

ICA BREMEN CERTIFICATION OF LABORATORIES B) TECHNIAL PREREQUISITES AND ACCORDING CSITC ROUND TRIAL DATA

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INTRODUCTION

It is unquestionable that instrument testing of cotton is extremely important and beneficial for today's cotton business, and hence most cotton producing countries move towards instrument testing. Already now, nearly 50% of all bales produced worldwide are tested accordingly. The ICAC Task Force on Commercial Standardization of Instrument testing of Cotton (CSITC Task Force) was installed in 2003/2004 in order to care for standardized / harmonized testing and therefore comparable instrument test results, suitable for commercial use. Cotton testing laboratories have to be capable to produce the same test results for the same cotton – independently from where the laboratory is located.

How can this be achieved? Laboratories have to care for good laboratory practice and have to intensely compare their results to those of other labs. For this purpose,

- The CSITC Task Force as well as the ITMF International Committee on Cotton Testing Methods (ITMF ICCTM) will jointly issue a "Guideline for Commercial Standardization of Instrument Testing of Cotton" [ICAC/ITMF 2012] for approval during this conference week.
- The CSITC Round Trials were developed and installed, comparing now 80 laboratories with nearly 140 instruments, evaluating the results, and helping laboratories in improving their accuracy and precision – in cooperation between the ICAC, USDA-AMS and the Bremen Fibre Institute (FIBRE).
- Finally ICA Bremen, a joint venture between ICA Ltd, Liverpool/UK, and Bremer Baumwollboerse, Germany, in cooperation with the Bremen Fibre Institute (FIBRE), is developing a laboratory certification for assuring high level laboratory performance. Certification will start in 2012.

The question is: Why is a laboratory certification useful for the laboratories and the cotton community?

This can easily be answered with a counter question: How do you know and how can you be sure that a laboratory produces suitable test results? For estimating the quality of a laboratory,

- You should have suitable information about the given prerequisites like the testing instrument and its condition, the laboratory conditioning, the calibration, daily checks, the maintenance, the testing procedure, the quality management etc. – and understand if these prerequisites are appropriate.
- You should have a close direct look at a laboratory with on-site visits.
- You should see the test results / performance of the laboratory in comparison to results from other laboratories that you trust.

- You should take the chance to look at the daily performance with e.g. looking at the daily documentation or by checking test results on actually given samples.

The ICA Bremen certification conducts these activities, on a high level of professional competence, on a neutral and independent basis and with objective criteria. With this, the high level of laboratory performance can be proven objectively. Besides high volume instruments, later on other instruments may be included.

Certainly it is of greatest importance to understand the level of the certification. A certification could be done on a basic level, allowing all seriously interested laboratories to pass. The ICA Bremen Certification is instead aimed at a high level, which cannot be achieved by many typical laboratories, and which is suitable to e.g. avoid or resolve quality disputes.

CRITERIA USED FOR CERTIFYING A LABORATORY

The certification procedure was explained by Kai Hughes in the previous presentation. This presentation does only refer to the laboratory assessment criteria.

Generally the assessment includes:

- Evaluation based on answers to a questionnaire and according documentations
- Evaluation based on round trial results
- On-site inspection
- Subsequent checks
 - Triennial inspection
 - Inspections after major changes/complaints
 - Verification samples
 - Continuous monitoring of Round Trial results
 - Arbitrary checks of lab documentations

The evaluation is based on the requirements and recommendations in the CSITC Guideline for Commercial Standardization of Instrument Testing of Cotton as well as on criteria developed by USDA-AMS. The criteria for recognition will be adapted over time in order to assure a continuous high level of laboratory performance. It is of central importance to have objective criteria, which allow evaluating the performance of the laboratories as free as possible from subjective impressions.

The criteria for certifying a laboratory are given in eight separate modules:

1. Laboratory specification / conditioning
2. Instrument and maintenance
3. Calibration and internal verification
4. Testing procedure and samples
5. External verification
6. Quality management
7. Human resources
8. On-site inspection

It is not possible to mention all criteria in this presentation, so only some important keywords are given for an overview.

1 Laboratory specification and conditioning

1.1 Standard atmospheric conditions

Standard atmospheric conditions according to ASTM D 1776 (21°C +/- 1°C and 65% +/-2%) or alternatively ISO 139 have to be kept 24h / 7 days. At any time the conditions exceed the tolerances, testing must cease.

1.2 Monitoring of atmospheric conditions

Temperature and humidity have to be monitored and recorded continuously with independent, externally calibrated checking sensors; at least 2 sensors. Records must be stored.

1.3 Sample conditioning

Samples have to be brought to moisture equilibrium according to a documented procedure. For conditioning, suitable racks have to be used, and any coverings are not permitted. Samples have to be conditioned from the dry side. To ensure minimum conditioning time, the starting time for conditioning has to be recorded.

Rapid conditioning is suitable, but nevertheless the laboratory has to be kept in standard atmospheric conditions.

Cotton calibration and cotton sample moisture content have to be checked periodically to verify the appropriate moisture content.

1.4 Other laboratory specifications, including e.g. electricity and compressed air.

2 Instrument and maintenance

This module includes suitable instruments, periodical instrument service, intense check at the beginning and end of each testing season, documented maintenance according to a given procedure, and re-check for operation after corrective actions.

3 Calibration and internal verification

For calibration, only Universal HVI Calibration Cotton Standards, currently solely delivered by USDA-AMS, Universal HVI Micronaire Calibration Cotton and USDA Colour and Trash Calibration materials are allowed. Expiry dates have to be respected, and overuse has to be avoided.

Calibrations should only be done on an "as needed" basis. On the other side, calibration checks / in-house verifications have to be performed at the beginning of every shift and additionally during the shift in order to ensure the accuracy of the data. Results should be systematically documented and examined for trends. When deviations occur, possible reasons for the deviations have to be identified before calibrating.

When operating multiple high volume instruments in one laboratory, a documented procedure has to be used to ensure that all instruments are operating on the same level.

4 Testing procedure and samples

Unless otherwise defined, each test shall consist of at least 1 micronaire measurement, 2 combs for length/strength, 2 colour readings for Rd and +b, with higher number of measurements or tests for non homogenous cotton. The number of measurements/tests must be clearly specified.

For testing, extreme care should be taken for underweight samples, cut/trimmed samples, significantly non-uniform samples, leafy samples, bad preparation and very low grade samples. Inappropriate sample conditions have to be documented, and the customer should be advised.

5 External verification

The laboratory must maintain participation in the CSITC Round Trials throughout the whole testing season. Laboratories seeking certification must submit the results of the previous two consecutive quarters.

Performance is reviewed for the average score of all properties, and for each single evaluated property.

Currently it is fixed that the combined instrument score, combining all 6 cotton properties, must not exceed 1.2 times the median of all participating laboratories in the single Round Trials. If the performance is unacceptable for two consecutive quarters, or if the average of all scores for one year (after excluding one outlier) exceeds 1.0 times the median of all participating laboratories, the laboratory may be suspended from the certification.

For each single property, the instrument score must not exceed 1.5 times the median of all participating laboratories in each single Round Trial. If the performance is unacceptable for three consecutive quarters, the laboratory may be suspended from the certification.

Laboratories are encouraged to participate in other round trials like the Bremen Cotton Round Trial or the USDA HVI Checktest. Results can also be used in assessing the laboratory's level of performance.

And laboratories are encouraged to participate in re-test programmes, or will be asked to send samples for re-test to ICA Bremen.

6 Quality Management

The laboratory should have an externally evaluated quality assurance programme, e.g. based on ISO, or an internal quality assurance programme. The quality management should cover management requirements and technical requirements.

The laboratory has to care for

- documented procedures for all quality relevant activities as
 - laboratory and sample conditioning and monitoring
 - instrument and equipment service and maintenance
 - instrument calibration and verification, comparison of instruments
 - sample flow and identification
 - testing
 - corrective actions
- defined responsibilities and authorities
- a document control system

After the review, the lab must submit a corrective action plan and a preventive action plan.

7 Human resources

For instrument testing, all quality relevant tasks have to be defined and listed. Each person involved in instrument testing has to be competent to perform the assigned quality relevant tasks on the basis of appropriate education, training, skills and experience.

A documentation needs to be given, which assigns the authorization of each person to each quality relevant task. Only the persons that are authorized for the task may conduct this task.

A key testing competence person is mandatory.

8 On-site inspection

On-site inspections are used to verify the effectiveness and correct implementation of the criteria named above in practice.

THE CSITC ROUND TRIAL – AN IMPORTANT TOOL FOR ESTIMATING LABORATORY PERFORMANCE

The defined aims of the CSITC Round Trial system are:

- A.** Evaluation of the test methods / test result variation: important for instrument testing, but not for single laboratories.
- B.** Evaluation / rating of the participating instruments/laboratories, based on the accuracy of the results. This is the basis for supporting the ICA Bremen Certification.
- C.** Detailed analysis of laboratory results to achieve more accurate results, based on accuracy and precision. This will help laboratories to fulfil ICA Bremen Certification requirements.

The CSITC Round Trial system has been created and started in 2007. The Round Trial system is headed by the International Cotton Advisory Committee (ICAC), and it was developed and is conducted regularly in co-operation between the USDA-AMS

and the Bremen Fiber Institute (FIBRE) [CSITC 2012] [Drieling 2011]. The CSITC Round Trial system cannot fully replace the existing round trials, but adds significantly. A comparison of the existing round trials is given in table 1. The major advantages of each round trial system are underlined.

Table 1. Comparison of international round trial systems on cotton

Attribute	USDA HVI Checktest	Bremen Cotton Round Test	CSITC Global Round Trial
Number of participants	50 to 80 HV instr.	130 to 150 HV instr.	110 reg. labs (2011) 115 to 137 instr. (2011)
Kinds of instruments	Restricted to High Volume	<u>Every kind</u>	Restricted to High Volume
Cottons: Origin and type	USA; Upland	World; broad range of prop.	4 US Upland; 1 international
Costs	Yearly fee	<u>Free of charge</u>	Yearly fee: 1000 USD
Frequency	<u>12 times/year each 2 samples</u>	3 times/year each 1 sample	<u>4 times/year each 5 samples</u>
Number of tests per sample	Asked for 12 tests per sample	Proposed: 6 tests per sample	<u>30 tests per sample: 5 days with each 6 tests</u>
Aim	Information for the laboratory	Information for the laboratory	<u>Official laboratory evaluation and detailed analysis for the laboratory</u>
Evaluation of	Laboratory average	Laboratory average	<u>Laboratory average and all single data</u>
Evaluation of	Accuracy only	Accuracy only	<u>Accuracy and precision</u>

The evaluation of the participating laboratories/instruments is done regarding the trueness of the instrument test results; precision is not taken into account. The procedure for the analysis is easy to follow, and it is useful to understand the evaluation process. Therefore the steps of evaluation for one exemplary instrument are shown in figure 8. The steps are:

- Step 1: The evaluation is done in comparison to the reference results, which were calculated from the inter-laboratory averages.
- Step 2: For each cotton and each parameter, the average result of all tests for all days of this instrument is calculated (average of 30 test results).
- Step 3: For each cotton and each parameter, the distance between the laboratory result and the reference result is calculated.
- Step 4: For each parameter, the average absolute distance of all cottons is calculated.
- Step 5: For each parameter, the mean absolute distance is divided by a “Scale Factor”. This step allows a comparison between the parameters. The scale factors are based on the USDA Reproducibility Limits in 2000. For Rd this result was slightly enlarged regarding the decision of the CSITC Task Force due to the increased variability of these results. The result of this step is a Summary Evaluation for Each Property.

- Step 6: Based on the evaluations for each property, the Combined Summary Evaluation of All Properties is calculated by averaging the results of each property. (Additionally it is possible to apply different relevance factors for each property, but at this stage this is not done.)

Performance of Laboratory 115							
		Micronaire	Strength	Length	Uniformity	Color Rd	Color +b
Reference Values	Cotton 1	3,83	32,82	1,207	82,42	76,31	12,14
	Cotton 2	5,17	28,22	1,136	81,90	78,06	11,53
	Cotton 3	4,40	25,54	0,948	78,53	74,86	10,86
	Cotton 4	3,81	32,89	1,177	83,65	76,08	10,98
Laboratory Average of All Days	Cotton 1	3,80	33,62	1,207	82,71	75,37	11,38
	Cotton 2	5,23	28,50	1,134	81,44	76,05	10,82
	Cotton 3	4,36	26,11	0,969	76,13	73,62	10,41
	Cotton 4	3,79	32,72	1,182	83,83	75,29	10,17
Rel. Distance to Reference	Cotton 1	-0,03	0,80	0,000	0,29	-0,94	-0,76
	Cotton 2	0,06	0,28	-0,003	-0,46	-2,00	-0,71
	Cotton 3	-0,04	0,57	0,021	-2,40	-1,24	-0,45
	Cotton 4	-0,02	-0,18	0,005	0,18	-0,79	-0,81
Mean Absolute Distance to Reference		0,04	0,46	0,007	0,83	1,24	0,68
Scale Factor (Based on USDA Reproducibility Limits except Rd)		0,10	1,50	0,02	1,00	1,50	0,50
Summary Evaluation for Each Property (=Mean Abs. Distance divided by Scale Factor)		0,38	0,31	0,36	0,83	0,83	1,37
Relevance of Property		1,00	1,00	1,00	1,00	1,00	1,00
Summary Evaluation of All Properties (=Average of all properties)		0,68					

Figure 1. Example for the steps of evaluation for a single instrument (#115)

The evaluation is only done for the six reliable instrument parameters: Micronaire, Strength, Length, Length Uniformity, Colour Rd and Colour +b. With 5 cotton samples, and 4 of them taken for evaluation, and 30 tests on 5 days, the round trial results give a reliable overview about the instrument's performance, as needed for laboratory certification.

Figure 2 shows a typical instrument evaluation distribution. Usually 50% of the participating instruments show an evaluation score better than (result lower than) 0.5 units in the evaluation of the combined properties. The current certification criteria means that in a single round trial, an instrument may not be worse than approx. 0.6 units. And for a year, the instrument may not be worse than approx. 0.5 units – so it has to belong to the better half of the instruments throughout the year.

Figure 3 shows four exemplary instruments in different laboratories with their evaluation results over time. It can be seen that laboratory "A" was not operating its instruments properly at the beginning, but learned with the time. Laboratory "B" is principally performing around the median of all instruments, but not consistently suitable. Laboratory "C" is typically slightly better than the median of all instruments. Laboratory "D" is, after an initial phase, showing an outstanding performance, although single outliers still may appear. The rules of the ICA Bremen Certification try to respect this typical behaviour in order to distinguish between suitable and non suitable laboratories.

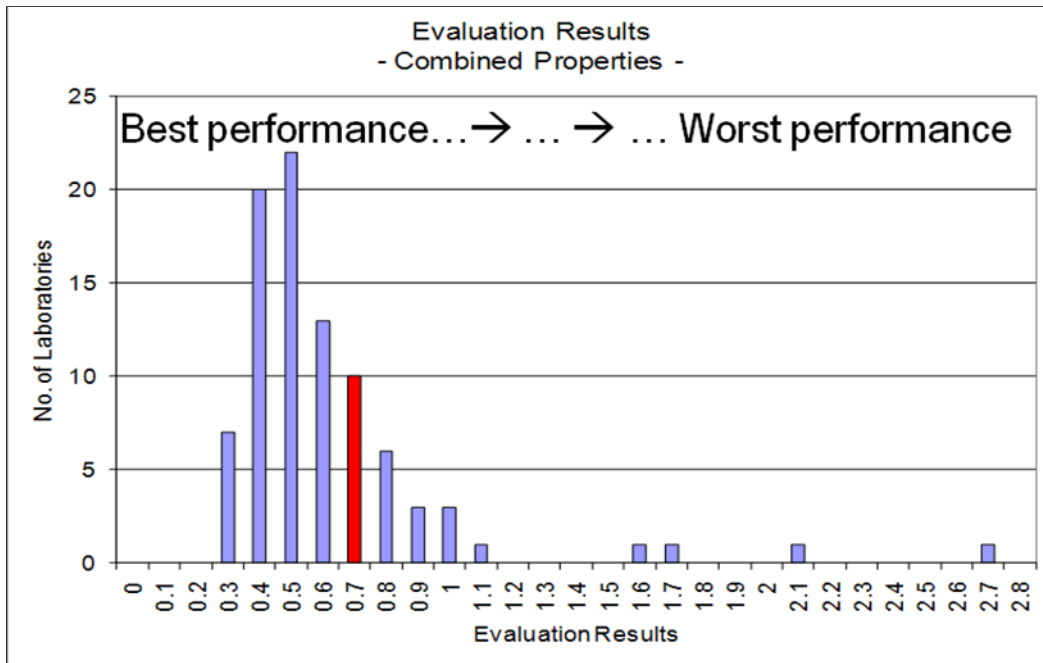


Figure 2. Typical distribution of instrument evaluations in one Round Trial

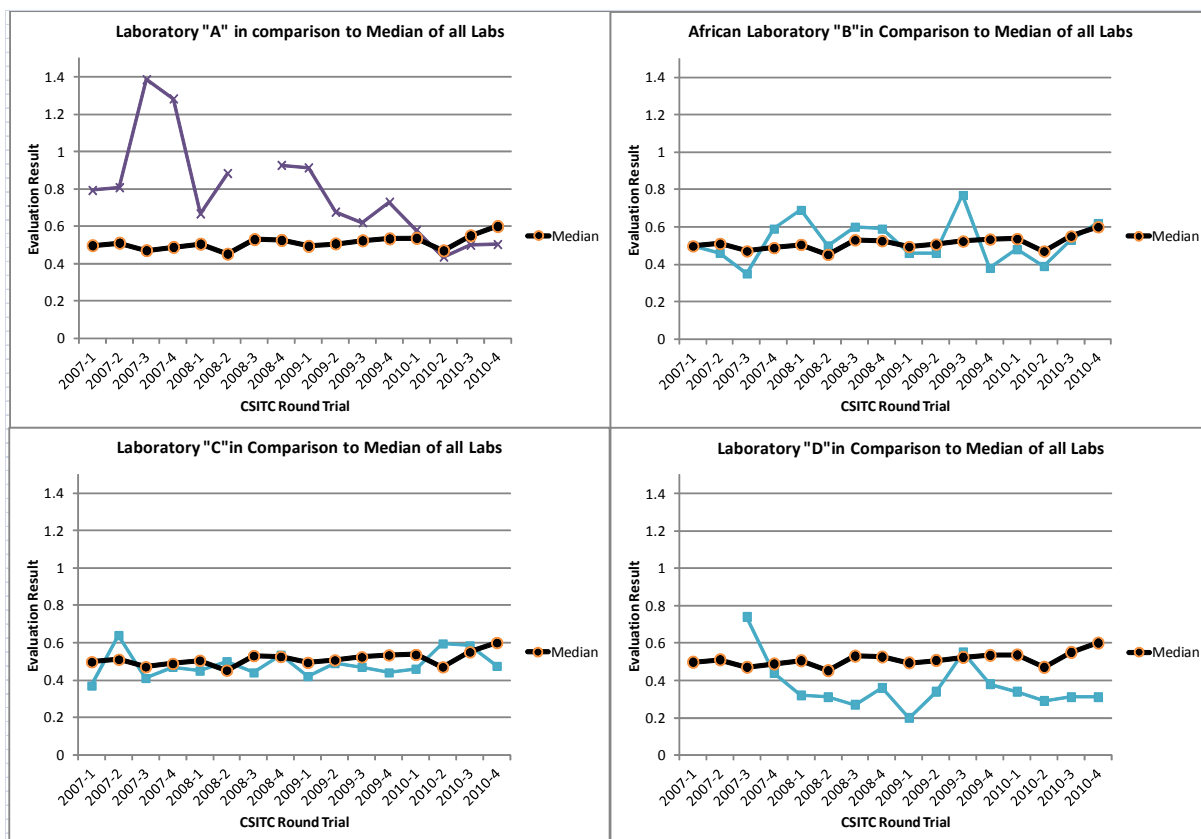


Figure 3. Instrument evaluations of 4 instruments over time

Besides the evaluation of the combined properties, the evaluation of each single property is considered for the certification. As the median evaluation of the single properties is slightly differing from the overall median evaluation, the laboratory result is for the certification compared to the median evaluation of all instruments for each single property. And as the variation of the evaluation for single properties is higher than for the overall evaluation, some less strict rules apply for the single properties.

Table 2. Median evaluations for the six evaluated properties (statistics from 2007-1 to 2011-4)

	Median Evaluations						
	Evaluation Combined Prop.	Evaluation Micronaire	Evaluation Strength	Evaluation Length	Evaluation Uniformity	Evaluation Color Rd	Evaluation Color +b
min	0.45	0.40	0.32	0.32	0.30	0.37	0.39
max	0.60	0.64	0.64	0.52	0.49	0.71	0.74
Average	0.51	0.51	0.47	0.41	0.37	0.50	0.49

Since Round Trial 2011-3, a new "Within Limits" evaluation is calculated in addition to the usual evaluation. In this evaluation it is counted, how many samples or test results of a laboratory are within given commercial limits. This new kind of evaluation is usually seen as being closer to daily commercial practice as the more "abstract" evaluation shown above. So with the time, the analysis of these results might add to or even replace the given instrument evaluation for the ICA Bremen Certification process. An important step for this will be to fix internationally accepted commercial limits for the different cotton properties.

Within Limits Evaluation						
Based on Single Test Results						
	Micronaire	Strength	Length	Uniformity	Color Rd	Color +b
Limits	0.20	2.0	0.030	2.0	1.5	1.0
	units	g/tex	inch	%	units	units
% of Instruments 100% within limits	68.9	34.6	32.4	52.9	43.2	63.6
% of Instruments ≥95% within limits	89.6	57.4	74.3	83.8	60.6	81.8
% of Instruments ≥75% within limits	95.6	78.7	91.2	99.3	81.8	89.4
% of Instruments ≥65% within limits	96.3	86.0	96.3	99.3	85.6	93.9
Percentage of Results Within Limits						
Instrument	Micronaire	Strength	Length	Uniformity	Color Rd	Color +b
GL113-002-03	100	98	100	99	92	100
GL113-002-04	100	98	97	93	98	100
GL113-003-01	100	100	100	99	100	100
GL113-004-01	89	87	68	93	100	83

Figure 4. Within limits evaluation: Statistics for one round trial (above), and result lines for single instruments (below)

SUMMARY

ICA Bremen is introducing a cotton laboratory certification scheme, certifying a high quality level on a neutral and independent basis and with objective criteria. The certification is including evaluations based on given documents, CSITC Round Trial results, on-site inspection and on subsequent checks. The CSITC Round Trial is for this certification an important and suitable tool for evaluating the accuracy of the laboratories' results. Both are valuable measures for supporting the cotton value added chain.

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