similarly, as with other fiber tests, also in the stickiness detection is not possible to repeat the measurement twice by using exactly the same sample. In fact, the peculiarity of the function principle of the **Stickiness Tester** (i.e. the web formation by a micro card unit) and the subsequent detection of sticky points (which are first attached to the metal surface of the drums, then optically inspected and finally removed and wasted) completely change the sample at the end of testing, thus making the repetition unfeasible.

In this case, repeatability is provided by means of the standard deviation, SD, and the coefficient of variation, CV%, between different days of testing on the same sample (but not exactly the same specimen), as described in details in the next paragraph 12.3. Table 1 summarizes here in advance the results of the next paragraph for 5 different samples A-E, considering two different ways of evaluation: SD(1) is the standard deviation of all the results performed during 15 days of testing, whereas SD(6) is the average of all the SD for single days based on 6 tests. By taking into account the mean values for all the results (90 per sample in 15 days), CV%(1) and CV%(6) are calculated respectively from SD(1) and SD(6).

COTTON SAMPLE	MEAN all reults (90)	SD(1)	CV%(1)	SD(6)	CV%(6)
A	412.0	76.5	18.6	74.8	18.2
B	25.3	16.1	63.8	13.8	54.4
C	240.2	70.7	29.4	61.6	25.7
D	571.9	97.0	17.0	91.4	16.0
E	119.5	49.8	41.7	45.2	37.9

Table 1: *Repeatability on the same sample (not the same specimen) during 15 days of testing on 5 different samples A-E with the same instrument. The SD calculation as follows: (1) on all results of 15 days (= 90 tests), (2) the average on all SD for each day based on 6 tests.*

As evidenced, the repeatability of the Stickiness Grade depends on the sample to be tested, lowering CV% with an increase of Stickiness Grade, as displayed in Figure 6. However, this behavior seems to be common to other stickiness methods, which can be considered as reference for the Stickiness Tester, as shown in Figure 29. A more detailed analysis is provided in paragraph 16.

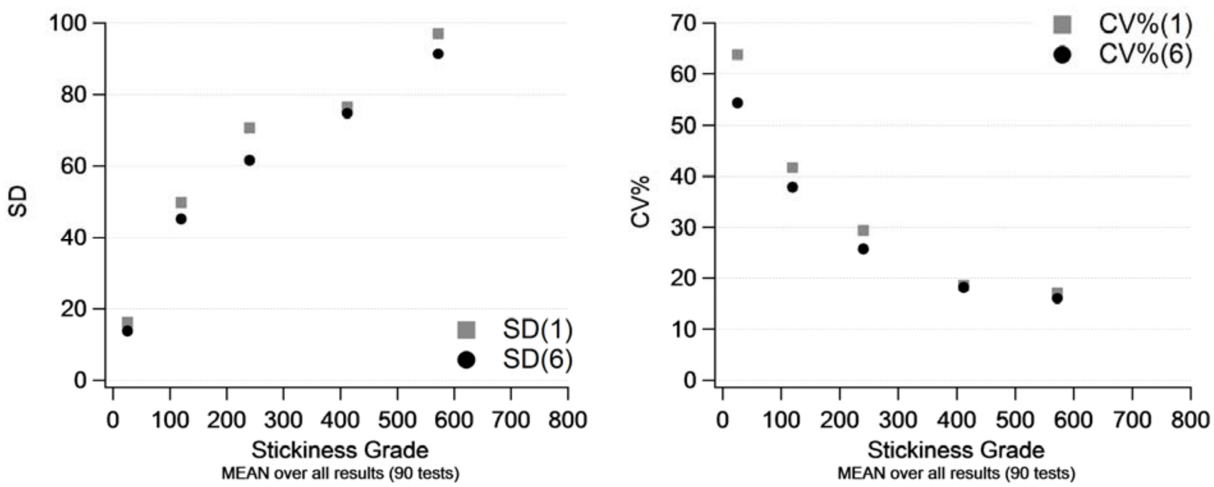


Figure 6: *(left) Standard Deviation, SD, and (right) Coefficient of Variation, CV%, for the following calculations: (1) on all results of 15 days (= 90 tests), (2) the average on all SD for each day based on 6 tests.*

12.3 Repeatability on similar test materials

The repeatability of test results of the **Stickiness Tester** was investigated by using 5 different samples[13] (A-E), which were selected in order to cover the entire range of detection, as demonstrated in Figure 7.

For each sample, 6 tests were performed for 15 consecutive days (3 weeks) by the same operator with the same instrument (**CONTEST-S**). The instrument was cleaned

[13] See Chapter 18 for the description of sample preparation.

at the beginning of each day, as ordinarily recommended in the periodical maintenance instructions (please, refer to Chapter 17 for more information).

The testing sequence was designed in such a way that the operator interchanged the samples after each test up to collect 6 repetitions per sample (i.e., A_1, B_1, C_1, …, C_6, D_6, E_6), in addition to alternate between sticky cottons and non sticky cottons with the target to exhibit that any potential contamination of results from test to test is excluded in the present analysis (please, refer to Chapter 22 for a detailed analysis).

The parameter used for the long-term evaluation of repeatability was the Stickiness Grade (St Grade). In this analysis, the mean values for each day are considered as the most representative estimate for the St Grade. Figure 7 shows the distribution of all the results (450 tests, 5 samples) to ensure that the whole range of sensitivity of the instrument was considered in this study.

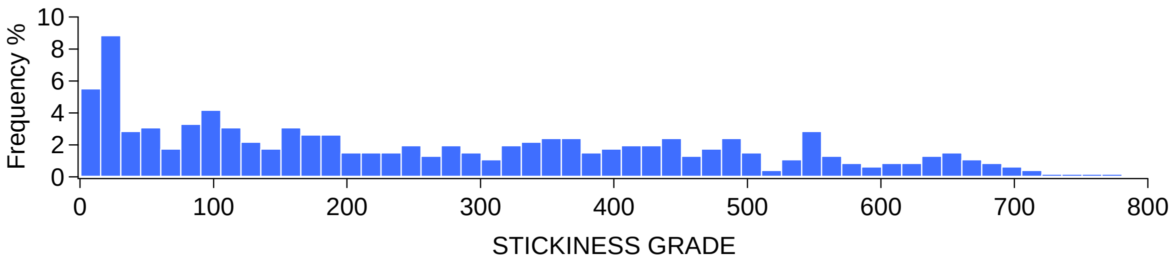


Figure 7: *Distribution of all the results (450 tests, 5 samples) in the repeatability analysis.*

For all the samples (A-E), Tables 2 – 6 summarize for each day of testing the following parameters: minimum (min), maximum (Max), 2nd quartile (Q2), 3rd quartile (Q3), median, mean, standard deviation (SD), coefficient of variation (CV%). For each week (5 days), a basic statistic for the mean values is presented. In Table 2 for cotton A, the conditioning values for the temperature, T(°C), and the relative humidity, RH (%), of the laboratory are presented.

In addition, Figure 8 - 12 display respectively all the cottons (A-E):

- on the left side: the result distribution for all the 15 days, as well as the statistical analysis by using all the results together;
- on the right side: the distribution of results along the 15 days (each point represents min-max, Q2-3, median, mean and SD of the day).

COTTON A (6 tests)										
Day	min	Max	Q2	Q3	Median	Mean	SD	CV%	T (°C)	RH(%)
1	334	544	366	455	400	431	71.4	16.6	21.1	66
2	319	486	320	485	346	407	86.2	21.2	21.3	65
3	323	623	343	490	380	445	116.9	26.3	21.3	66
4	372	513	379	425	400	420	50.3	12.0	21.3	66
5	354	600	386	476	472	466	81.4	17.5	21.4	66
Mean (of 5 days)						**433.6**			**21.3**	**65.8**
Median (of 5 days)						431.0			21.3	66.0
Standard Deviation						22.7			0.11	0.45
CV% between the days						5.2			0.51	0.68
6	373	520	378	468	390	434	61.4	14.2	21.1	66
7	319	559	335	448	369	416	88.2	21.2	21.3	66
8	339	495	363	449	418	423	55.1	13.0	21.1	66
9	270	427	289	409	332	359	64.9	18.1	21.2	66
10	286	467	330	415	377	389	60.8	15.6	21.5	67
Mean (of 5 days)						**403.9**			**21.2**	**66.2**
Median (of 5 days)						415.5			21.2	66.0
Standard Deviation						30.1			0.17	0.45
CV% between the days						7.4			0.79	0.68
11	298	529	320	471	366	413	91.7	22.2	21.1	67
12	266	510	302	467	351	400	98.6	24.7	21.6	65
13	276	501	314	416	375	389	75.6	19.4	20.9	66
14	374	560	387	432	417	436	65.1	14.9	20.6	68
15	273	435	302	373	349	356	54.2	15.3	21.2	65
Mean (of 5 days)						**398.5**			**21.1**	**66.2**
Median (of 5 days)						399.7			21.1	66.0
Standard Deviation						29.7			0.37	1.30
CV% between the days						7.4			1.76	1.97

Table 2: *Results of the repeatability analysis on Cotton A.*

COTTON B (6 tests)

Day	min	Max	Q2	Q3	Median	Mean	SD	CV%
1	10	90	11	36	18	34	31.3	93.4
2	9	90	13	38	25	36	29.5	82.3
3	6	59	7	39	15	28	21.8	79.3
4	16	48	19	38	33	32	11.6	36.0
5	15	28	16	23	21	21	4.7	22.2
Mean (of 5 days)						**30.1**		
Median (of 5 days)						32.3		
Standard Deviation						5.8		
CV% between the days						19.4		
6	11	46	18	30	25	28	11.4	41.1
7	7	36	10	15	14	17	10.0	60.8
8	13	53	20	40	37	35	13.8	39.7
9	15	46	16	28	19	26	11.5	45.3
10	18	35	19	30	21	26	7.6	29.8
Mean (of 5 days)						**26.0**		
Median (of 5 days)						25.5		
Standard Deviation						6.5		
CV% between the days						25.0		
11	10	52	13	38	28	30	15.7	52.1
12	6	26	11	22	16	18	7.1	40.0
13	5	46	6	23	17	20	15.1	75.3
14	8	34	8	22	16	18	9.9	54.2
15	6	19	7	16	12	13	5.4	42.9
Mean (of 5 days)						**19.8**		
Median (of 5 days)						18.2		
Standard Deviation						6.4		
CV% between the days						32.5		

COTTON C (6 tests)

Day	min	Max	Q2	Q3	Median	Mean	SD	CV%
1	135	332	152	246	208	223	70.8	31.8
2	214	405	229	332	288	303	69.5	23.0
3	136	342	159	263	238	237	72.7	30.7
4	269	370	273	339	327	320	39.2	12.3
5	160	239	163	228	193	202	34.4	17.0
Mean (of 5 days)						**256.9**		
Median (of 5 days)						237.0		
Standard Deviation						51.5		
CV% between the days						20.1		
6	172	306	187	220	203	220	45.5	20.7
7	138	272	146	239	171	202	55.1	27.3
8	194	447	198	337	204	287	103.5	36.1
9	151	368	162	278	253	250	79.1	31.6
10	184	293	203	258	255	245	37.3	15.2
Mean (of 5 days)						**240.8**		
Median (of 5 days)						244.8		
Standard Deviation						32.2		
CV% between the days						13.4		
11	188	354	193	306	228	263	68.1	25.9
12	159	300	170	212	186	208	50.6	24.3
13	109	282	133	256	184	207	68.0	32.9
14	128	375	157	302	187	246	94.1	38.2
15	157	246	161	202	170	190	36.6	19.2
Mean (of 5 days)						**223.0**		
Median (of 5 days)						208.2		
Standard Deviation						30.4		
CV% between the days						13.6		

Table 3 / 4: *Results of the repeatability analysis on Cotton B (left, Table 3) and C (right, Table 4).*

COTTON D (6 tests)

Day	min	Max	Q2	Q3	Median	Mean	SD	CV%
1	493	635	496	585	553	558	57.1	10.2
2	552	757	558	684	567	635	85.8	13.5
3	493	750	520	666	579	617	94.3	15.3
4	399	616	419	567	552	523	85.2	16.3
5	479	694	510	630	544	586	78.8	13.4
Mean (of 5 days)						**583.7**		
Median (of 5 days)						586.2		
Standard Deviation						44.7		
CV% between the days						7.7		
6	537	702	557	650	613	621	59.0	9.5
7	471	766	471	614	544	580	119.4	20.6
8	436	712	486	668	604	604	102.8	17.0
9	442	686	453	575	499	540	101.2	18.7
10	460	644	470	619	581	567	79.1	14.0
Mean (of 5 days)						**582.4**		
Median (of 5 days)						579.8		
Standard Deviation						31.6		
CV% between the days						5.4		
11	485	667	520	654	569	597	72.8	12.2
12	324	658	370	525	436	481	121.3	25.2
13	347	718	415	659	620	581	140.1	24.1
14	433	723	485	654	555	592	105.4	17.8
15	418	606	431	516	482	497	69.4	14.0
Mean (of 5 days)						**549.5**		
Median (of 5 days)						580.8		
Standard Deviation						56.2		
CV% between the days						10.2		

COTTON E (6 tests)

Day	min	Max	Q2	Q3	Median	Mean	SD	CV%
1	54	320	66	162	97	146	96.3	66.2
2	85	167	93	139	124	126	30.2	24.0
3	52	167	70	130	101	111	41.7	37.5
4	72	185	84	134	109	122	41.4	34.0
5	74	254	83	119	100	126	65.3	51.7
Mean (of 5 days)						**126.1**		
Median (of 5 days)						125.7		
Standard Deviation						12.4		
CV% between the days						9.9		
6	70	140	75	114	103	103	25.8	25.0
7	58	157	60	118	86	100	42.6	42.7
8	99	216	104	146	125	140	44.0	31.4
9	60	250	68	149	104	131	70.1	53.4
10	73	155	77	101	89	100	29.2	29.2
Mean (of 5 days)						**114.8**		
Median (of 5 days)						103.2		
Standard Deviation						19.3		
CV% between the days						16.8		
11	65	162	85	140	110	120	37.1	30.9
12	98	159	99	130	116	122	23.1	18.9
13	73	144	74	95	76	93	27.2	29.3
14	91	122	95	117	104	108	12.4	11.5
15	77	310	81	155	91	145	92.1	63.5
Mean (of 5 days)						**117.7**		
Median (of 5 days)						120.2		
Standard Deviation						19.3		
CV% between the days						16.4		

Table 5 / 6: *Results of the repeatability analysis on Cotton D (left, Table 5) and E (right, Table 6).*

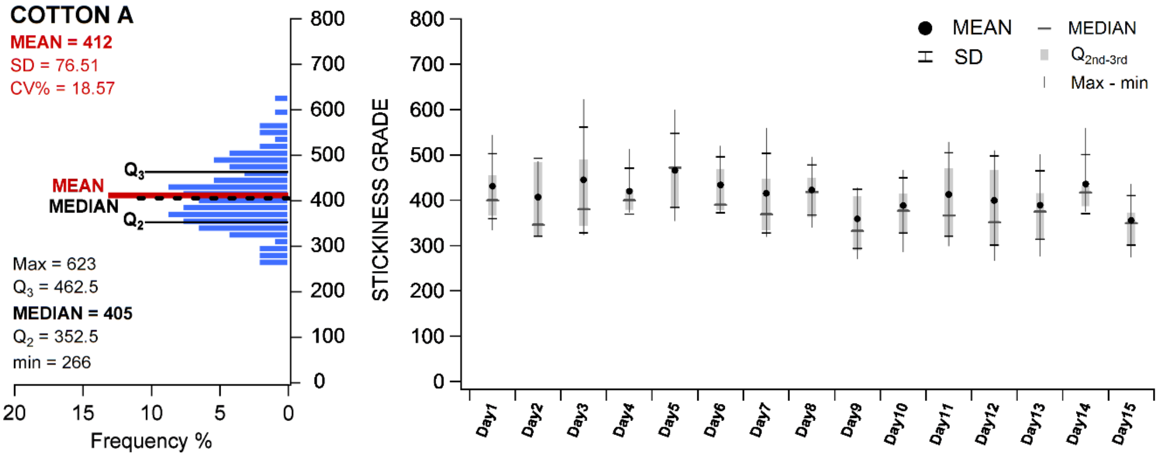


Figure 8: *Cotton A: distribution of results and statistic per day (right) and for all results (left).*

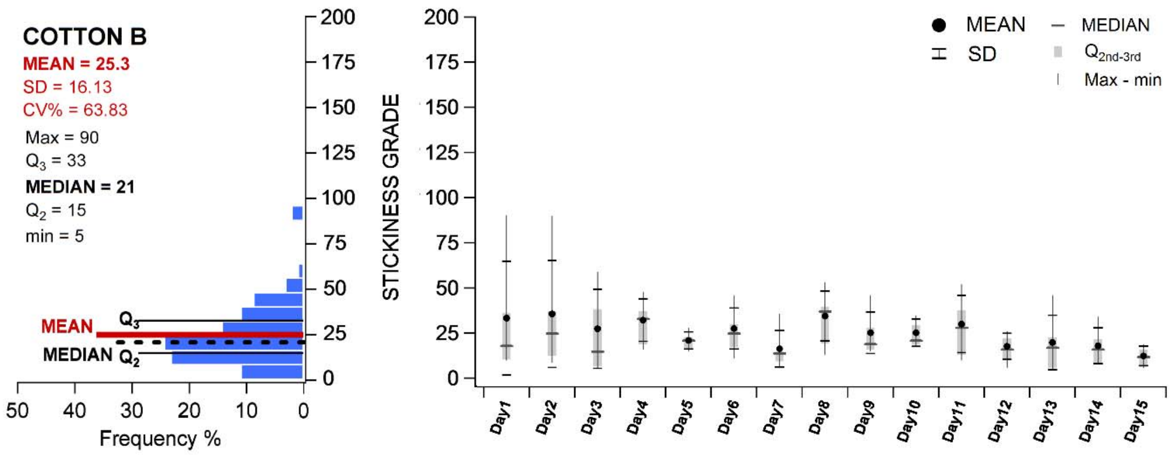


Figure 9: *Cotton B: distribution of results and statistic per day (right) and for all results (left).*

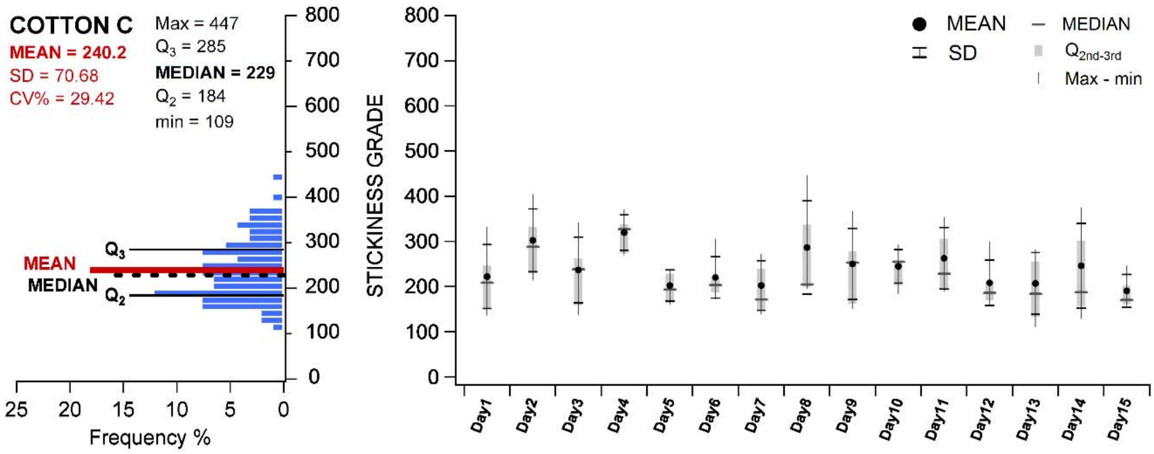


Figure 10: *Cotton C: distribution of results and statistic per day (right) and for all results (left).*

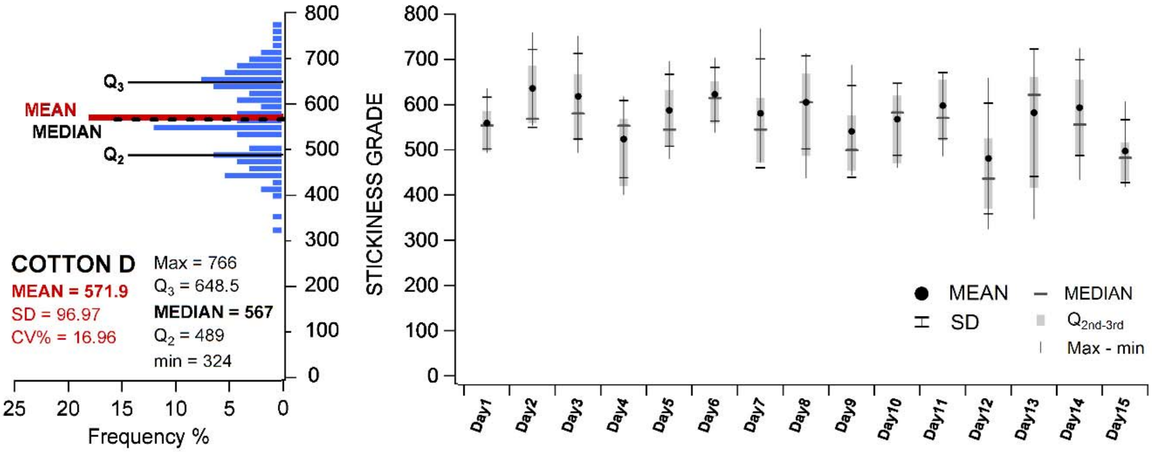


Figure 11: *Cotton D: distribution of results and statistic per day (right) and for all results (left).*

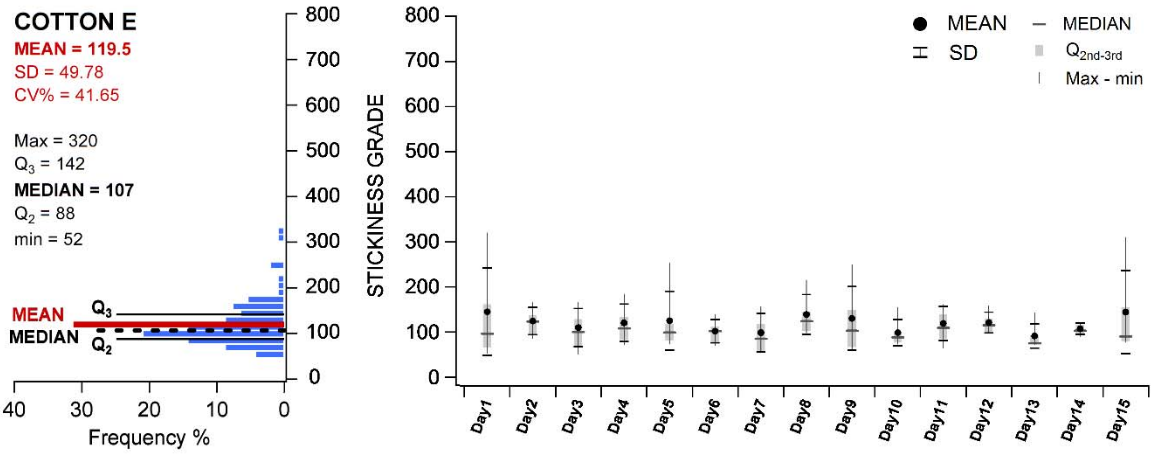


Figure 12: *Cotton E: distribution of results and statistic per day (right) and for all results (left).*

In order to statistically evaluate the variation between days, an Analysis of Variance (ANOVA) was conducted to compare the daily mean values between the weeks. Considering G groups (3 weeks) composed by n_G results (5 days/week)[14], it is possible to calculate the Variance-between ($Var_{between}$) the weeks by:

$$Var_{between} = \left(\frac{n_G}{G-1}\right)\sum_{G=1}^{3}(m_G - m)^2 \tag{2}$$

and the Variance-within (Var_{within}) the weeks by:

$$Var_{within} = \left(\frac{1}{N-G}\right)\sum_{G=1}^{3}\sum_{d=1}^{5}(x_{dG} - m_G)^2 \tag{3}$$

[14] For each day, 6 tests were performed per sample.

with

$$G = 3 \text{ (weeks)}$$

$$n_G = 5 \text{ (days per week)}$$

$$N = G \cdot n_G = 15 \text{ (total number of days)}$$

$$x_{dG} = \text{mean value of day } d \text{ of week } G$$

$$m_G = \frac{1}{n_G} \sum_{d=1}^{5} x_{dG} \tag{4}$$

$$m = \frac{1}{N} \sum_{G=1}^{3} \sum_{d=1}^{5} x_{dG} \tag{5}$$

For each cotton i, it is thus possible to establish that the variance between weeks is not significantly different than the variance within the weeks if it is valid

$$F_i = \frac{Var_i^{between}}{Var_i^{within}} < F(0.95; G-1; N-G) = 3.8853 \tag{6}$$

where 3.8853 is the value of the Fisher-distribution with $G-1=2$ and $N-G=12$ degrees of freedom, with a rejection region of 5%. As shown in Table 7, no significant difference between the weeks was obtained for the **Stickiness Tester**.

COTTON	F-calculated	F(0.95;2;12)	EVALUATION
A	2.3333	**3.8853**	No significant difference between weeks
B	3.4351		No significant difference between weeks
C	0.9354		No significant difference between weeks
D	0.9177		No significant difference between weeks
E	0.5683		No significant difference between weeks

Table 7: *ANOVA of the repeatability investigation on the mean values per day for each cotton sample.*

12.4 Reproducibility

For the investigation of reproducibility, 6 different **Stickiness Tester**, which are supplied in 3 different instrument types, were involved in a round test including 4 different laboratories:

- **CONTEST-S** (CS, at the manufacturer),
- **CONTEST-F** (CF, at the manufacturer),
- **CONTEST**[15] (C, at the manufacturer),
- **CONTEST-F** (CF, at customer, EU),
- **CONTEST**[15] (C, at customer, USA),
- **CONTEST**[15] (C, at customer, MIDDLE EAST).

Each laboratory received limited portions (of about 35 g) from the 5 cotton samples (A-E), which were already used in the repeatability analysis, with the instruction to perform 6 tests per cotton by using the same sample-interchanging sequence, which was already presented in chapter 12.3 (i.e., A_1, B_1, C_1, …, C_6, D_6, E_6). The samples were shipped to the laboratories with the samples labelled randomly.

The instructions to be followed by all the participants were similar than those suggested in the ITMF-ICCTM Stickiness Round Tests, in particular:

- pre-conditioning of the samples in standard atmosphere according to ASTM D1776 for at least 48 hours;
- cleaning of the instrument before starting the round test;
- testing in one day and by the same operator;
- recording of the Stickiness Grade results.

Tables 8-12 summarize the results of all the instruments.

COTTON A (6 tests per instrument)						
LABORATORY	MANUFACTURER			CUSTOMERS		
LOCATION	*ITALY*			*EU*	*ME*	*USA*
INSTRUMENT	CS	CF	C	CF	C	C
TEMP (°C)	21.4	21.3	21.6	21.2	20.7	19.4
RH (%)	65.3	64.9	66.2	68.5	64.0	64.0
test 1	219	362	334	440	309	283
test 2	325	367	315	317	486	266
test 3	341	361	240	567	565	245
test 4	300	296	462	512	493	244
test 5	364	172	381	500	541	315
test 6	329	391	431	329	359	111
Mean	**313**	**325**	**361**	**444**	**459**	**244**
Median	327	362	358	470	490	256
SD	50.6	81.3	81.2	102.2	102.3	70.3
CV%	16.2	25.0	22.5	23.0	22.3	28.8
Q95%	50.5	81.2	81.1	102.1	102.2	70.3

Grand Mean of all instruments (6)	**357.6**
Median of all results (36)	337.5
SD of all results (36)	108.3
SD between instruments (6)	82.1
CV% between instruments (6)	23.0

COTTON B (6 tests per instrument)						
LABORATORY	MANUFACTURER			CUSTOMERS		
LOCATION	*ITALY*			*EU*	*ME*	*USA*
INSTRUMENT	CS	CF	C	CF	C	C
TEMP (°C)	21.4	21.3	21.6	21.2	20.7	19.4
RH (%)	65.3	64.9	66.2	68.5	64.0	64.0
test 1	31	13	16	43	25	62
test 2	39	9	19	45	64	24
test 3	8	6	14	26	36	14
test 4	52	17	11	25	89	18
test 5	43	39	15	23	10	14
test 6	28	30	20	19	23	22
Mean	**34**	**19**	**16**	**30**	**41**	**26**
Median	35	15	16	26	31	20
SD	15.2	12.9	3.3	11.0	29.7	18.3
CV%	45.3	67.8	20.9	36.5	72.1	71.1
Q95%	15.1	12.9	3.3	11.0	29.6	18.2

Grand Mean of all instruments (6)	**27.6**
Median of all results (36)	23.0
SD of all results (36)	18.0
SD between instruments (6)	9.4
CV% between instruments (6)	34.1

Table 8 / 9: *Results of the reproducibility analysis on Cotton A (left, Table 8) and B (right, Table 9).*

[15] CONTEST is an instrument, integrating the Stickiness Tester with other measurements, such as Micronaire, Maturity ratio, Fineness (calculated), Neps count and size, Seed Coat count and size, Trash count and size.

COTTON C (6 tests per instrument)						
LABORATORY	MANUFACTURER			CUSTOMERS		
LOCATION	*ITALY*			*EU*	*ME*	*USA*
INSTRUMENT	CS	CF	C	CF	C	C
TEMP (°C)	21.4	21.3	21.6	21.2	20.7	19.4
RH (%)	65.3	64.9	66.2	68.5	64.0	64.0
test 1	225	224	126	213	313	167
test 2	183	205	185	254	97	153
test 3	120	210	166	232	241	192
test 4	204	147	178	137	272	140
test 5	163	155	163	208	219	124
test 6	175	193	208	238	179	116
Mean	**178**	**189**	**171**	**214**	**220**	**149**
Median	179	199	172	223	230	147
SD	36.1	31.2	27.3	41.1	75.6	28.2
CV%	20.2	16.5	16.0	19.3	34.4	19.0
Q95%	36.0	31.1	27.3	41.1	75.6	28.2

Grand Mean of all instruments (6)	**186.8**
Median of all results (36)	184.0
SD of all results (36)	47.2
SD between instruments (6)	26.9
CV% between instruments (6)	14.4

COTTON D (6 tests per instrument)						
LABORATORY	MANUFACTURER			CUSTOMERS		
LOCATION	*ITALY*			*EU*	*ME*	*USA*
INSTRUMENT	CS	CF	C	CF	C	C
TEMP (°C)	21.4	21.3	21.6	21.2	20.7	19.4
RH (%)	65.3	64.9	66.2	68.5	64.0	64.0
test 1	419	439	488	680	712	501
test 2	421	444	426	646	565	531
test 3	612	344	379	505	387	494
test 4	489	444	528	544	527	386
test 5	488	488	656	453	498	530
test 6	536	513	480	619	512	497
Mean	**494**	**445**	**493**	**575**	**534**	**490**
Median	489	444	484	582	520	499
SD	73.1	57.8	95.4	88.0	105.9	53.4
CV%	14.8	13.0	19.4	15.3	19.9	10.9
Q95%	73.0	57.8	95.3	87.9	105.8	53.4

Grand Mean of all instruments (6)	**505.0**
Median of all results (36)	497.5
SD of all results (36)	85.6
SD between instruments (6)	44.0
CV% between instruments (6)	8.7

Table 10 / 11: *Results of the reproducibility analysis on Cotton C (left, Table 10) and D (right, Table 11).*

COTTON E (6 tests per instrument)						
LABORATORY	MANUFACTURER			CUSTOMERS		
LOCATION	*ITALY*			*EU*	*ME*	*USA*
INSTRUMENT	CS	CF	C	CF	C	C
TEMP (°C)	21.4	21.3	21.6	21.2	20.7	19.4
RH (%)	65.3	64.9	66.2	68.5	64.0	64.0
test 1	99	188	182	189	122	129
test 2	129	221	127	159	226	157
test 3	41	71	53	91	193	102
test 4	222	242	73	223	85	114
test 5	103	135	231	113	91	93
test 6	179	88	125	115	89	93
Mean	**129**	**158**	**132**	**148**	**134**	**115**
Median	116	162	126	137	107	108
SD	64.0	70.6	66.5	51.0	60.6	24.9
CV%	49.7	44.8	50.4	34.4	45.1	21.7
Q95%	63.9	70.5	66.4	50.9	60.5	24.9

Grand Mean of all instruments (6)	**135.9**
Median of all results (36)	123.5
SD of all results (36)	55.8
SD between instruments (6)	15.1
CV% between instruments (6)	11.1

Table 12: *Results of the reproducibility analysis on Cotton E.*

The ANOVA approach was used again to statistically evaluate the reproducibility of the **Stickiness Tester** by comparing the variation between/within instruments, as already described in the previous section 12.3. For each cotton i, it can be established that the variance between instruments is not significantly different than the variance within the instruments if it is valid

$$F_i = \frac{Var_i^{between}}{Var_i^{within}} < F(0.95; G-1; N-G) = 2.5336 \tag{7}$$

where 2.5336 is the value of the Fisher-distribution with $G-1=5$ and $N-G=30$ degrees of freedom with a rejection region of 5% (for $G=6$ instruments ; $n_G=6$ tests per cotton; $N=G\cdot n_G=36$ results to be compared per cotton). As shown in Table 13, it is obtained a significant difference between instruments only for the cotton A. For all the other cotton samples (B-E), no significant difference is observed.

COTTON	F-calculated	F(0.95;5;30)	EVALUATION
A	5.8316		Significant difference between instruments
B	1.8262		No significant difference between instruments
C	2.3177	**2.5336**	No significant difference between instruments
D	1.0486		No significant difference between instruments
E	0.4029		No significant difference between instruments

Table 13: *ANOVA of the reproducibility study among different instruments/laboratories for each cotton.*

At this stage, a T-test was carried out to focus on the case of cotton A. For each cotton, Table 14 compares the difference between the Mean values for all the instruments and their Grand Mean (Δ = $Mean_{inst}$ – Grand Mean) with the resolution discrimination $Q(95\%, 6)$, which is calculated by:

$$Q(95\%, n) = t(95\%, n) \cdot \frac{SD_{avg}}{\sqrt{n}} \tag{8}$$

where SD_{avg} is the mean of the standard deviations for all the instruments, $n=6$ tests per sample, and $t(95\%, n) = 2.447$ is the t-value of the Student distribution, which is calculated with a confidence interval of 95% for n tests. Therefore, whenever it occurs that

$$-Q_i(95\%, 6) < \Delta_i < Q_i(95\%, 6) \tag{9}$$

no significant difference can be observed between the instrument i and the statistic ensemble of all the results for the selected cotton. The T-test shows 3 instruments out of the confidence range for cotton A and 2 of these ones correspond to laboratories where the conditioning parameters were slightly out of the standard (ASTM D1776) during the round test, thus drawing attention to the importance of a proper conditioning of samples for the detection of stickiness.

T-test

LABORATORY		MANUFACTURER			CUSTOMERS		
LOCATION		*ITALY*			*EU*	*ME*	*USA*
INSTRUMENT		CS	CF	C	CF	C	C
TEMP (°C)		21.4	21.3	21.6	21.2	20.7	**19.4**
RH (%)		65.3	64.9	66.2	**68.5**	64.0	64.0
COTTON A (6 tests)	Mean	313	325	361	444	459	244
	SD	50.6	81.3	81.2	102.2	102.3	70.3
	Δ	-45	-33	3	87	101	-114
COTTON B (6 tests)	Mean	34	19	16	30	41	26
	SD	15	13	3	11	30	18
	Δ	6	-9	-12	3	14	-2
COTTON C (6 tests)	Mean	178	189	171	214	220	149
	SD	36	31	27	41	76	28
	Δ	-8	2	-16	27	33	-38
COTTON D (6 tests)	Mean	494	445	493	575	534	490
	SD	73	58	95	88	106	53
	Δ	-11	-60	-12	69	28	-15
COTTON E (6 tests)	Mean	129	158	132	148	134	115
	SD	64	71	66	51	61	25
	Δ	-7	22	-4	12	-2	-21

T(95%, 6) = 2.447

Grand Mean (6 instruments)	Q(95%, 6)	EVALUATION
357.6	81.2	significant difference
27.6	15.0	no significant difference
186.8	39.9	no significant difference
505.0	78.9	no significant difference
135.9	56.2	no significant difference

Table 14: *(left) Mean value and standard deviation (SD) for each instrument/laboratory, as well as the discrepancy Δ between the mean and the Grand Mean for all the instruments. In addition, the conditioning parameters, T°C and RH%, are reported. (right) Grand Mean values for all the cottons, as well as the resolution discrimination Q(95%) for 6 tests; for each cotton, the evaluation of T-test is listed.*

In Figure 13, the temperature (TEMP) and the relative humidity (RH) are shown for each instrument/laboratory in comparison with the standard ASTM D1776. In Figure 14, the variability of the Mean ± SD values for each instrument are displayed with the Grand Mean ± Q(95%,6) for each cotton sample. In this way, concerning the three instruments out of the t-confidence range for cotton A (i.e. CF_{EU}, C_{ME}, C_{USA}), the following conclusions can be presented:

1. the case of CF_{EU} may be justified by the overcome of the RH standard condition of about +1.5%, which should induce a general overestimation of stickiness, as it can be observed for all the cotton samples ($\Delta > 0$), especially for cotton A, C, and D;

2. the case of C_{USA} is opposite than CF_{EU}, where a lowering of temperature of about -0.6°C with respect to the standard condition may induce a general underestimation of the stickiness measurement, as it can be noted for all the cotton samples ($\Delta < 0$), in particular for cotton A and C;

3. the case of C_{ME} cannot be ascribed to conditioning; however, the significant difference may be statistically re-evaluated on the basis that Δ is comparable to both SD and Q(95%,6) for cotton A: $\Delta \leq$ SD and $\Delta \approx$ Q(95%,6).

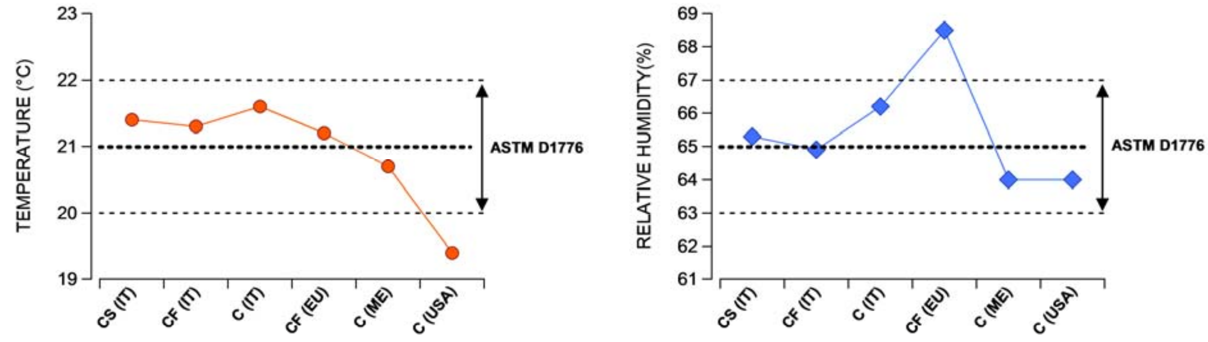


Figure 13: *Conditioning parameters during the reproducibility study for all the participant instruments/laboratories: (left) temperature (°C), (right) relative humidity (%).*

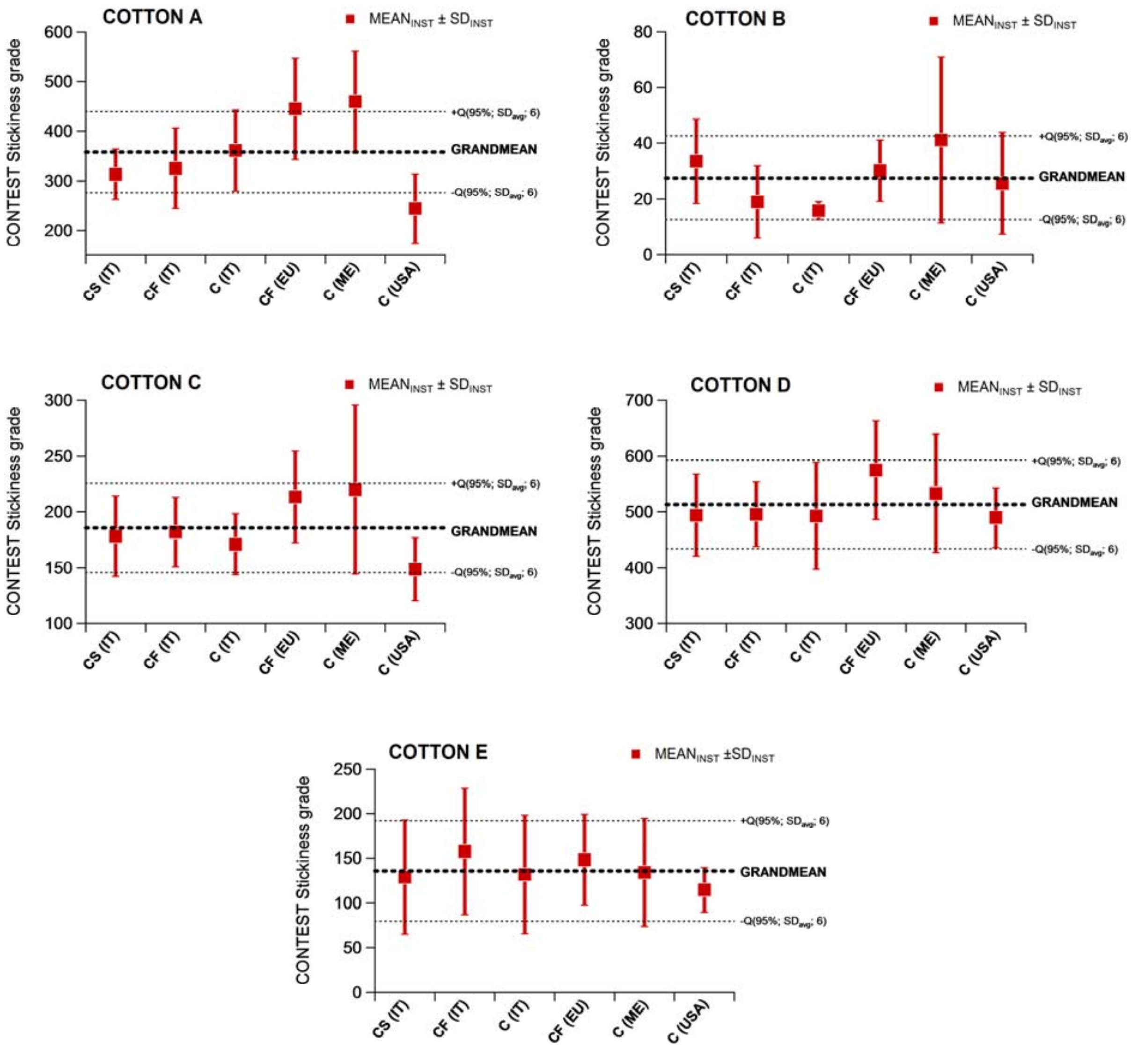


Figure 14: *For each cotton (A-E): mean values ± standard deviation for all the instruments/laboratories in comparison with their Grand Mean ± resolution discrimination Q(95%) for 6 tests.*

12.5 Summary: Resolution Discrimination for different samples

Table 14 summarizes the resolution discrimination Q(95%,n), which is calculated by using the formula (8), for all the cotton samples (A-E) involved in the reproducibility investigation.

13. Comparison to Reference Method

Refer to Chapter 14, where the analysis of international round trials is described, involving different methods for the stickiness detection.

14. Comparison to other test methods in Round Trials

From 2014 the international round tests for stickiness of the International Cotton Committee on Testing Method of the International Textile Manufacturers Federation (ICCTM-ITMF) have involved several techniques with the aim of harmonizing the results of different stickiness testers, as well as assuring the reliability/comparability of different methods, such as:

- **chemical** (Chemicart Klebrigkeit / ChemCare, Kotiti, Total sugar content, HPLC, etc…),
- **physical** (Caramelization, FT-IR, HSI-NIR, etc…),
- **mechanical** (Minicard),
- **thermo-physical** (SCT, H2SD, CONTEST, TDM-A).

As already discussed in Chapter 10, the only techniques which may be considered as reference methods for **CONTEST – Stickiness Tester** are those sharing a similar principle of detection, such as the MINICARD (concerning the specimen preparation), as well as the SCT and the H2SD (about the thermodetector principle). Therefore, in the following analysis only the results of the aforementioned methods were considered for all the round test sessions, which were attended by the **Stickiness Tester**[16] from 2017-1 up to 2019-2.

Figure 15 displays the distributions of all the results of all the instruments/laboratories per method. Since each method shows a different unit, any direct comparison between methods is clearly unfeasible at this level of analysis.

[16] In the period from SRT2017-1 to 2019-2, the Stickiness Tester were equipped in different instruments: Mesdan CONTEST, CONTEST-S, CONTEST-F, as well as Loepfe FIBERMAP.

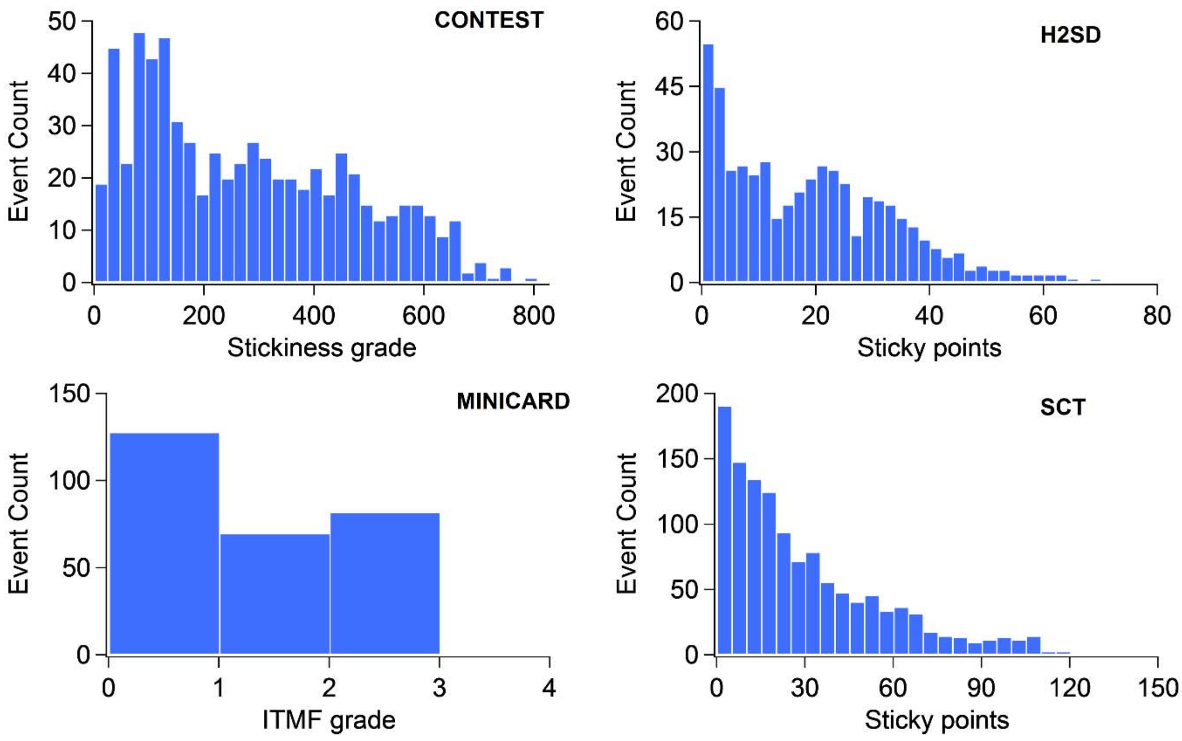


Figure 15: *Distribution of results for all the instruments/laboratories, which have been participating to the international Round Tests for Stickiness of ICCTM-ITMF from 2017-1 up to 2019-2, divided per method: CONTEST (top left), H2SD (top right), MINICARD (bottom left), SCT (bottom right).*

14.1 Conversion rules and comparison to reference methods

In spite of different units, the comparison between different methods may be achieved[17] by evaluating:

1. a common scale for all the methods;
2. or a conversion rule for each couple of methods.

The second option is developed in this section by using simple linear regression analysis between specific couples of methods; in this way, it is possible to minimize the conversion error between different units, as long as a robust condition of linearity is confirmed between the sets of data. This approach requires *reference* instruments per method to be compared on the basis of a common set of *reference* samples.

Because high variations in the results between laboratories with the same method have been so far observed[18], it is not possible to identify a single detector/laboratory per method (MINICARD, SCT, H2SD, CONTEST), which is representative for all the other instruments/laboratories (i.e., the *reference* instrument required above). Therefore, in this situation the reference instrument is substituted with the mean value of the distribution of all the results among all the laboratories/instruments sharing the same

[17] Provided that all the methods actually detect the same stickiness. See Chapter 10.
[18] General conclusions for the ITMF-ICCTM SRT2019-2.

method (MINICARD, SCT, H2SD, CONTEST); the mean values thus obtained are guessed to be the most representative evaluation of stickiness for each cotton and for each method selected.

Figure 16, 17, and 18 display the linear regression analysis for some combinations of methods[19] by considering the mean values per method (as described above) for all the cottons from SRT2017-1 to 2019-2 (to be considered as *reference* materials).

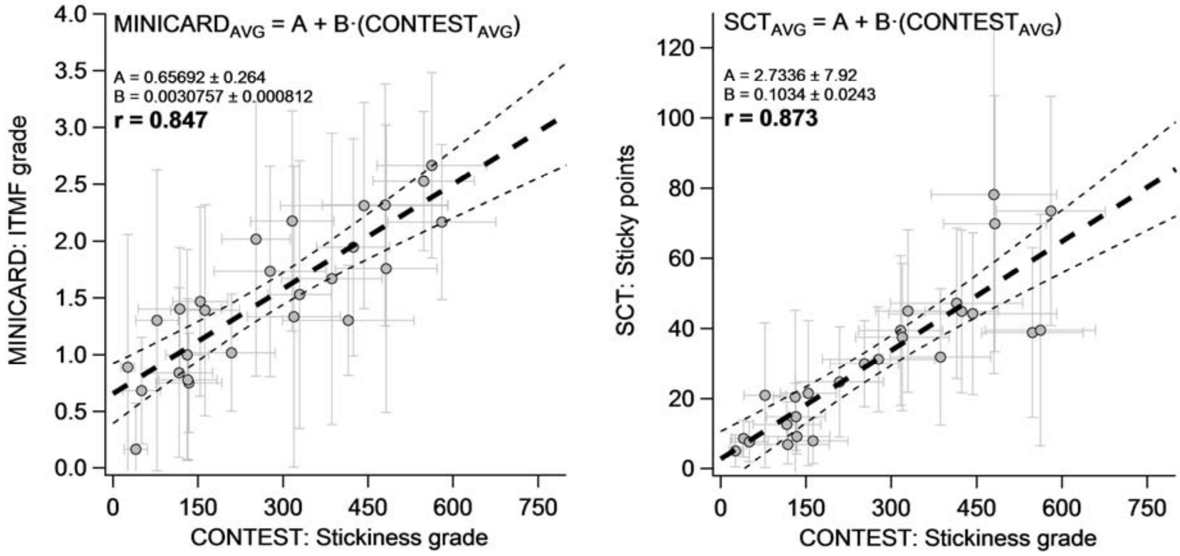


Figure 16: *Linear regression analysis for mean values between methods among all the participant laboratories from SRT2017-1 to 2019-2: (left) MINICARD vs CONTEST, (right) SCT vs CONTEST.*

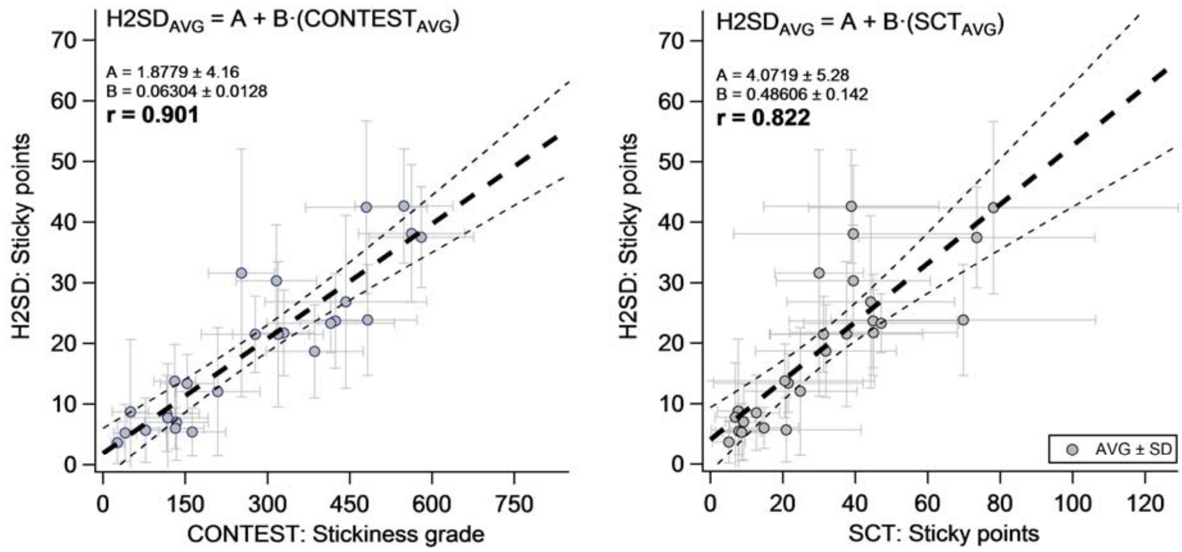


Figure 17: *Linear regression analysis for mean values between methods among all the participant laboratories from SRT2017-1 to 2019-2: (left) H2SD vs CONTEST, (right) H2SD vs SCT.*

[19] The combinations shown are the ones (1) useful to calculate the CONTEST conversion rules with the other methods and (2) sufficient to show all the correlation coefficients r for the four methods.

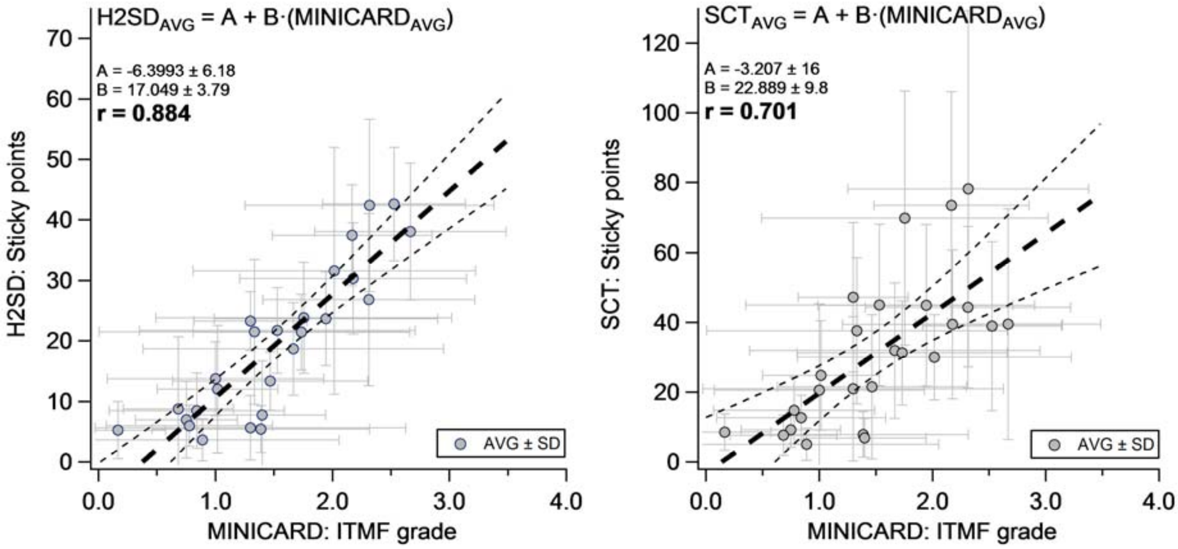


Figure 18: *Linear regression analysis for mean values between methods among all the participant laboratories from SRT2017-1 to 2019-2: (left) H2SD vs MINICARD, (right) SCT vs MINICARD.*

Table 15 summarizes the correlations results (i.e., the Bravais-Pearson Coefficient r), whereas Table 16 and 17 report respectively the linear regression coefficients A and B, which define the conversion rule between methods by the formula:

$$Y = A + B \cdot X \tag{10}$$

Concerning the correlation coefficient r, **CONTEST** shows a comparable result (0.847) versus MINICARD in comparison with SCT (0.701) and H2SD (0.884), whereas **CONTEST** exhibits the best correlation r versus SCT (0.873) and H2SD (0.901) with respect to all the other methods.

r		x-axis			
		MINICARD	SCT	H2SD	CONTEST
y-axis	MINICARD	1	0.701	0.884	0.847
	SCT	0.701	1	0.822	0.873
	H2SD	0.884	0.822	1	0.901
	CONTEST	0.847	0.873	0.901	1

Table 15: *Bravais-Pearson correlation coefficients r between different methods.*

A		x-axis			
		MINICARD	SCT	H2SD	CONTEST
y-axis	MINICARD	0	0.83316	0.6214	0.65692
	SCT	-3.207	0	4.4238	2.7336
	H2SD	-6.3993	4.0719	0	1.8779
	CONTEST	-75.555	45.436	27.793	0

B		x-axis			
		MINICARD	SCT	H2SD	CONTEST
y-axis	MINICARD	1	0.0215	0.045876	0.0030757
	SCT	22.889	1	1.3924	0.1034
	H2SD	17.049	0.48606	1	0.06304
	CONTEST	233.39	7.3699	12.872	1

Table 16 / 17: *Linear regression coefficients A (left, Table 16) and B (right, Table 17) of the conversion formula (10) for all the combinations of methods.*

In Figure 19, the distributions of all the results of all the instruments/laboratories per reference method (H2SD, SCT, MINICARD; colored bars) are compared with the **CONTEST** distribution of converted results (black line) by using the equation (10). Since the offset A is generally small in comparison with the maximum detected value per method, it is possible to approximate A ≈ 0 in such a way to avoid any bias in the distribution of converted data. In this way, Figure 19 shows similarities and discrepancies for each couple of methods along the scale of detection.

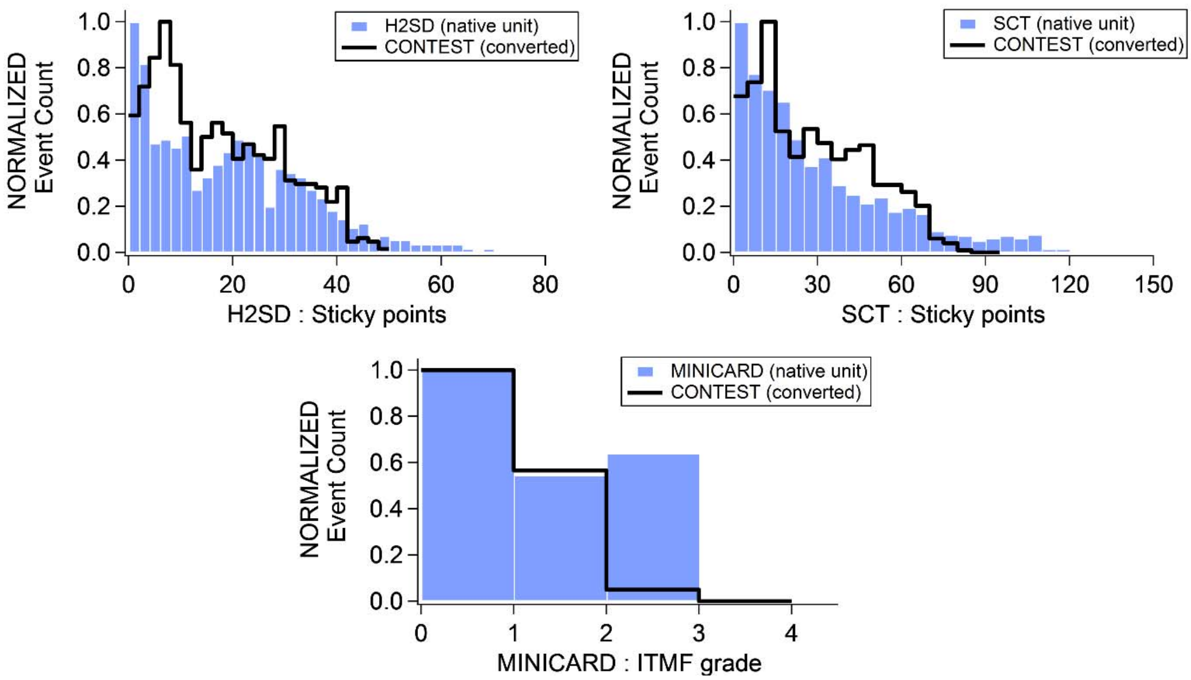


Figure 19: *Distribution of results in native unit for the following methods: H2SD (top left), SCT (top right), MINICARD (bottom). Black lines stand for the CONTEST distribution of results after the conversion into the method unit by the equation (10) with A=0 and B listed in Table 17.*

In order to provide a term of comparison, Figure 20 shows the distribution of converted results for H2SD and SCT respectively versus SCT and H2SD.

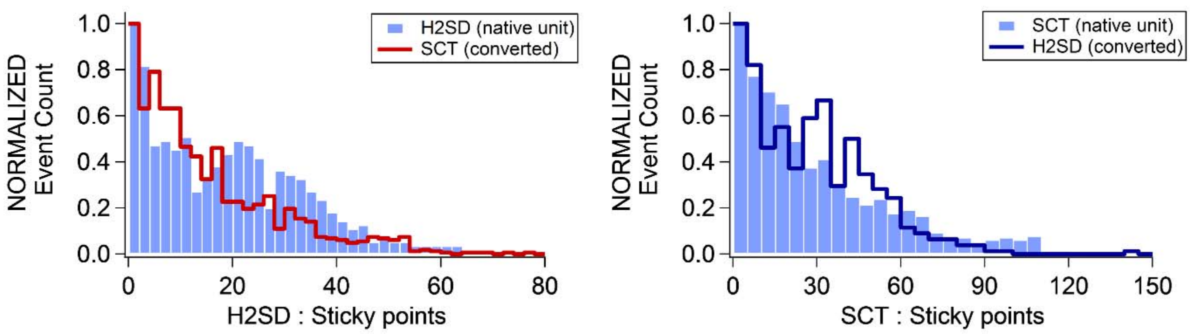


Figure 20: *Distribution of results for: (left) H2SD (bars, native unit) with converted results of SCT (red line) and (right) SCT (bars, native unit) with converted results of H2SD (blue line). Equation (10) was used for the conversion with A=0 and B listed in Table 17.*

Figure 21, 22, 23 display for each cotton the distribution of results among all the participant instruments between **CONTEST – Stickiness Tester** (converted unit) and the other methods (native unit). In the charts below, the following parameters are shown for each cotton: Mean, SD, min, Q_{2nd}, Median, Q_{3rd}, Max.

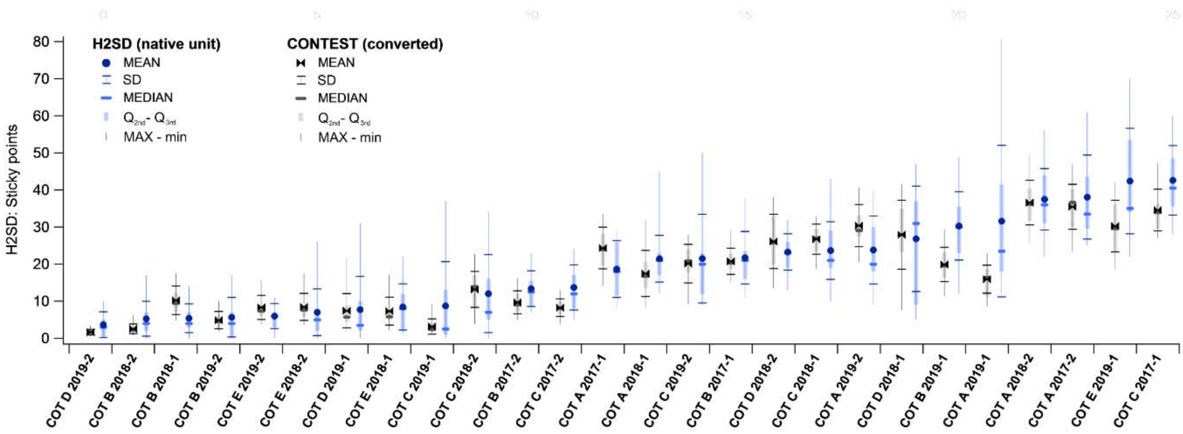


Figure 21: *Distribution of results per cotton among all the participant instrument/laboratories: H2SD (native unit) and CONTEST (converted into the H2SD unit).*

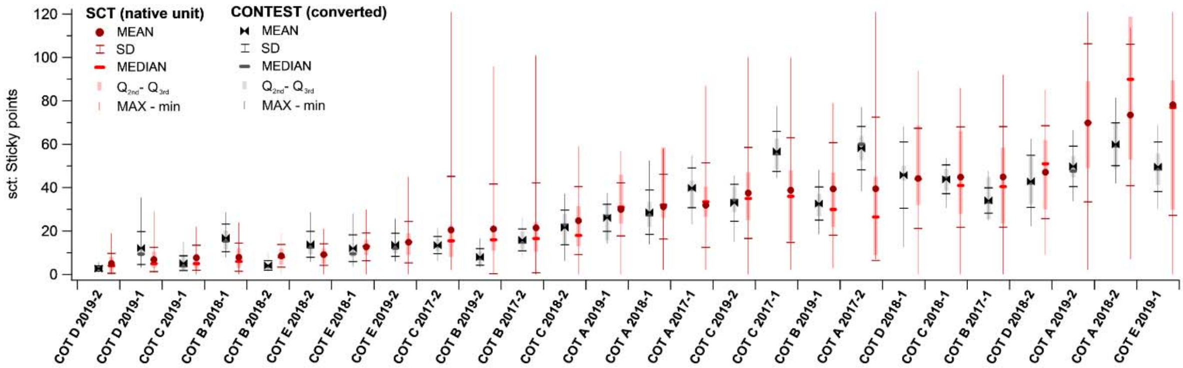


Figure 22: *Distribution of results per cotton among all the participant instrument/laboratories: SCT (native unit) and CONTEST (converted into the SCT unit).*

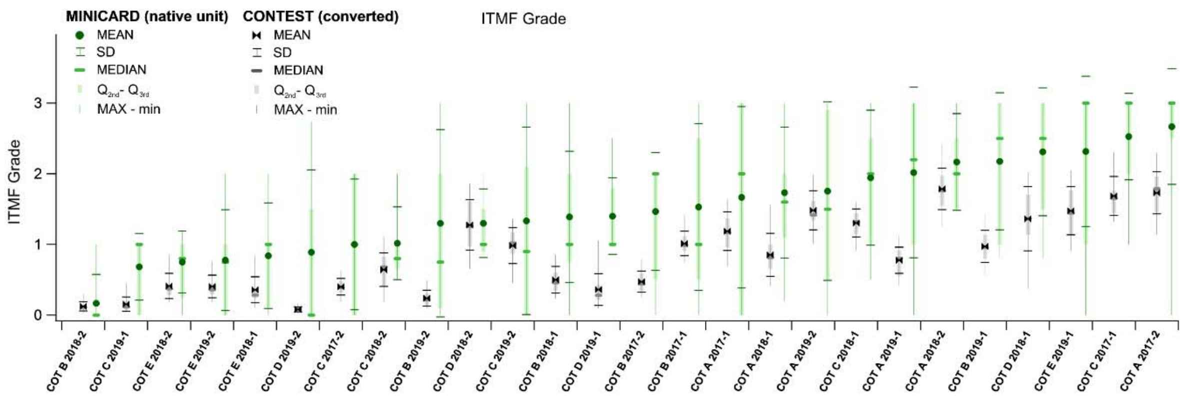


Figure 23: *Distribution of results per cotton among all the participant instrument/laboratories: MINICARD (native unit) and CONTEST (converted into the MINICARD unit).*

Figure 24, 25, 26 provide a detailed view of the discrepancy between the mean values of **CONTEST – Stickiness Tester** (converted unit) and the other methods (native unit). From these charts the mean absolute difference between methods, Δ, is calculated.

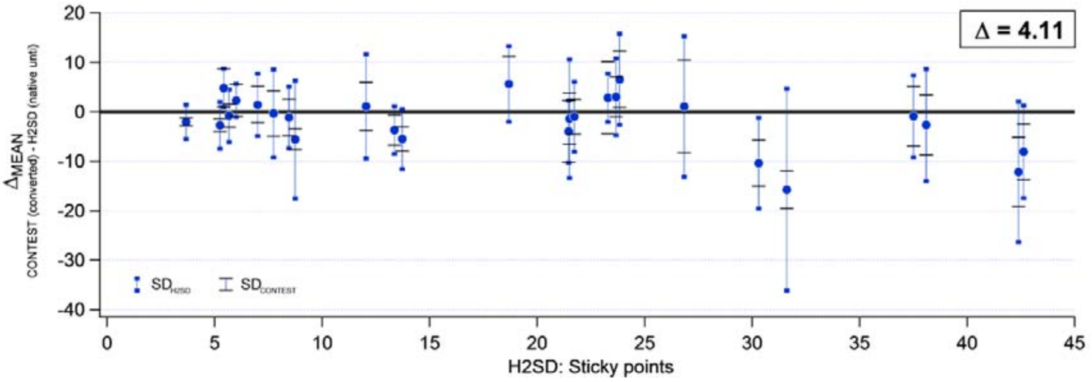


Figure 24: *For each cotton, the discrepancy between mean values is shown between H2SD (native unit) and CONTEST (converted); the standard deviations between instruments for H2SD and CONTEST are displayed. The mean absolute distance Δ between H2SD and CONTEST is calculated.*

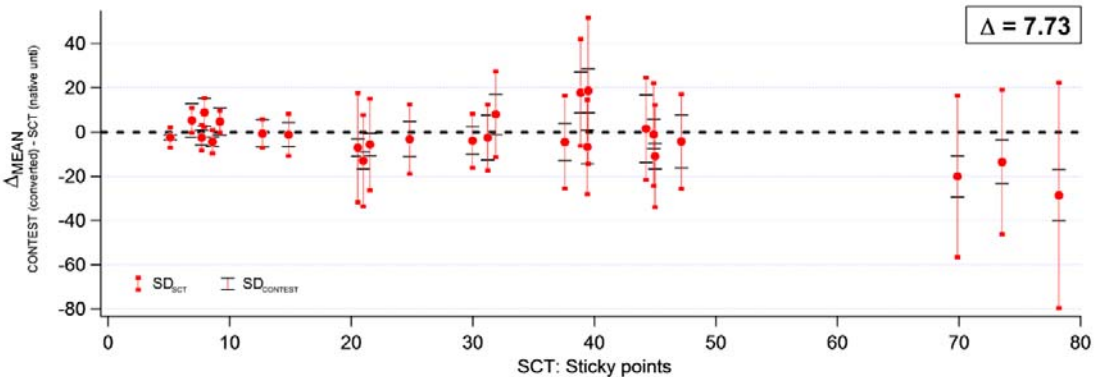


Figure 25: *For each cotton, the discrepancy between mean values is shown between SCT (native unit) and CONTEST (converted); the standard deviations between instruments for SCT and CONTEST are displayed. The mean absolute distance Δ between SCT and CONTEST is calculated.*

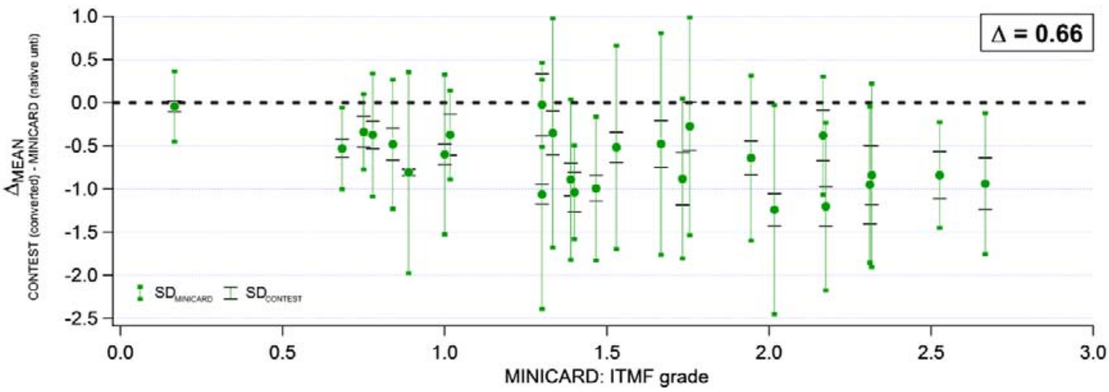


Figure 26: *For each cotton, the discrepancy between mean values is shown between MINICARD (native unit) and CONTEST (converted); the standard deviations between instruments for MINICARD and CONTEST are displayed. The mean absolute distance Δ between the methods is calculated.*

Table 18 summarizes the main results of Chapter 14.1 about the comparison between the reference methods (H2SD, SCT, MINICARD) and the **CONTEST – Stickiness Tester** in the ITMF-ICCTM Stickiness Round Trials from 2017-1 up to 2019-2, where:

- r is the Bravais-Pearson coefficient of correlation between **CONTEST** and the other reference methods (H2SD, SCT, MINICARD);

- A and B are the coefficients of the linear regression analysis. About the unit conversion from the **CONTEST** unit (Stickiness Grade) into the native unit (Sticky points or ITMF grade) of the reference methods (H2SD, SCT, MINICARD), equation (10) were used with the approximation A ≈ 0.

- Δ is the mean absolute distance of mean values between **CONTEST** (in converted unit) and the reference methods (in native units), as follows:

$$\Delta = \frac{1}{N}\sum_{cot=1}^{N} \|\Delta_{cot}\| \qquad (11)$$

with N number of cottons (26) from SRT2017-1 up to SRT2019-2.

METHOD	r	A	B	Δ	UNIT
H2SD	**0.901**	1.8779	0.06304	**4.11**	Sticky points
SCT	**0.873**	2.7336	0.1034	**7.73**	Sticky points
MINICARD	**0.847**	0.65692	0.0030757	**0.66**	ITMF grade

***Table 18**: The main results about the comparison between the Stickiness Tester and the other methods are listed: Bravais-Pearson coefficient of correlation r, linear regression coefficients A and B for the conversion rules between methods, mean absolute distance Δ between methods.*

14.2 Inter-Laboratory analysis in Round Trials

A different kind of comparison among the detectors may be presented with no reference to the unit of each method. In fact, by considering for each cotton the variability inter-laboratory per method[20], it is possible to calculate the coefficient of variation inter-laboratory, as shown in Figure 27 (left side). In addition, the number of instruments/laboratories involved for each Stickiness Round Test session is displayed in Figure 27 (right side). Figure 28 shows the inter-laboratory CV as a function of the Grand Mean per cotton samples: for all the methods it is generally noted an increase of variability along with a lowering of stickiness.

[20] The inter-laboratory analysis between intruments by method has been available in the ITMF-ICCTM Round Test Reports since SRT2018-1.

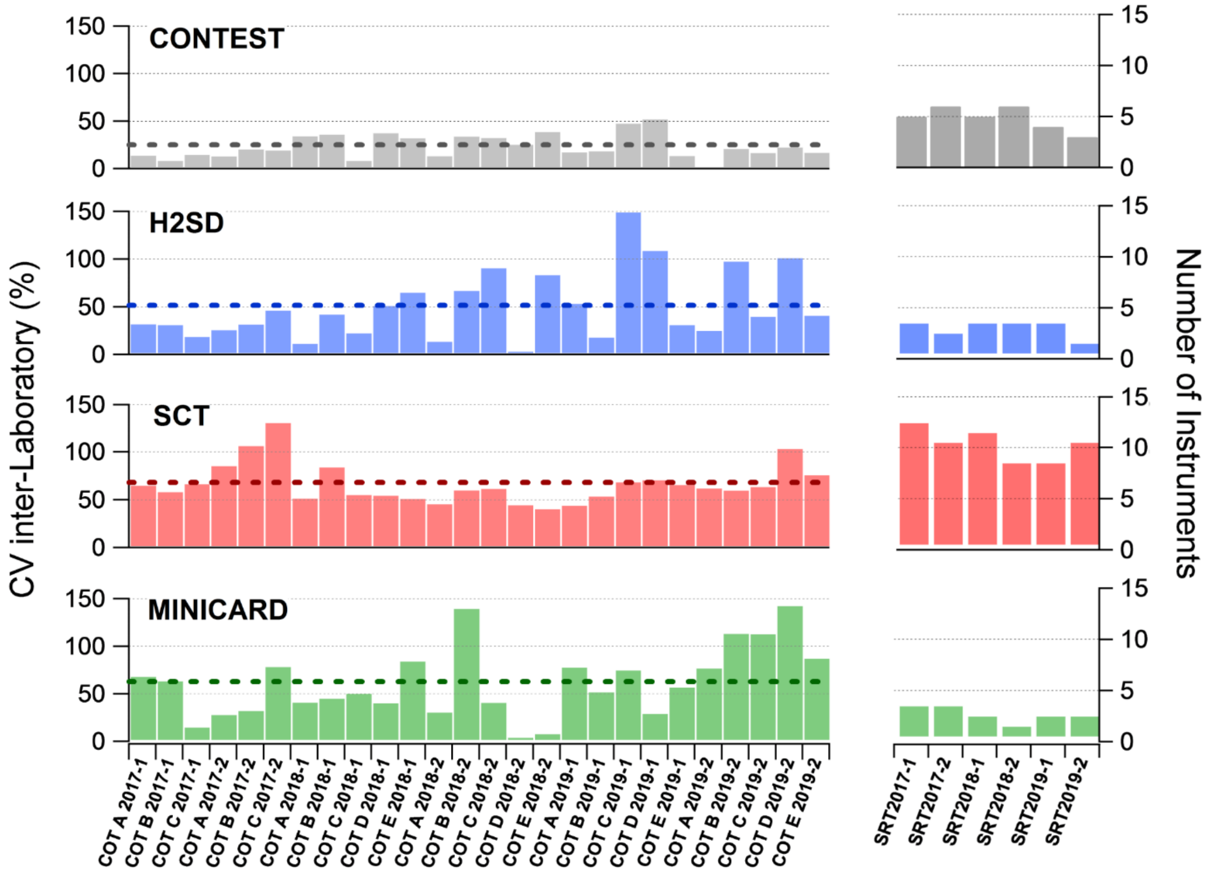


Figure 27: *(left) For each cotton sample, the inter-laboratory coefficient of variation ($CV_{interLab}$) per method; the dotted-lines stand for the average value for all the inter-laboratory CV. (right) The number of instruments/laboratories involved for each round tests session.*

Table 19 lists the average of the coefficients of variation between instruments per method among all the cottons from SRT2017-1 to SRT2019-2, calculated as follows:

$$\overline{CV}_{interLab} = \frac{1}{N}\sum_{cot=1}^{N} CVinterLab_{cot} \qquad (12)$$

as well as the number of instruments/laboratories involved for each round test session.

METHOD	$\overline{CV}_{interLab}$	STICKINESS ROUND TEST SESSION						
		2017-1	*2017-2*	*2018-1*	*2018-2*	*2019-1*	*2019-2*	
CONTEST	25.0%	5	6	5	6	4	3	Number of Instruments
H2SD	51.6%	4	3	4	4	4	2	
SCT	68.0%	13	11	12	9	9	11	
MINICARD	62.8%	4	4	3	2	3	3	

Table 19: *(left) Average of the inter-laboratory CV per method; (right) number of instruments/laboratories per method involved for each round test session.*

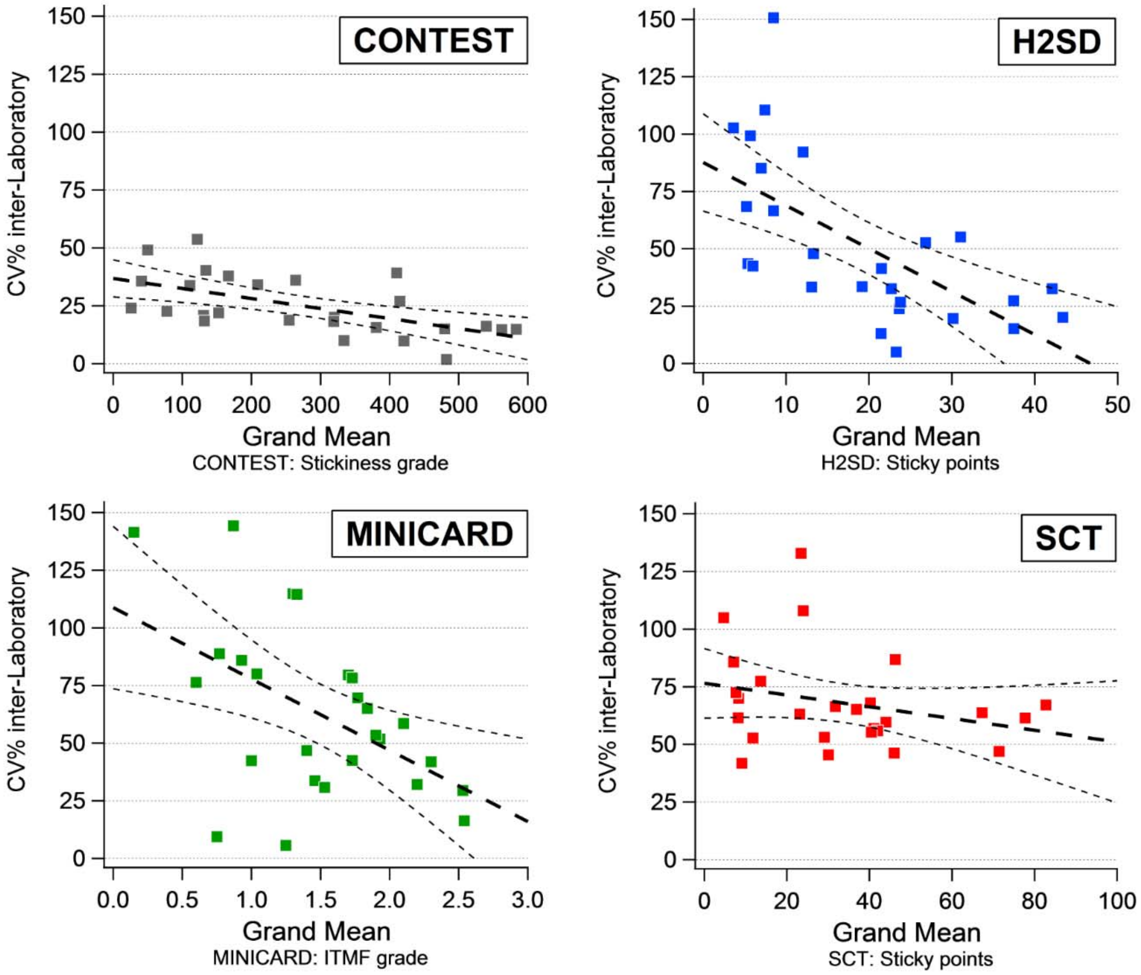


Figure 28: *For each cotton sample, the inter-laboratory CV as a function of the Grand Mean among all the instruments: (top left) CONTEST, (top right) H2SD, (bottom left) MINICARD, (bottom right) SCT.*

15. Manufacturer-independent check

Refer to Chapter 12.4, where the reproducibility study was carried out involving three laboratories, which are independent of the manufacturer.

16. External influences and measurement uncertainty

The external factors, which are mainly involved in the measurement uncertainty of the **Stickiness Tester**, are listed below:

1. Material variability: it is an unpredictable factor mainly related to the kind of material, the sampling and the level of stickiness[21];

2. Sample conditioning: the stability of the laboratory conditioning within the standards (ASTM D1776) is essential for reliability during testing, as well as for the pre-conditioning of the sample, which requires at least 24 hours of exposure. If the laboratory conditions are out of the above tolerances, the evaluation of stickiness (St Grade, St Cnt) may change significantly;

3. Operator influence: it is very limited due to the carding of the specimen into a fiber web by the micro card unit provided with the **Stickiness Tester**; this process in fully automatic for each sample. Considering the sample preparation by the operator, instead, out of the tolerance for the sample weight (3.5 ± 0.2 g) the sticky point count/g is significantly affected, whereas out of the sample length tolerance (30 ± 3 cm) the efficiency of the web formation may be reduced.

The previous factors affect the entire uncertainty, which can be calculated according to the formula:

$$SD = \sqrt{SD_1^2 + SD_2^2 + SD_3^2 + SD_4^2} \tag{13}$$

where SD_1, SD_2 and SD_3 stand respectively for the factors of variability described above, whereas SD_4 is related to the instrument inaccuracy (not mentioned before, since it is not an external factor).

16.1 Material variability

On the basis of the SD results of Chapter 12.2 for the repeatability study on different samples A - E with only one instrument, by one operator and within the same laboratory, it may be reasonable in first approximation to consider $SD_2 = SD_3 \approx 0$, because the laboratory involved was in compliance with the standard ASTM D1776 during testing and the instrument was used by a single operator, who followed the proper instructions of sample preparation (weight = 3.5 ± 0.2 g, length = 30 ± 3 cm).

Hence, it follows that:

$$SD \approx \sqrt{SD_1^2 + SD_4^2} \tag{14}$$

leading to an evaluation of the material variability, SD_1, as:

$$SD_1 \approx \sqrt{SD^2 - SD_4^2} \tag{15}$$

[21] In the case of a ideal homogenization of the sample, which makes the material to bring the same stickiness content in every possible subsamples, the expected distribution of sticky points is Poisson/Binomial-like. This leads to increasing variance with the number of sticky points.

Supposing that the instrument inaccuracy can be approximated as a certain constant factor by virtue of the real time monitoring of the components within certain limits of acceptability in the device (i.e., the stability of laser signals, drum temperatures, drum speed), it should be possible to argue that the material variance increases along with the sample stickiness, similarly as shown in Figure 6 (left). As a confirmation that such a behaviour is related to the material itself and it is not typical of only the **Stickiness Tester**, Figure 29 (below) show the charts of the entire uncertainty, SD, for all the methods taken into account in this document for the Stickiness Round Test from 2017-1 up to 2019-2, which should be modelled by eq.13.

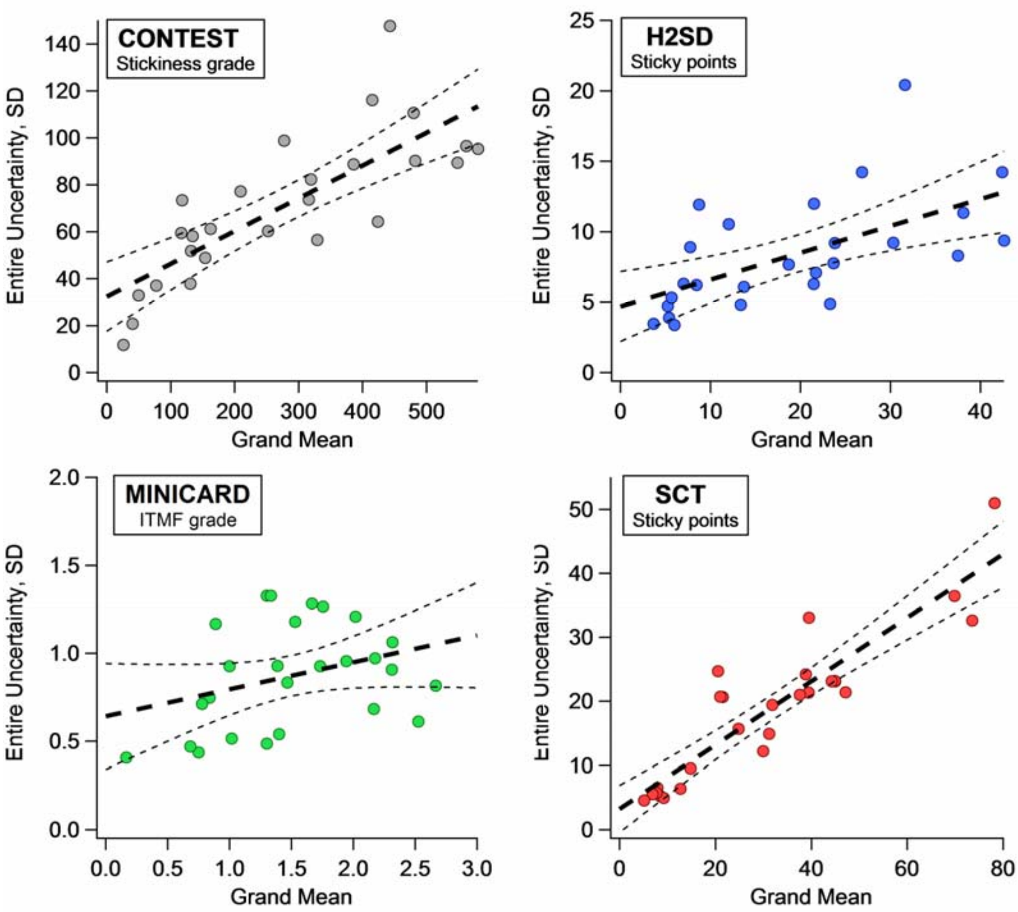


Figure 29: *For each cotton sample of the Stickiness Round Test from 2017-1 up to 2019-2, the entire uncertainty, SD, as a function of the Grand Mean among all the instruments: (top left) CONTEST, (top right) H2SD, (bottom left) MINICARD, (bottom right) SCT.*

In spite of some discrepancies, all the methods seem to exhibit a common trend of increasing uncertainty, SD, with the stickiness evaluation, which should not be ascribed to the method themselves, but rather it follows the intra-sample Poisson-type distribution of results, which is typical for stickiness measurements for well homogenized samples[22].

[22] E. Hequet, R. Frydrych and M. Watson, “The use of the High Speed Stickiness Detector on a large range of cotton coming from different countries”, *In Proc. Beltwide Cotton Prod. Res. Conf.*, Memphis, TN. 4-8 Jan. 1997.

16.2 Sample conditioning

On the basis of the results presented in this document, it is unfeasible to properly quantify the uncertainty related to the sample conditioning, since the design of the experiments were not suitably prepared for such investigation.

In first approximation, however, it is possible to provide an indication about how much the conditioning may affect the final result by considering in the reproducibility study of chapter 12.4 (Table 14, Figure 13-14) the laboratories which were out of standard (ASTM D1776) during the period of testing, labelled as EU (RH = +1.5%) and USA (Temp = -0.6°C). With the assumption that both sampling and sample preparation were carried out properly, such a discrepancies from the standard led to a mean increase of the stickiness evaluation among all the cotton samples (A-E) of about +14.2% for EU and -15.5% for USA for the St Grade.

16.3 Operator influence

About the influence of the operator on the stickiness evaluation, two cases should be discerned depending on the sample preparation.

If the sample is unproperly prepared, the inaccuracy is proportional to the deviation from the recommended weight (3.5 g): for instance, for 1.75 g instead of 3.5 g, the final evaluation for St Cnt (or St Grade) would be approximately the half. Thereby, the sample weight can affect the scaling to 1 gram (exactly as in the other methods).

Otherwise, if the sample weight is within the recommended limits of 3.5 ± 0.2 g, the maximum uncertainty related to the sample preparation should be approximately about ±15 sticky points on the total count (St Cnt) for the max ever result (250 cnt/g) recorded up to now.

In this situation, as long as the sample is prepared as per the recommended instructions for weight, length and shape (refer to Chapter 8.2 for the detailed description), the stickiness measurement should be generally almost independent of the operator. In order to provide a demonstration of this, a special test session was designed with the following criteria:

- the same 5 cotton samples (A-E) of chapter 12 were used;
- a total of 10 tests per cotton for each operator were performed;
- the samples were alternated after each test (like in chapter 12.3 and 12.4);
- 4 different operators were selected with different level of skill;
- for each sample, 2 different shifts (of 5 tests) per operator were considered;
- the sequence of the operators was changed from the first to the second shift;
- all the samples were tested in only one day (200 tests in total);
- the instrument was cleaned only once before starting the test session.

In addition, different skills were considered for the selection of operators:

- **MASSIMO** (MAS): high trained skills on the instrument;
- **SERGIO** (SER): medium trained skills on the instrument;
- **RUGGERO** (RUG): low trained skills on the instrument;
- **DANIEL** (DAN): never used the instrument before.

For each cotton A-E, Figure 30-34 show: (top left) the distribution of all the results for all the operators, (top right) the sequence of the distributions of results for each operator/shift, (bottom) all the results along the sequence of the operator shifts[23].

Table 20 - 24 list all the results, as well as the statistic for each operator.

Finally, an ANOVA study is provided in order to statistically evaluate the influence of different operators on the stickiness grade, as displayed in Table 25.

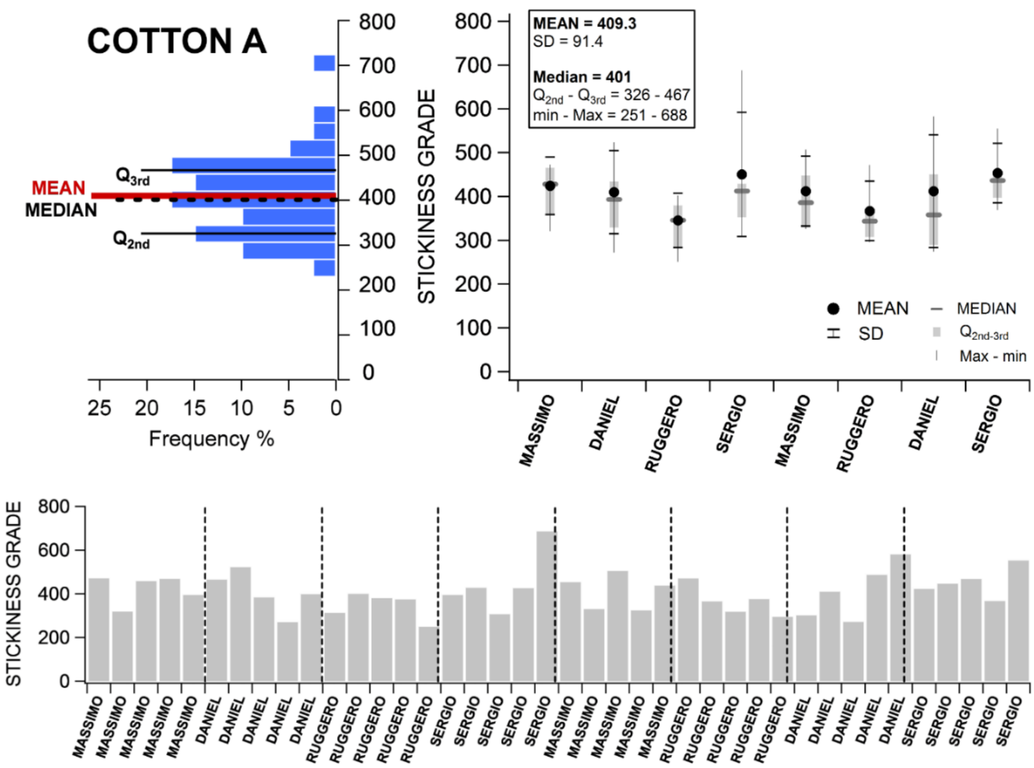


Figure 30: *Cotton A: (top left) distribution of all the results (200 tests); (top right) distribution of results per operator along the sequence of shifts, (bottom) all the results along the sequence of shifts.*

[23] The actual sequence of all the results, in which the A-E samples were alternated after each test by each operator, like in chapter 12.3-12.4, is here omitted because it is not the focus of the investigation.

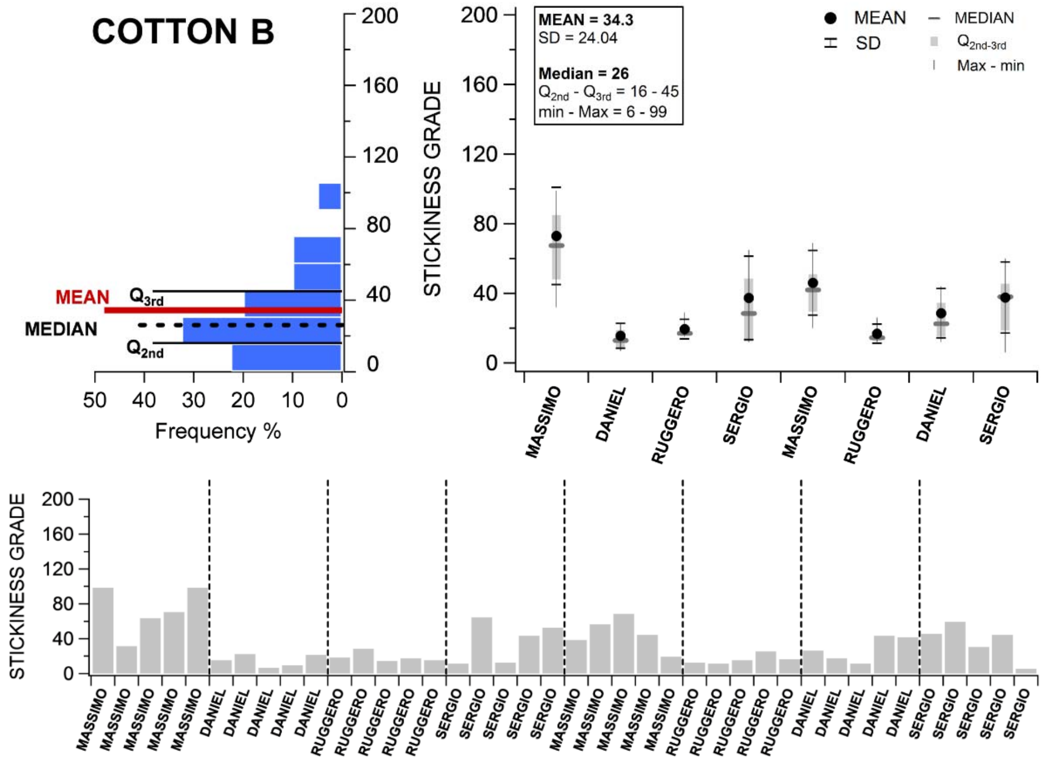


Figure 31: *Cotton B: (top left) distribution of all the results (200 tests); (top right) distribution of results per operator along the sequence of shifts, (bottom) all the results along the sequence of shifts.*

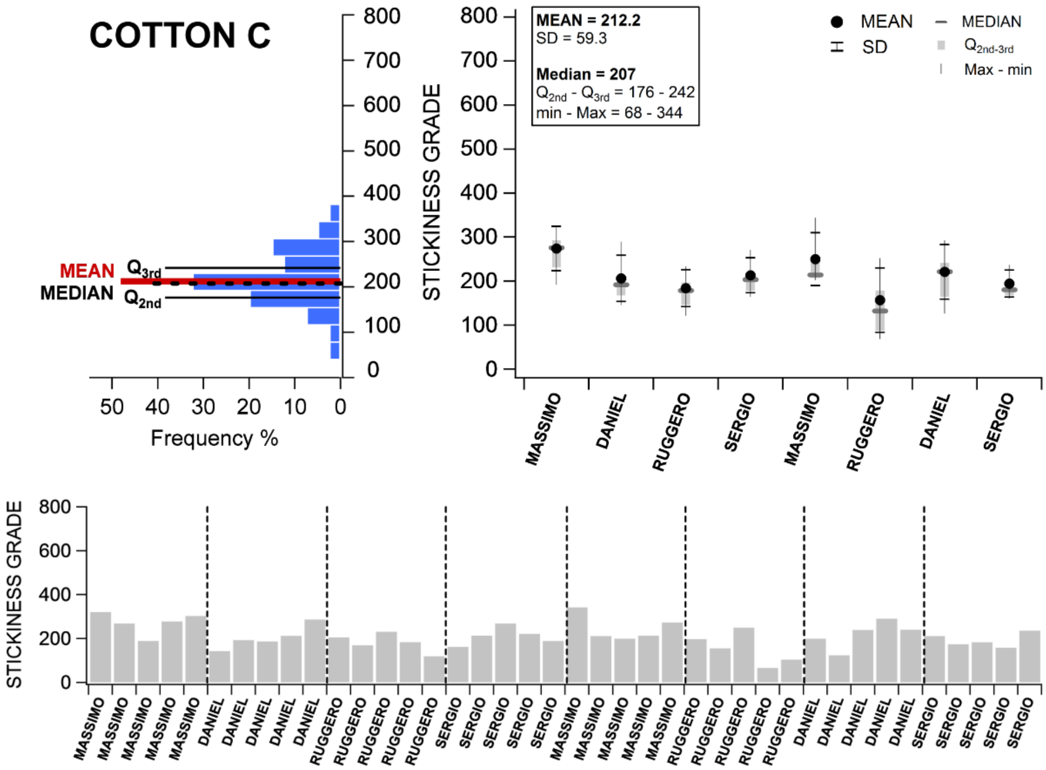


Figure 32: *Cotton C: (top left) distribution of all the results (200 tests); (top right) distribution of results per operator along the sequence of shifts, (bottom) all the results along the sequence of shifts.*

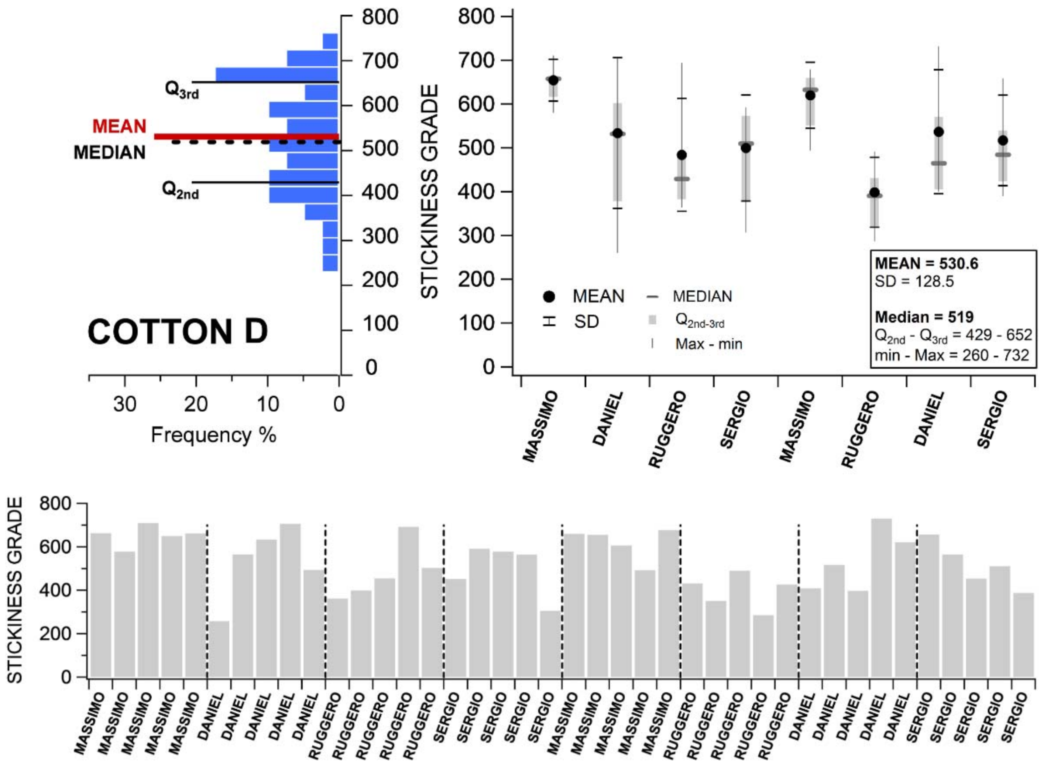


Figure 33: *Cotton D: (top left) distribution of all the results (200 tests); (top right) distribution of results per operator along the sequence of shifts, (bottom) all the results along the sequence of shifts.*

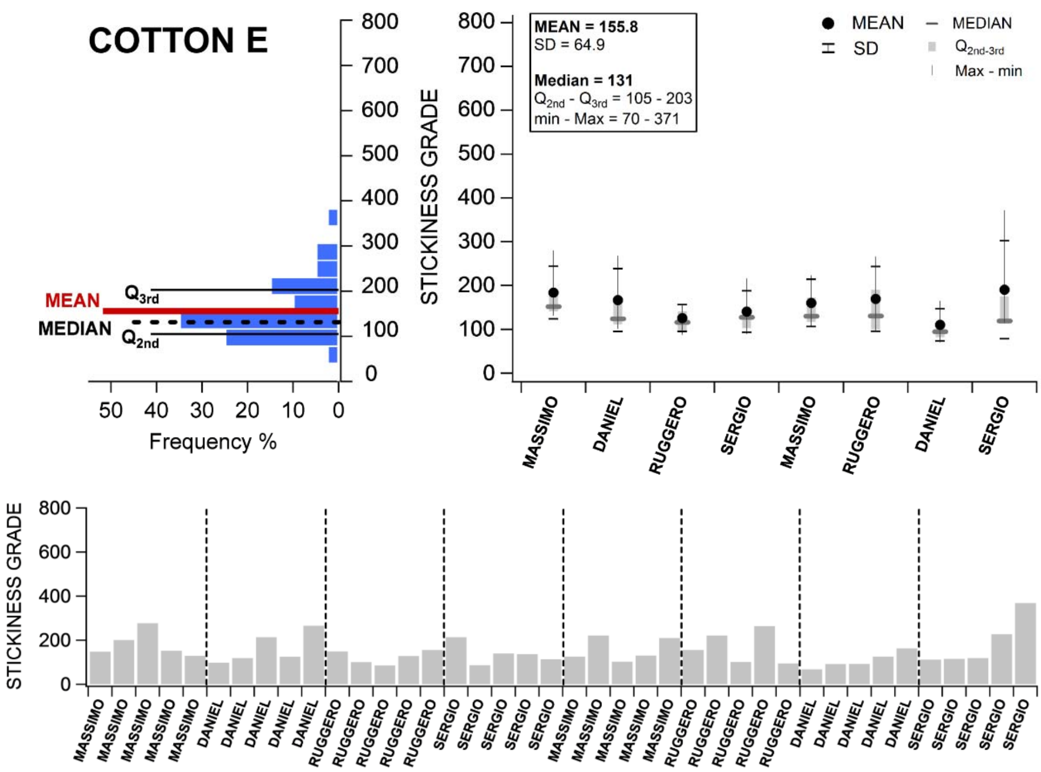


Figure 34: *Cotton E: (top left) distribution of all the results (200 tests); (top right) distribution of results per operator along the sequence of shifts, (bottom) all the results along the sequence of shifts.*

COTTON A					
OPERATOR		MAS	DAN	RUG	SER
SKILL		HIGH	BASIC	LOW	MEDIUM
SHIFT 1	*test 1*	473	467	315	397
	test 2	321	524	402	430
	test 3	460	386	382	309
	test 4	471	272	377	428
	test 5	397	401	251	688
SHIFT 2	*test 6*	455	304	472	425
	test 7	332	412	368	448
	test 8	507	274	320	470
	test 9	326	489	378	369
	test 10	440	583	296	555
Mean		**418**	**411**	**356**	**452**
Median		448	407	373	429
SD		69.2	106.4	62.3	104.8
CV%		16.5	25.9	17.5	23.2
Q95%		48.7	74.9	43.9	73.8

Grand Mean of all operators (4)	**409.4**
Median of all results (40)	401.5
SD of all results (40)	91.4
SD between operators (4)	39.7
CV% between operators (4)	9.7

COTTON B					
OPERATOR		MAS	DAN	RUG	SER
SKILL		HIGH	BASIC	LOW	MEDIUM
SHIFT 1	*test 1*	99	16	19	12
	test 2	32	23	29	65
	test 3	64	7	15	13
	test 4	71	10	18	44
	test 5	99	22	16	53
SHIFT 2	*test 6*	39	27	13	46
	test 7	57	18	12	60
	test 8	69	12	16	31
	test 9	45	44	26	45
	test 10	20	42	17	6
Mean		**60**	**22**	**18**	**38**
Median		61	20	17	45
SD		26.5	12.6	5.4	21.0
CV%		44.5	57.1	30.0	55.9
Q95%		18.7	8.9	3.8	14.8

Grand Mean of all operators (4)	**34.3**
Median of all results (40)	26.5
SD of all results (40)	24.0
SD between operators (4)	18.8
CV% between operators (4)	54.7

Table 20 / 21: *Results and statistic per operator: (left, Table 20) Cotton A, (right, Table 21) Cotton B.*

COTTON C					
OPERATOR		MAS	DAN	RUG	SER
SKILL		HIGH	BASIC	LOW	MEDIUM
SHIFT 1	*test 1*	322	145	207	164
	test 2	271	194	171	216
	test 3	191	189	233	271
	test 4	280	214	186	223
	test 5	305	289	121	191
SHIFT 2	*test 6*	344	202	199	213
	test 7	213	126	158	176
	test 8	202	241	252	185
	test 9	215	293	68	160
	test 10	275	242	106	237
Mean		**262**	**214**	**170**	**204**
Median		273	208	179	202
SD		53.7	54.8	57.9	35.0
CV%		20.5	25.7	34.1	17.2
Q95%		37.9	38.6	40.8	24.6

Grand Mean of all operators (4)	**212.3**
Median of all results (40)	210.0
SD of all results (40)	59.3
SD between operators (4)	37.9
CV% between operators (4)	17.9

COTTON D					
OPERATOR		MAS	DAN	RUG	SER
SKILL		HIGH	BASIC	LOW	MEDIUM
SHIFT 1	*test 1*	665	260	364	454
	test 2	580	568	401	593
	test 3	711	636	457	580
	test 4	652	708	694	566
	test 5	664	496	505	307
SHIFT 2	*test 6*	662	411	433	659
	test 7	657	519	353	567
	test 8	608	400	492	456
	test 9	494	732	287	513
	test 10	679	623	429	390
Mean		**637**	**535**	**442**	**509**
Median		660	544	431	540
SD		62.0	148.6	110.6	106.5
CV%		9.7	27.8	25.1	20.9
Q95%		43.6	104.7	77.9	75.0

Grand Mean of all operators (4)	**530.6**
Median of all results (40)	542.5
SD of all results (40)	128.5
SD between operators (4)	81.3
CV% between operators (4)	15.3

Table 22 / 23: *Results and statistic per operator: (left, Table 22) Cotton C, (right, Table 23) Cotton D.*

COTTON E					
OPERATOR		MAS	DAN	RUG	SER
SKILL		HIGH	BASIC	LOW	MEDIUM
SHIFT 1	*test 1*	150	101	151	216
	test 2	203	121	102	89
	test 3	280	216	87	142
	test 4	154	127	130	139
	test 5	131	268	158	116
SHIFT 2	*test 6*	128	70	158	114
	test 7	223	94	223	117
	test 8	105	95	103	121
	test 9	132	128	266	229
	test 10	212	165	97	371
Mean		**172**	**139**	**148**	**165**
Median		152	124	141	130
SD		55.1	61.5	58.3	85.2
CV%		32.1	44.4	39.5	51.5
Q95%		38.8	43.3	41.0	60.0

Grand Mean of all operators (4)	**155.8**
Median of all results (40)	131.5
SD of all results (40)	64.9
SD between operators (4)	15.5
CV% between operators (4)	9.9

Table 24: *Results and statistic per operator for Cotton E.*

The ANOVA approach was used to statistically evaluate the operator influence on the **Stickiness Tester** by comparing the variation between/within operators, as already described in the previous section 12.3 and 12.4. For each cotton i, it can be established that the variance between operators is not significantly different than the variance within operators if it is valid

$$F_i = \frac{Var_i^{between}}{Var_i^{within}} < F(0.95; G-1; N-G) = 2.8663 \quad (16)$$

where 2.8663 is the value of the Fisher-distribution with $G-1=3$ and $N-G=36$ degrees of freedom with a rejection region of 5% (for $G=4$ operators ; $n_G=10$ tests per cotton; $N=G\cdot n_G=40$ results to be compared per cotton). As shown in Table 25, no significant difference between operators is observed for the cotton samples (A-E).

COTTON	F-calculated	F(0.95;3;36)	EVALUATION
A	0.2088	**2.8663**	No significant difference between operators
B	1.0863		No significant difference between operators
C	0.5625		No significant difference between operators
D	0.5474		No significant difference between operators
E	0.0561		No significant difference between operators

Table 25: *ANOVA of the operator influence for all the cottons (A-E).*

17. Maintenance / Service

17.1 Periodical maintenance

To ensure long instrument life, as well as its proper functioning, it is recommended to perform a scheduled maintenance as follows:

- **DAILY**: every day recommended before testing[24] or, equivalently, over a total of 300-400 consecutive tests (estimated time: 10 min);
  - *filters;*
  - *suction hoods dedicated to web formation;*
  - *carding unit (external part only);*
  - *metal wire of DOFFER card (not removed);*
  - *metal wire of TAKE-OFF (removed) by polyester brush and dust cloth;*
  - *blade-mechanism by vacuum cleaner*

- **WEEKLY**: recommended once a week (estimated time: 10 min) (in addition to the daily maintenance):
  - *metal wire of MAIN DRUM card (removed) by metal brush;*
  - *metal wires of all the cards (not removed) by vacuum cleaner;*
  - *air switches, hoses, pipes and suction nozzles;*

- **MONTHLY**: recommended once a month (estimated time: 20 min). (in addition to the weekly maintenance):
  - *metal wires of all the cards (removed) by metal brush.*

The maintenance program requires specific actions of easy cleaning from any accumulation of fiber dust by means of compressed air blast or vacuum cleaner for the components. All the maintenance actions are described in detail in the Instruction & User Manual in Chapter 6 for CONTEST-F and in Chapter 7 for CONTEST-S.

17.2 Service

The Official Assistance Service provides to the users the following support:

1. Full maintenance of the instrument (once a year, especially recommended in case of intensive processing of sticky samples);
2. Online assistance (remote connection with the instrument);
3. Official Assistance Service available all around the world.

Moreover, an offline troubleshooting is provided on the video whenever a machine error message occurs.

[24] The recommendation is valid only if a sufficient number of tests was already overcome in the previous days, i.e. a total of 200 tests at least.

18. Additional information

18.1 Sample preparation for recognition

For the entire recognition document[25], 5 different cotton samples were selected to cover the whole detection range of the **Stickiness Tester** (from "zero" up to the maximum ever detected in ITMF-ICCTM Round Test in 2017-2019).

18.2 Homogenized cotton

The cotton, which exhibits the highest stickiness level[26], was provided by the Laboratoire de Technologie et de Caractérisation des fibres naturelles, CIRAD-PERSYST, 73 rue Jean-François Breton, 34398 Montpellier Cedex 5, FRANCE. Its provenience is from the same lot used in the ITMF-ICCTM Stickiness Round Test 2019-1, cotton E.

The preparation of this cotton was done by an homogenizing machine developed during the CFC/ICAC/33 project (so called CSITC homogenizing machine), in such a way to ensure that any drawn sample from the original mass would carry the "same" stickiness potential as any other sample, without affecting too much the size of individual sticky points that could affect some measurement methods, such as in particular the **CONTEST- Stickiness Tester**.

The delivery of the sample was provided with a certification of stickiness by using the SCT thermodetector (Mean = **79**, Tol- = 67, Tol+ = 92, unit = sticky points), which is consistent with the average inter-laboratory result obtained in the SRT2019-1 for Cotton E ($Mean_{interLab}$ = **82.8**, $SD_{interLab}$ = 55.5, unit = sticky points).

18.3 Manufacturer-mixed cottons

The remaining 4 cottons, which cover the lower half of the detection range of the **Stickiness Tester**, were provided by the manufacturer and prepared as follows:

[25] Except of chapter 14, in which all the results of ITMF-ICCTM Round Tests were collected on homogenized cottons provided by the Stickiness Task Force.
[26] Corresponding to the maximum detection in the CONTEST Stickiness Grade.

- **Sampling from the bale – step 1**
  The sampling was carried out by manually picking up portions of material from a delimited area[27] of single bales of different origin (Figure 35, left). The layers are then overlapped to create the starting sample, as shown in Figure 35 (right).

Figure 35: *Step 1: (left) different portions taken from a single bale, (right) overlapping of the portions to create the starting sample.*

- **Subsample division – step 2**
  From the starting sample at the end of the step 1, a manual division was exerted in order to have first 2 subsamples, then 4 subsamples and finally 16 subsamples, as shown in Figure 36. Figure 37 schematically describes the sequence of random mixing and how the subsamples were overlapped.

Figure 36: *The 16 subsamples after the 1st subdivision from the starting sample (step 2).*

[27] In order to limit the variability, which derives from the within-variability of the entire bale.

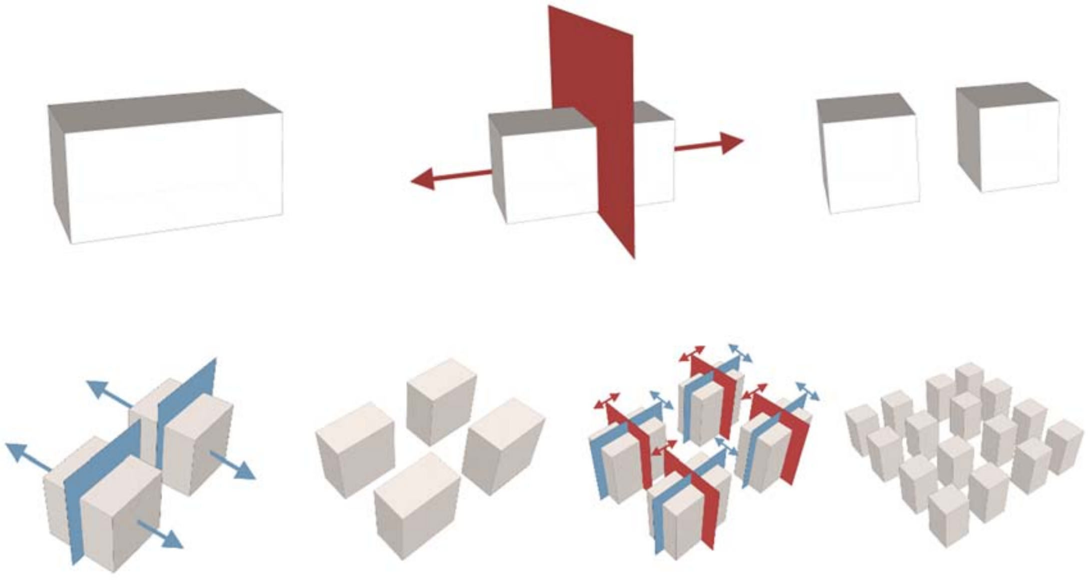


Figure 37: *Step 2: (from top left to bottom right) diagrams for the subdivision of the starting sample in 16 different subsamples.*

- **Mixing – step 3**
  At this stage, the 16 subsamples are first stretched, as displayed in Figure 38,

Figure 38: *Preparation of subsamples for the mixing.*

and then overlapped with a precise sequence in order to randomly mix all the subsamples, thus obtaining again a single sample. See Figure 39 and 40 for the mixing sequence, which was followed.

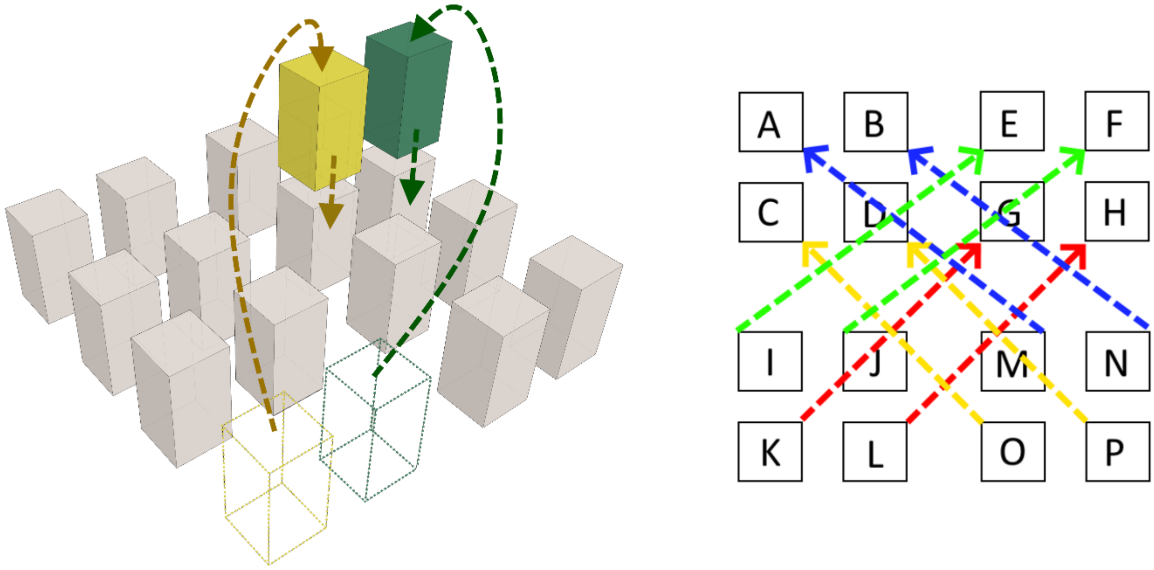


Figure 39: *(left) Diagram to show how the mixing was executed; (right) combinations of subsamples for the mixing (step 3).*

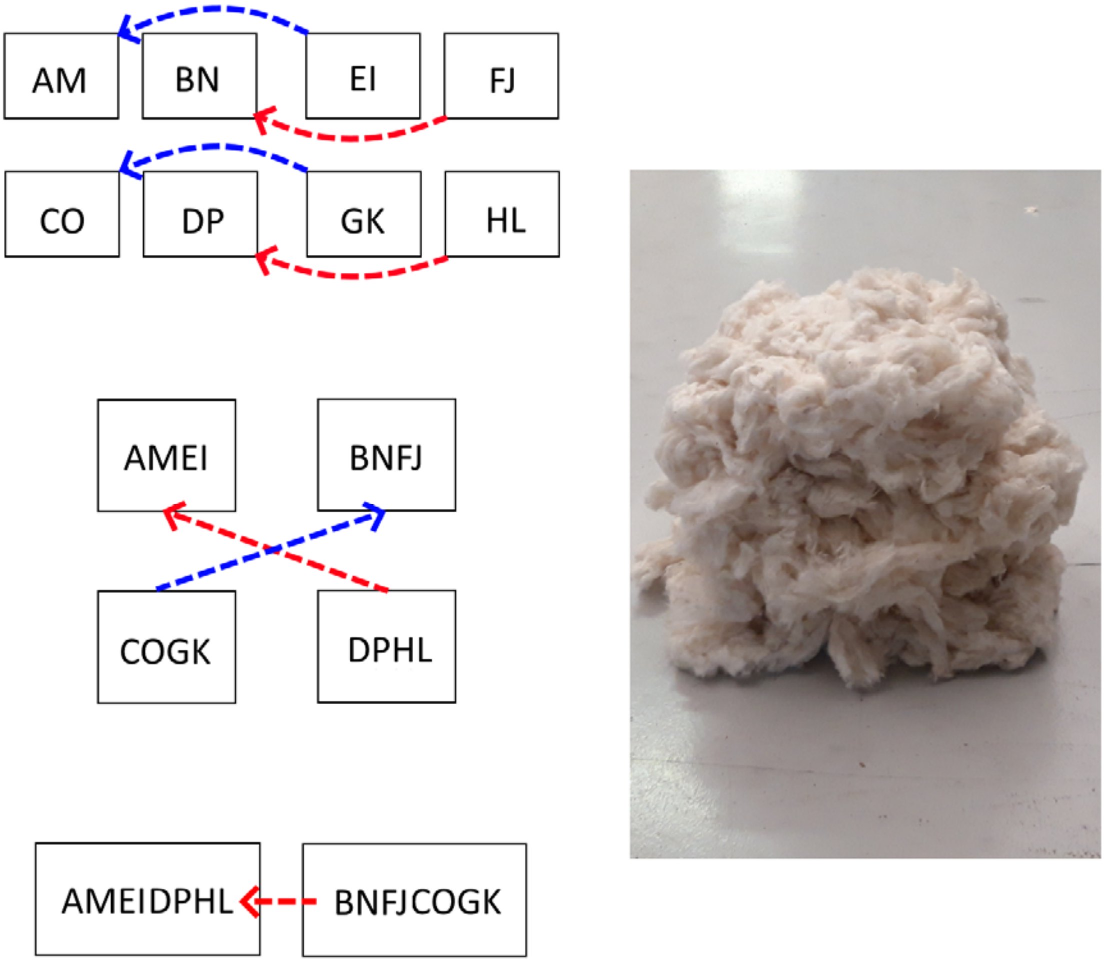


Figure 40: *(left) From top to bottom: mixing sequence until to recreate a single sample; (right) the sample at the end of the first mixing procedure.*

- **Repetition n.1 – step 4**
  From the single sample obtained at the end of step 3, step 2 and 3 are repeated for the second time, increasing the mixing.

Figure 41: *(left) The 16 subsamples after the 2nd subdivision from the sample of step 3; (right) the sample at the end of the second mixing procedure.*

- **Repetition n.2 – step 5**
  From the single sample obtained at the end of step 4, step 2 and 3 are repeated again for the third time, further increasing the mixing. Thus, the final sample is reached.

Figure 42: *(left) The 16 subsamples after the 3rd subdivision from the sample of step 4; (right) the sample at the end of the third mixing procedure, ready to be used in testing.*

The target of mixing procedure described above has not to be intended as the achievement of a level of homogenization comparable like by using the CSITC machine (which is not available at the manufacturer), but rather the target is simply to obtain a good level of mixing of small portions, in such a way that any set of 6 subsamples from the final sample of step 5 should bring the same variability of stickiness as any other set of 6 subsamples, thus preserving the same average of stickiness level.

19. Technical Data / Instrument settings

19.1 Technical features

Stickiness count (St Cnt)	Total count / g
Stickiness average size (St Size)	From 1 to 5 [a.u.]
Stickiness classes (St Class)	5 classes (from 1 to 5)
Stickiness grade (St Grade)	Based on stickiness counting and classification
Software language	English
Screen size	Wide touch screen monitor
Network capability	Provided via Ethernet port
Backup	Storage of test results
Testing speed	About 30-60 seconds / sample

19.2 Compressed air conditions

Air pressure	6 bar
Dirt particles air filter (size)	5 microns
Air flow (average)	40 litres/min
Inlet air quality class	3.7.4 (according to ISO 8573)

19.3 Dimensions / Power supply

Weight	340 Kg
Dimensions	(L) 1510 x (W) 960 x (H) 1410 mm
Power supply	230 Vac, 50/60 Hz, single-phase, 2 kW

20. Manufacturer contacts for information

20.1 Company and contact information

MESDAN S.p.A.
Via Masserino, 6
25080 Puegnago del Garda (BS) - ITALY
Export Department: Tel +39-0365-653142, e-mail: sales@mesdan.it
Telefax +39-0365-651011
www.mesdan.com

20.2 Contact person

Gabriele Salvinelli, Ph.D.
Mesdan Lab - Research and Development
Email: salvinelligabriele@mesdan.it

21. Responsible ITMF ICCTM Coordinators

Jean-Paul Gourlot, Ph.D.
Email: jean-paul.gourlot@cirad.fr

René van der Sluijs, Ph.D.
Email: sluijs@optusnet.com.au

Approved version, March 2020

22. Additional information per peer reviewers

22.1 Annex for the Repeatability on similar test material

The repeatability study is decribed in details in Chapter 12.3. In this section, all the results for all the days are represented in the same sequence as they were performed in order to evidence potential contamination of results from one specimen to the subsequent one.

The sequence of samples was designed in such a way that the cottons characterized by the highest stickiness, A and D, were followed by the cottons with the lowest stickiness, B and E; in this way, it is possibile to focus on potential contamination whenever the results of the low stickiness samples, B and E, tend to align with the previous test results, significatly increasing their typical values, namely the mean values of all the results per cotton.

Figure 43 – 57 show all the results per day: black dots represent the mean values per cotton over all the results ± their standard deviation, whereas the bars stand for the single tests.

As evidenced, no significant contamination effects are shown along all the results.

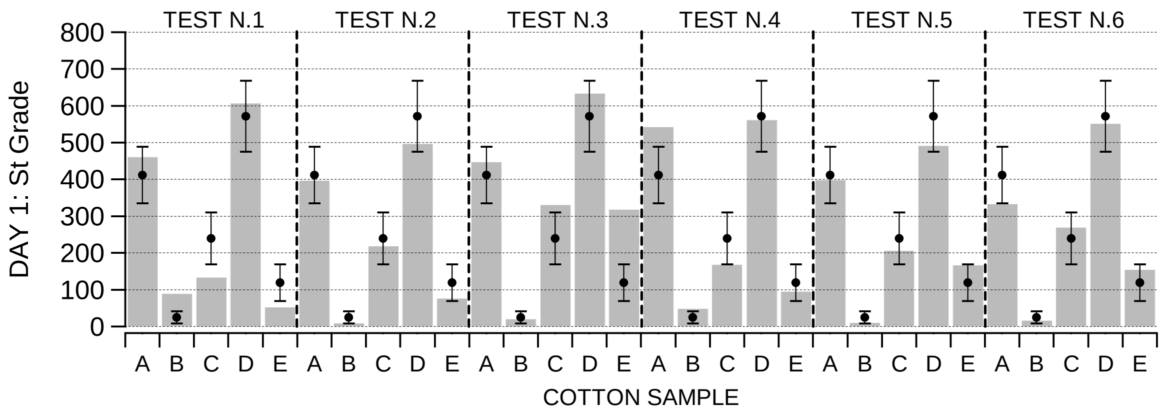


Figure 43: *Bars = day 1 results of the repeatability in Chapter 12.3. Black dots = Mean ± SD for all days.*

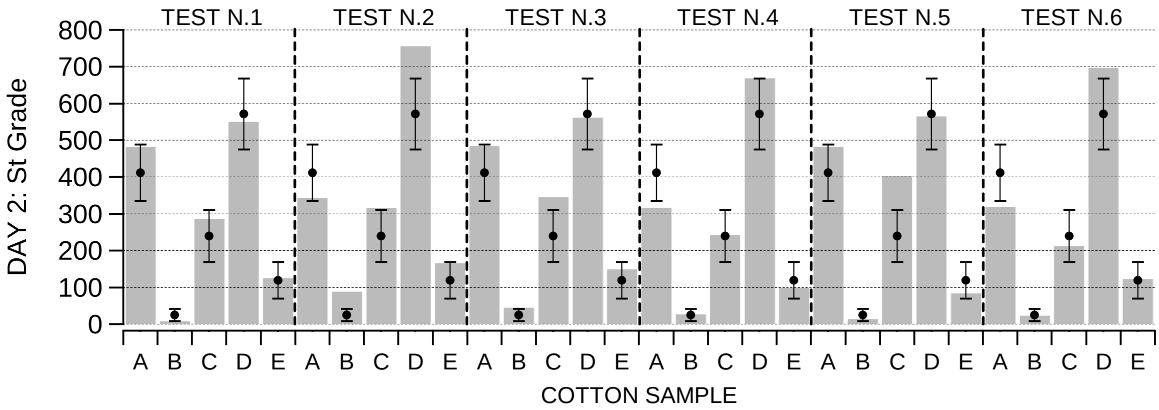


Figure 44: *Bars = day 2 results of the repeatability in Chapter 12.3. Black dots = Mean ± SD for all days.*

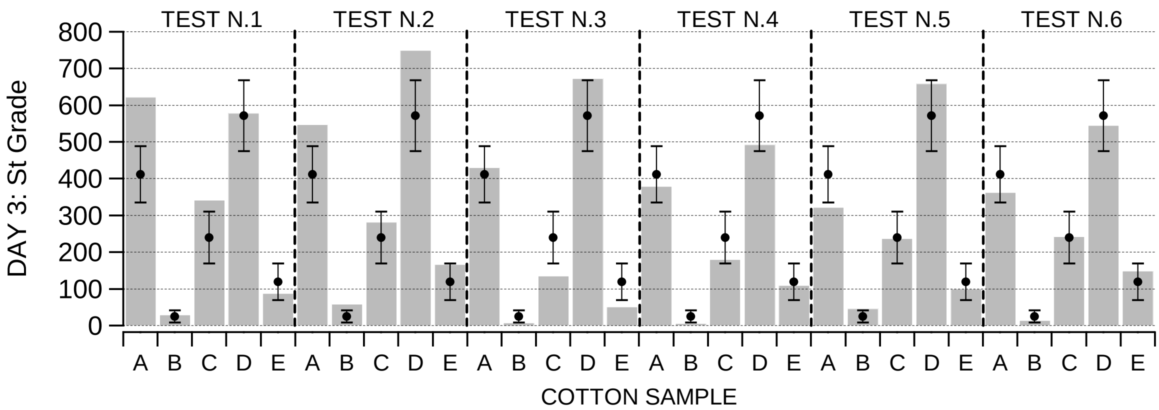


Figure 45: *Bars = day 3 results of the repeatability in Chapter 12.3. Black dots = Mean ± SD for all days.*

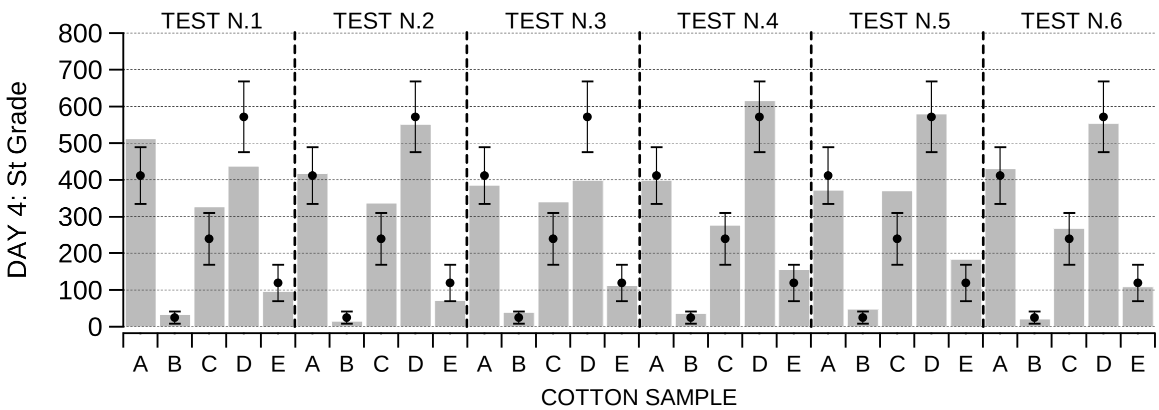


Figure 46: *Bars = day 4 results of the repeatability in Chapter 12.3. Black dots = Mean ± SD for all days.*

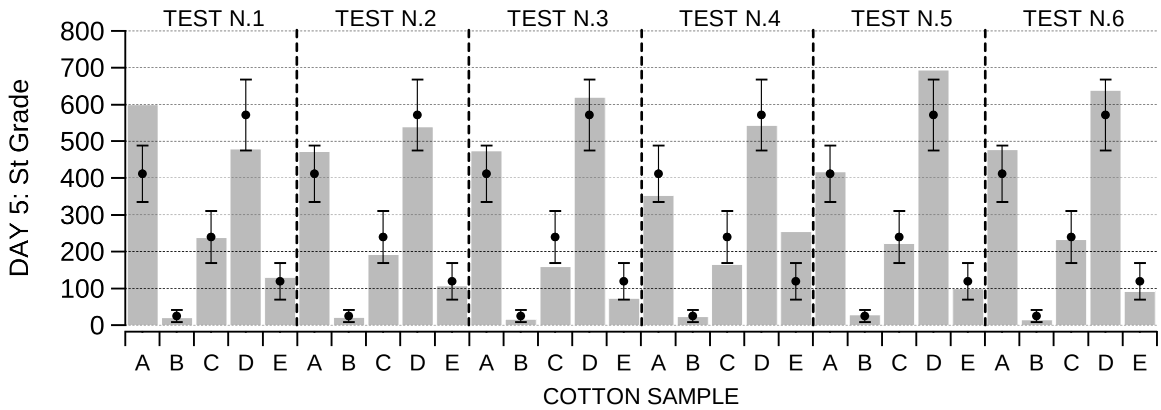


Figure 47: *Bars = day 5 results of the repeatability in Chapter 12.3. Black dots = Mean ± SD for all days.*

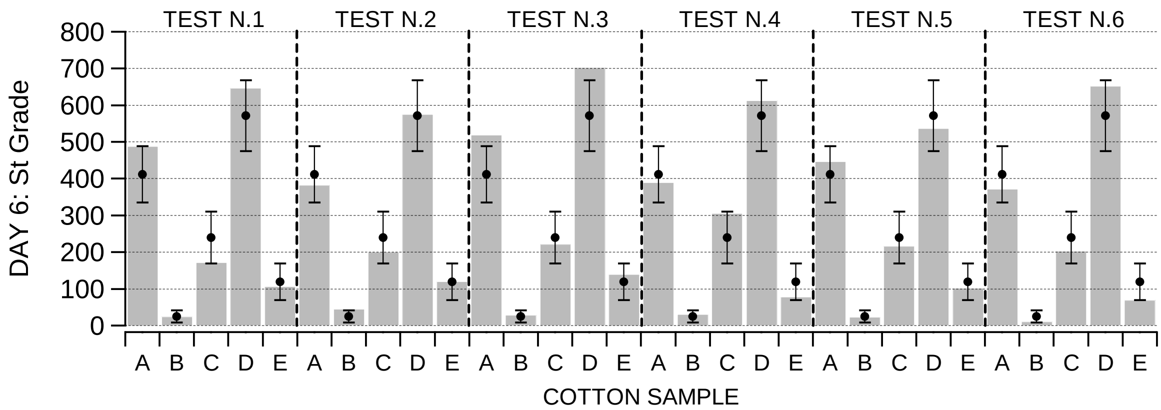


Figure 48: *Bars = day 6 results of the repeatability in Chapter 12.3. Black dots = Mean ± SD for all days.*

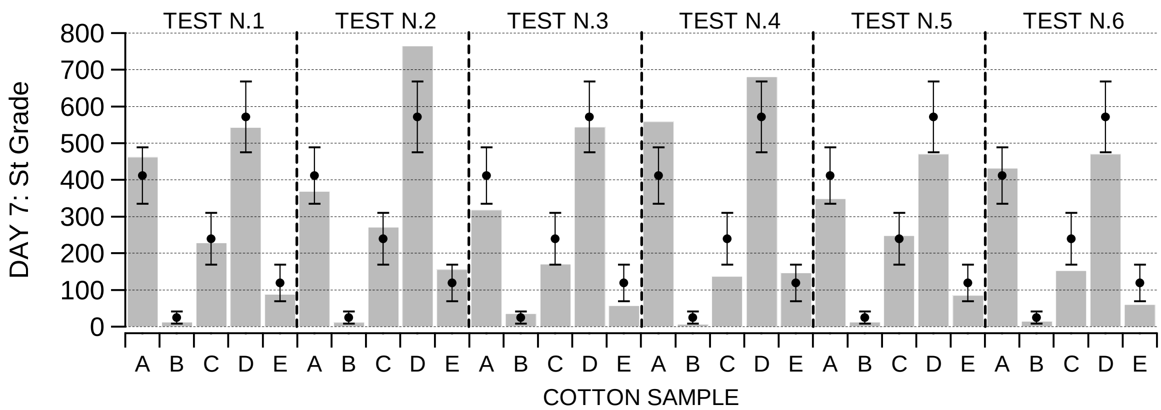


Figure 49: *Bars = day 7 results of the repeatability in Chapter 12.3. Black dots = Mean ± SD for all days.*

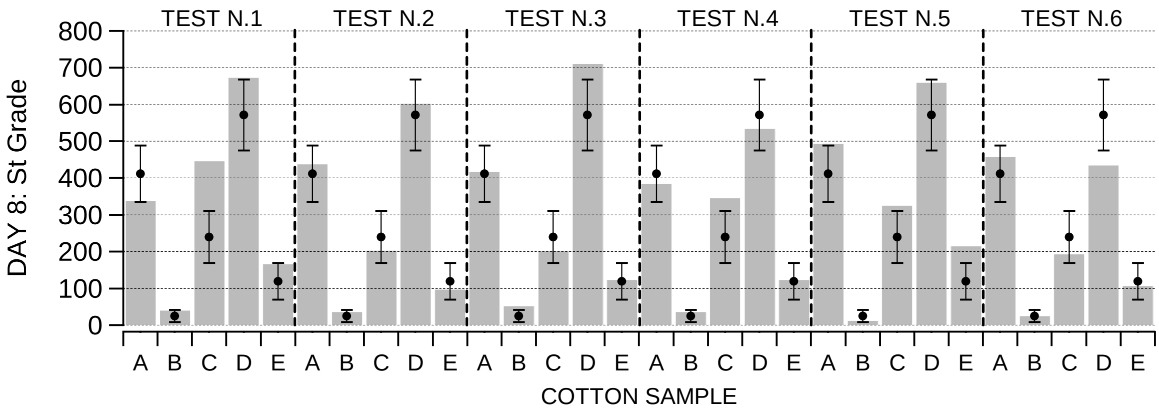
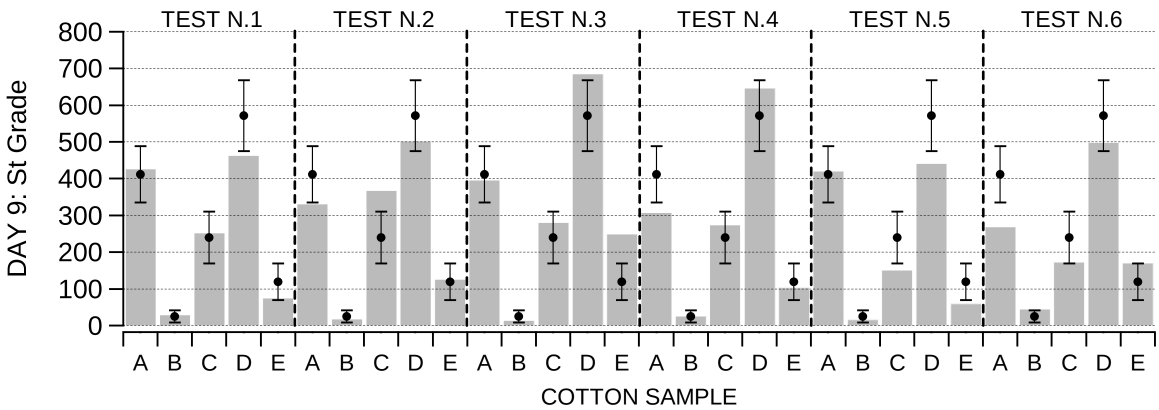


Figure 50: *Bars = day 8 results of the repeatability in Chapter 12.3. Black dots = Mean ± SD for all days.*



FigureF 51: *Bars = day 9 results of the repeatability in Chapter 12.3. Black dots = Mean ± SD for all days.*

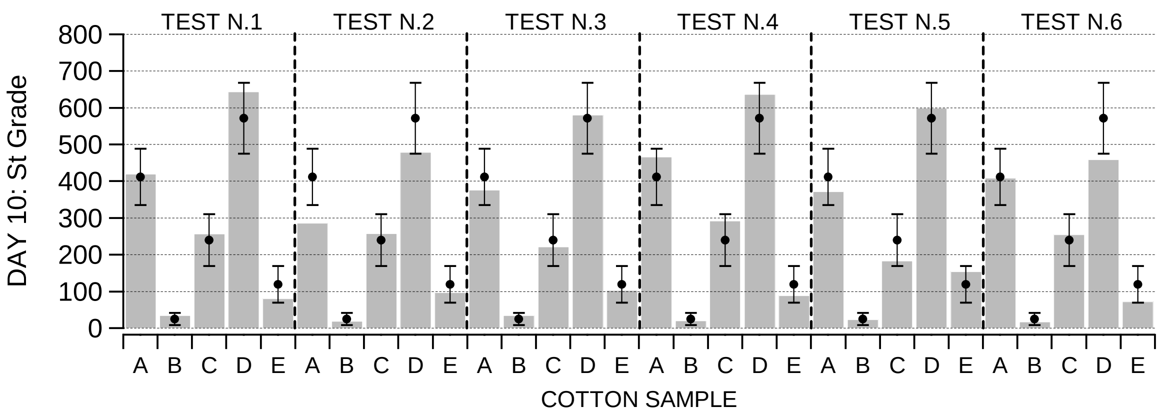


Figure 52: *Bars = day 10 results of the repeatability in Chapter 12.3. Black dots = Mean ± SD for all days.*

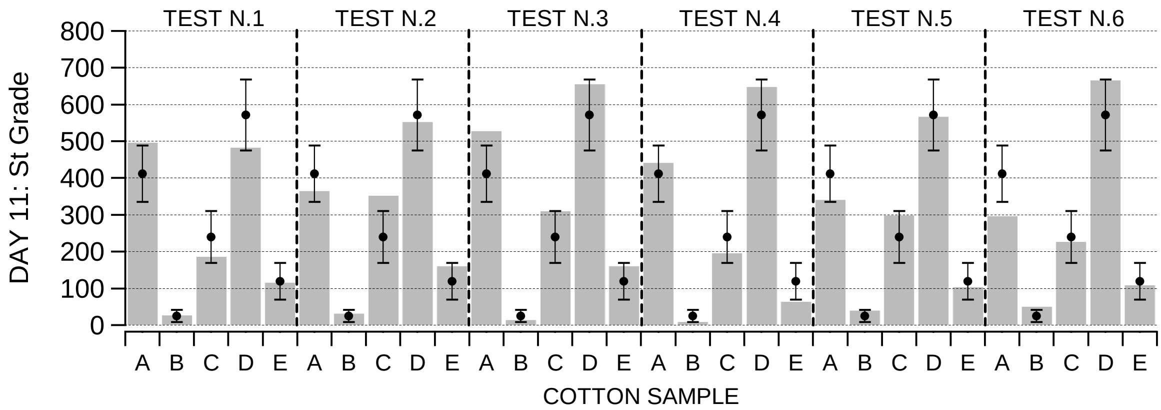


Figure 53: *Bars = day 11 results of the repeatability in Chapter 12.3. Black dots = Mean ± SD for all days.*

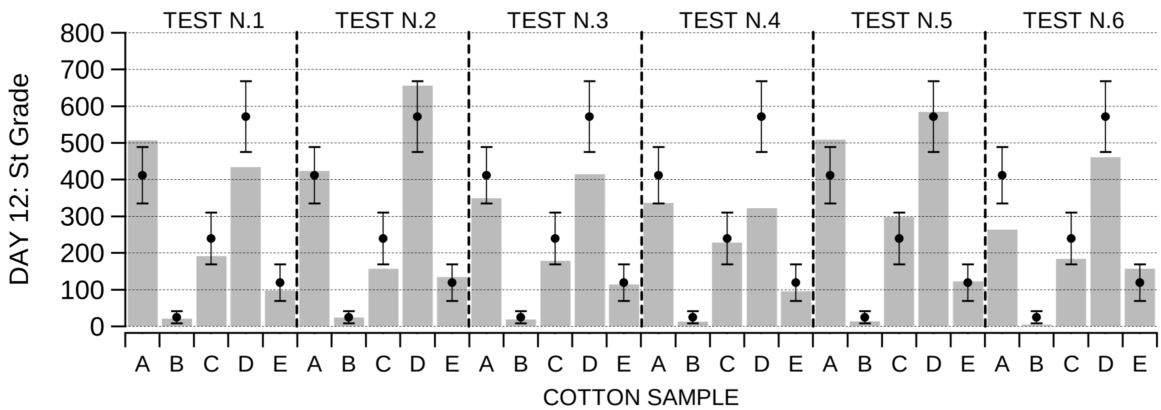


Figure 54: *Bars = day 12 results of the repeatability in Chapter 12.3. Black dots = Mean ± SD for all days.*

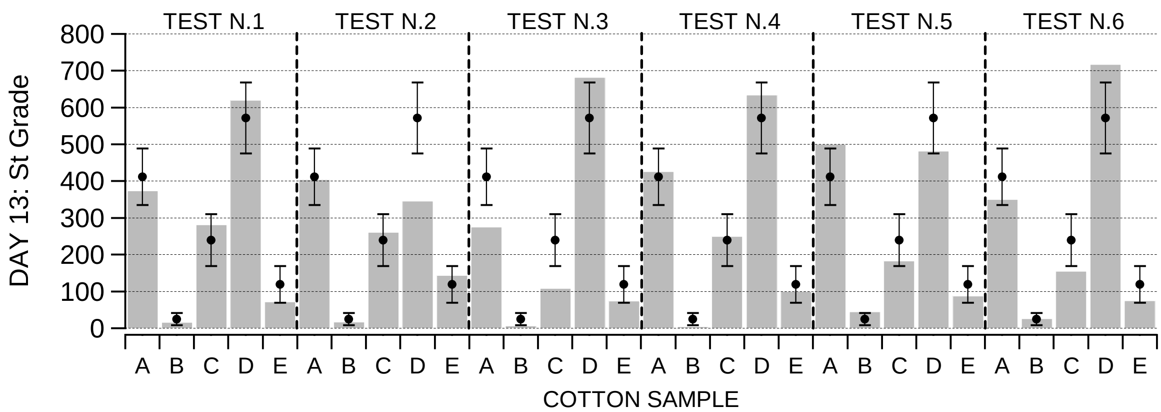


Figure 55: *Bars = day 13 results of the repeatability in Chapter 12.3. Black dots = Mean ± SD for all days.*

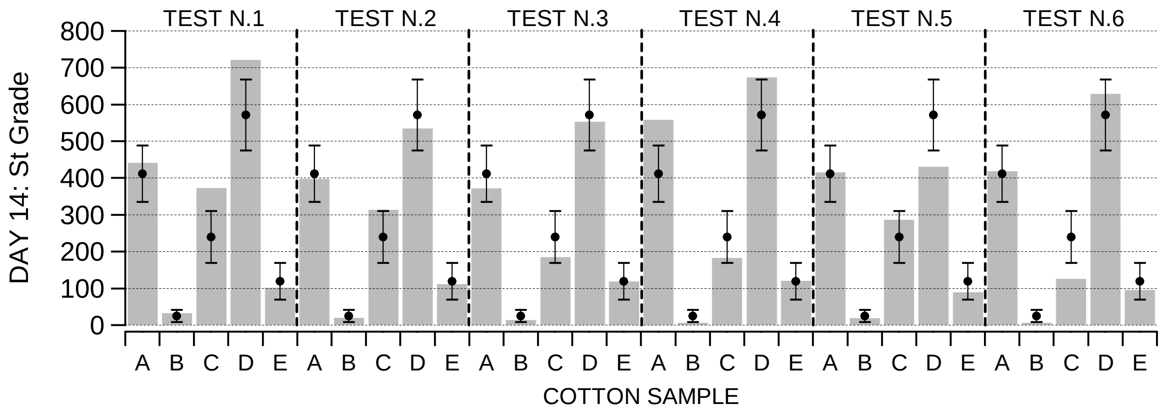


Figure 56: *Bars = day 14 results of the repeatability in Chapter 12.3. Black dots = Mean ± SD for all days.*

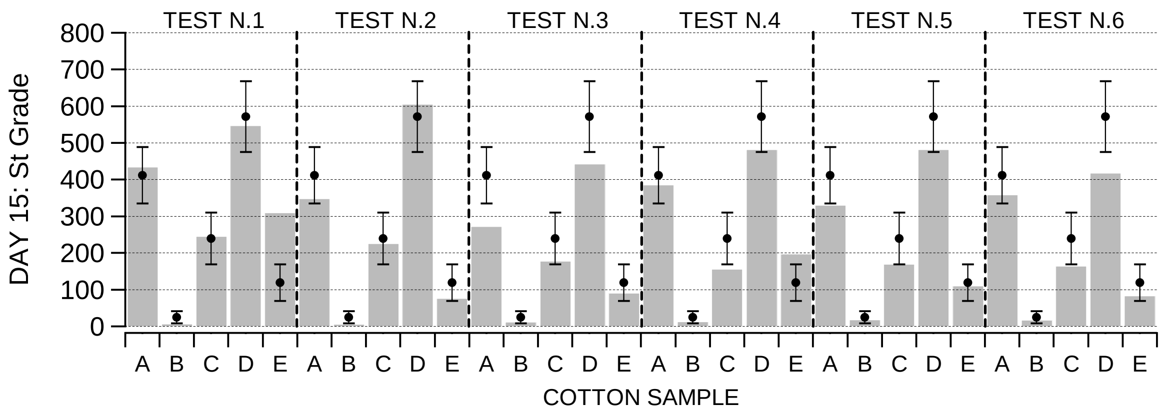


Figure 57: *Bars = day 15 results of the repeatability in Chapter 12.3. Black dots = Mean ± SD for all days.*

22.2 Annex concerning the contamination issue

To provide additional information about the potential contamination of drum surfaces between samples (and not only between specimens, as discussed in paragraph 22.1), a new test session was designed in such a way that some tests for certain samples were performed consecutively – as in normal testing – to be followed immediately after by some other tests on another kind of sample with a strongly different stickiness.

By using the remaining material from the study of chapter 12 for cotton B (the lowest in stickiness grade) and cotton D (the most sticky), it was carried out:

- 5 tests on cotton D (5 min);
- 5 tests on cotton B (5 min);

- 10 tests on cotton D (10 min);
- 10 tests on cotton B (10 min);

- 20 tests on cotton D (20 min);
- 20 tests on cotton B (20 min);

within the same day, with the same instrument, by the same operator and performing only one cleaning at the beginning of the experiment. In this way, it was provided information about a normal test session (5 min, 5 tests), as well as a long test session (10 min, 10 tests) and a very long test session (20 min, 20 tests), which rarely is performed in ordinary situations, except of research activities.

Finally, the choice of testing only cotton B and D was to reduce the number of tests instead of involving several samples with intermediate levels of stickiness: the maximum gap of St Grade between cotton B and D (considering the whole range of detection) stand for a guarantee to have applied the worst case scenario concerning the drum cleaning, in order to clearly certify its real efficiency.

Figure 58 summarizes all results: for all results per cotton, the thick-dotted lines represent the mean values, whereas the thin-dotted lines stand for the SD. The green thicks above data (Figure 58) show the extra cleaning cycles, which the instrument automatically performs in order to prevent the contamination, thus providing reliable results. The extra cleaning cycles are of two types:

1. light auto cleaning of the drums (about 5 sec);
2. heavy auto cleaning of the device (about 20 sec).

In both cases, the user is warned about the extra auto cleaning of the system, which last only few seconds before to restart testing.

As noted in Figure 58, no contamination is evidenced in the first tests of cotton B after the sequence of tests on cotton D in all the three different sessions. Table 26 and Figure 59 summarize the results of Mean, SD, CV, Median, Max and Min.

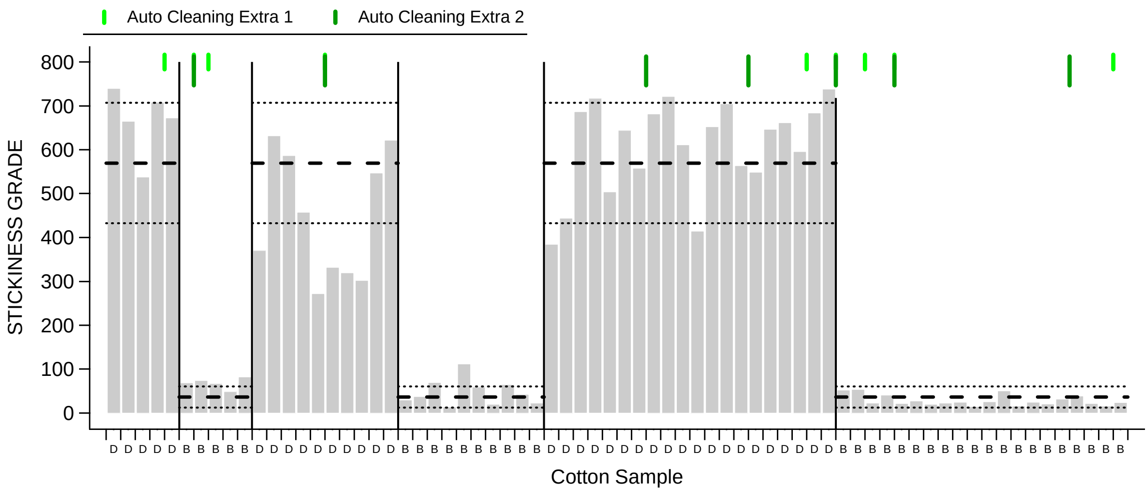


Figure 58: *Consecutive tests on two samples: cotton D (highest sticky) and B (lowest sticky). Three sessions with a different number of tests are displayed: 5 tests, 10 tests and 20 tests. (top) The green ticks display the occurrance of extra auto cleaning.*

COTTON		D	B
5 TESTS	**Mean**	**665**	**68.4**
	SD	77	12.4
	CV%	11.5	18.2
	Median	669	68
	Max	740	83
	min	538	49
10 TESTS	**Mean**	**444.3**	**47.4**
	SD	141	29.8
	CV%	32	62.9
	Median	371	38
	Max	632	112
	min	272	14
20 TESTS	**Mean**	**608.4**	**28.8**
	SD	105	12.2
	CV%	17.2	42.5
	Median	644	24
	Max	739	54
	min	385	15
ALL TESTS	**Mean**	**569.6**	**36.4**
	SD	138	23.6
	CV%	24.2	65
	Median	604	25
	Max	740	112
	min	272	9

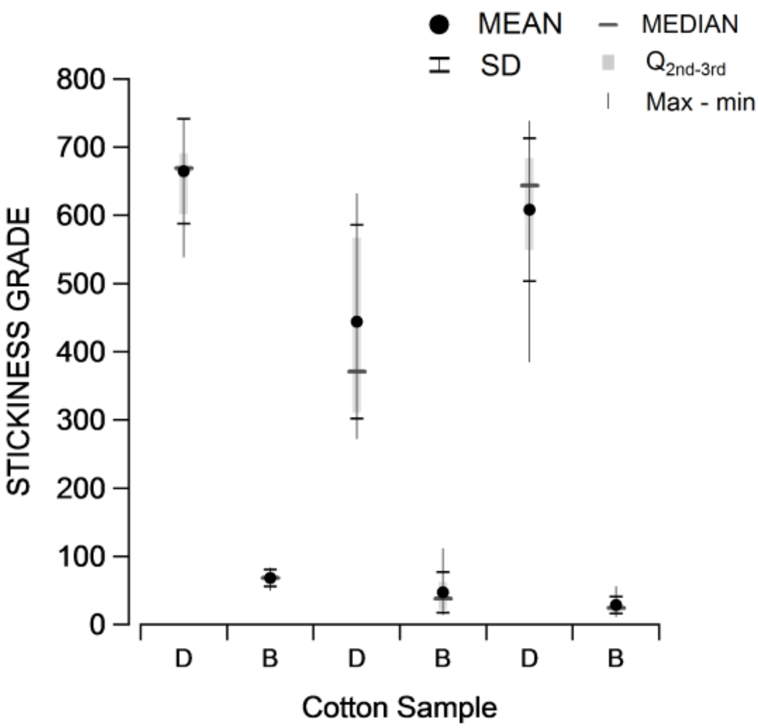


Table 26*: (left) Statistic for each session of Figure 58: 5 tests, 10 tests, 20 tests on cotton B and D.*
Figure 59*: (right) Graphical representation of the results listed in Table 26.*