Guideline for Standardized Instrument Testing of Cotton

ICAC Task Force on Commercial Standardization of Instrument Testing of Cotton (CSITC)

and

ITMF International Committee on Cotton Testing Methods (ICCTM)



Editors:

- Axel Drieling, Faserinstitut Bremen e.V. (FIBRE) / ICA Bremen, Bremen, Germany
- Jean-Paul Gourlot, CIRAD-LTC, Montpellier, France
- James Knowlton, USDA-AMS, Memphis, TN, USA

Contributors:

- Axel Drieling, Faserinstitut Bremen e.V. (FIBRE) / ICA Bremen, Bremen, Germany
- Jean-Paul Gourlot, CIRAD-LTC, Montpellier, France
- James Knowlton, USDA-AMS, Memphis, TN, USA
- Lawrance Hunter, CSIR and Nelson Mandela Metropolitan University, Port Elizabeth, South Africa
- Philipp Lehne, Faserinstitut Bremen e.V. (FIBRE), Bremen, Germany
- Andrew Macdonald, AMCON Consulting, Sao Paulo, Brazil
- Greg Parle, Auscott, Sydney, Australia
- Mona Qaud, Rieter, Switzerland / ITMF ICCTM HVI Working Group
- Anja Schleth, Uster Technologies Inc., Knoxville, TN, USA
- Ralph Schulzé, Consultant, Narrabri, Australia
- Marinus van der Sluijs, CSIRO, Materials Science and Engineering, Geelong, Australia
- V. Srinivasan, Premier Evolvics, Coimbatore, India

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INTERNATIONAL COTTON ADVISORY COMMITTEE

1629 K Street NW, Suite 702, Washington DC 20006 USA

Telephone +1-202-463-6660 Fax +1-202-463-6950 e-mail: secretariat@icac.org

Task Force on Commercial Standardization of Instrument Testing of Cotton (CSITC)



INTERNATIONAL TEXTILE MANUFACTURERS FEDERATION

Wiedingstrasse 9 CH-8055 Zürich Switzerland

Telephone +41-44-283-6380 Fax +41-44-283-6389 e-mail: secretariat@itmf.org

International Committee on Cotton Testing Methods (ICCTM)



Common Fund for Commodities Stadhouderskade 55 1072 AB Amsterdam The Netherlands

Web: www.common-fund.org

E-mail: managing.director@common-fund.org



European Commission Directorate-General for Development and Co-operation EuropeAid Rue de la Loi 41 B 1049 Bruxelles, Belgium

http://ec.europa.eu/europeaid/index_en.htm

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1. Preamble

The Guideline combines into an operational guide information from the:

- ASTM Standard Test Method
- the ITMF HVI User Guide
- the USDA Guidelines for HVI Testing
- manufacturers' instructions
- as well as the recommendations from the CSITC Task Force and latest knowledge.

2. Introduction

The CSITC Guideline is specifically directed at testing of Upland cotton varieties, which account for over 95% of world cotton production. Nevertheless, this Guideline covers extra fine cotton testing in the calibration and testing sections.

3. Necessary Basic Documents

The following documents shall be referenced by laboratories for testing purposes:

- → The current version of the ASTM D 5867 "Standard Test Methods for Measurement of Physical Properties of Cotton Fibers by High Volume Instruments" (current version: 2012)
- → Manufacturers' instrument manual(s)
- → ASTM D 1776 "Practice for Conditioning and Testing Textiles (current version: 2008)
- → ASTM D7410 "Standard Practice for Qualification of Cotton Classification Instruments for Cotton Marketing" (current version: 2007, reapproved 2012)

All documents shall be maintained in their latest versions.

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4. Definitions

Definitions regarding samples

- Test specimen: the fibers being actually tested in one measurement of the instrument (e.g. one Micronaire plug, one beard)
- Subsample: a defined part of a sample (e.g. a portion)
- Portion (or Side): One half of a bale sample when sampling both sides of a bale. The two portions are combined into one bale sample.
- Bale sample: A sample representing one bale.
- Gin sample: A bale sample taken during the ginning process from the final cotton lint product.
- Control sample: A bale sample taken subsequently to ginning e.g. in the warehouse.
- Other samples: Samples not specifically representing one bale.

Definitions regarding testing

- Measurement: One measurement on one specimen in one module of the instrument (e.g. one Micronaire plug, one beard)
- Test: Combination of measurements on one sample in one or more modules of the instrument for one result (one result line in the instrument report).
- Number of tests: Multiple repeats of tests to arrive at an average result for one sample.

5. CSITC Requirements for Cotton Testing

Currently the test results of the following six characteristics are confirmed by the CSITC Task Force to be sufficiently reliable for commercial purposes

- → Micronaire in units
- → Strength in g/tex
- → Length UHML in mm or decimal inches
- → Uniformity Index UI in %
- → Color Reflectance Rd in units
- → Color Yellowness +b in units

Sampling

- → Mechanical sampling at gin/press
- → Samples of not less than 200 g
- → Identify samples clearly (gin ID, bale number).

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Only calibration with the following calibration material is allowed

- → Universal HVI Calibration Cotton Standards (U-HVI-CCS) for length and strength parameters. For testing Extra Fine varieties¹ the USDA Extra Long Staple Standards shall be used like given in chapter 11.
- → Universal HVI Micronaire Calibration Cotton Standards for Micronaire shall be used.
- → USDA Color and Trash Calibration Materials for Rd / +b and for trash percent area and particle count
- \rightarrow The aforementioned calibration materials are available from USDA-AMS (order at www.ams.usda.gov/cotton \rightarrow Standardization) or from the instrument manufacturers.

Testing shall be done according to ASTM D5867

The CSITC characteristics are defined as named above <u>AND</u> combined with the named calibrations <u>AND</u> combined with testing according to the named standard test method.

Participation in the International CSITC Round Trials is necessary.

6. Sampling

Sampling shall be performed after the bale is formed (or being formed) and can be done either at the gin ("gin samples") or warehouse ("control samples"). Preferably, sampling should be done at the gin.

In order to cover the whole color measurement window, the sample size should be approximately 150 to 300mm long and 150mm wide. The weight should be at least 200g.

Each sample shall be identified with a tag (coupon) placed within the sample (between the portions for a two-sided sample), giving at least the gin or warehouse identification and bale number.

Sample all (i.e. 100%) bales. Alternatively, a sampling plan can be agreed upon between the supplier and the purchaser and applied.

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¹ For this type of cotton, the ICAC wording "extra fine" is used in this guideline. Else it is often referred to as extra long staple or Pima or G. barbadense.

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7. Laboratory Environment

7.1. Electrical

A consistent and reliable power supply is necessary to ensure proper operation and protection of instruments and personnel.

Follow the instrument manufacturers' specifications as published in their technical data sheet.

An uninterruptable power supply (UPS) is required for the testing instrument as specified by the instrument manufacturer.

In the case of power interruptions it is important that testing only be continued if the air conditioning is functional and the actual atmospheric conditions remain within the allowed limits.

7.2. Compressed Air

Follow the instrument manufacturer's specifications as published in their technical data sheet.

In case of having multiple instruments using a common air supply, ensure that each instrument always gets the required pressure and flow, even in case of all operating at the same time.

7.3. Space

Sufficient space shall be available for the instrument, the operator and the samples.

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8. Atmospheric Conditions / Conditioning

8.1. Standard Temperature, Standard Humidity and Monitoring/Recording

As the measured characteristics (mainly strength) are influenced by the cotton moisture content and methodology of conditioning, samples must be brought to a moisture content which is in equilibrium with the approved atmospheric conditions before and during testing.

The relevant ASTM Standard Practice is ASTM D 1776 "Standard Practice for Conditioning and Testing Textiles. For cotton testing".

- \rightarrow The allowed temperature range is fixed at 21 +/- 1°C (70 +/- 2°F)
- \rightarrow The allowed relative humidity range is fixed at 65 +/- 2% RH

The tolerance range around the humidity target (+/-2%RH) is even more important than the target (65%RH) itself, as calibration with cotton standards can compensate for slight variations in the absolute RH level, but cannot compensate for short term variations shorter than the time difference between two calibrations.

The laboratory has to be conditioned to the above conditions 24 hours a day, 7 days a week during the cotton classing season or when testing is on a continuous basis.

If, at any time the conditions exceed the tolerances, instrument testing must cease, and the conditions re-established. Records for the deviations and corrective actions must be maintained.

It is necessary to monitor the temperature and humidity continuously with independent sensors.

Sensors should be periodically calibrated and certified by an external body.

Besides monitoring, the temperature and humidity records must be kept and documented for traceability.

Sensors should be used at least in two locations. The best position for the sensors is close to the instrument as well as close to the samples.

With the acquired temperature and humidity data it is possible to check if the atmospheric conditions were as specified for both the testing and the conditioning of the samples. Sample testing should only be conducted when

- → the climate conditions do not exceed the allowed tolerances
- → and did not exceed the allowed tolerances during conditioning.

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8.2. Building / Laboratory Design

For maintaining the laboratory conditions within the allowed range, it is necessary to optimize the laboratory building. The most important factors affecting the laboratory conditions are the outside heat / radiation and vapor transfer, and their impacts have to be minimized.

In order to avoid rapid changes in atmospheric conditions, the exchange of air with other rooms should be at a minimum. For small labs (less than 150 m²), air locks for every door leading to unconditioned areas are highly recommended. For all laboratories, the doors should close automatically.

8.3. Ambient Air Management System and its Design

For sample conditioning and testing, an integrated Air Management System for simultaneously controlling temperature and humidity (integrated AMS, sometimes called "Heating, Ventilating and Air Conditioning System – HVAC") of the ambient air is required, rather than individual devices for temperature and humidity.

The integrated AMS has to be designed specifically for the laboratory or room to be conditioned in order to achieve constant climatic conditions and to avoid fluctuations. This should be done by an experienced, licensed company.

Any installed conditioning system has to be maintained and serviced at least according to the manufacturer's specification.

8.4. Passive Conditioning of the Samples

According to ASTM D 5867, the only requirement is to bring the laboratory samples to moisture equilibrium for testing in the atmosphere specified for testing textiles. Conditioned cotton samples will have to exhibit moisture content between 6.75 and 8.25% on a dry weight basis for Upland cottons when reaching moisture equilibrium^{2, 3}.

Samples should be conditioned from the dry side. Moist samples requiring preconditioning need to be brought to a relatively low moisture content in a dry atmosphere.

Samples not requiring preconditioning are brought to moisture equilibrium.

Conditioning time must under no circumstances be shorter than 12h [ASTM D 5867]. It is recommended to condition samples for at least 24 to 48 hours [ITMF].

² An immature cotton cannot absorb as much moisture as a mature one.

³ Extra fine / Barbadense cottons typically condition with a slightly lower moisture content.

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After any event during which the conditions exceeded the tolerances and conditions were reestablished, the cotton must reach the conditioned moisture content before instrument testing resumes.

Calibration cottons and test samples must be conditioned in the same conditioning area for a minimum of 72 h to ensure consistent moisture equilibrium.

Samples, including calibration materials, must be stored open in the conditioned laboratory. Conditioning of samples in sacks, wrappers or other coverings is not permissible. The samples have to be placed in single layers. The air needs to be able to penetrate the samples from all sides.

8.5. Rapid or Active Conditioning of the Samples

The same requirements as for passive conditioning are valid for rapid conditioning: to bring the laboratory samples to moisture equilibrium for testing in the appropriate atmosphere for testing textiles (ASTM D 1776).

A Rapid Conditioning System cannot, however, replace laboratory conditioning.

Attention: The use of a rapid conditioner will increase the demands on the laboratory conditioning system capacity. It must be able to source considerably more moisture. The usual loss of moisture in a 24 hour period can now take place within ~ 15 minutes.

When rapid conditioning, air should be forced through the samples for at least 15 min.

The manufacturer's instructions should be followed.

The moisture content of the samples must be checked periodically to verify that the appropriate equilibrium moisture content has been reached. Conditioned cotton samples will have to exhibit moisture content between 6.75 and 8.25% on a dry weight basis for Upland cottons when reaching moisture equilibrium.

8.6. Instrument Correction for Moisture

Any moisture correction must not replace laboratory conditioning and sample conditioning.

At this stage, moisture correction must not be applied to any measured characteristic.

However, if moisture correction is applied, it must be reported with the results that a moisture correction has been applied and that the results are therefore not adhering to CSITC requirements.

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9. Sample Handling in the Laboratory

The laboratory should ensure that any sample can be identified at any time, and that all relevant information can be allocated to the sample.

Deterioration, loss or damage to the test samples during storage, handling and preparation must be avoided and the integrity of the sample must be maintained.

10.Standardized Instruments for Testing of Cotton (SITC)

10.1. General

<u>S</u>tandardized <u>I</u>nstruments for <u>T</u>esting <u>C</u>otton, often referred to as High Volume Instruments or HVI (abbreviation protected by Uster), from here on called "<u>SITC</u>", are able to measure at least the six characteristics recommended by the CSITC Task Force and defined in section 5. The instruments usually consist of the following modules:

- Micronaire Module
- Length/Strength Module
- Color/Trash Module
- plus supporting tools (e.g. balance, fibrosampler)

An instrument must not be used for classification of cotton if it cannot be calibrated within the acceptable manufacturer's tolerance for any fiber property measurements.

The following table shows the instrument test results, format and abbreviations as provided directly from the instrument.

Test Result	Format	Abbreviation		
1.Micronaire	X.XX	Mic		
2.Maturity Index	X.XX	Mat		
3.Upper Half Mean Length	(in) X.XXX (mm) XX.XX	UHML		
4.Uniformity Index	XX.X	UI		
5.Short Fiber Index	XX.X	SFI		
6.Strength	XX.X	Str		
7.Elongation	XX.X	Elg		
8. Reflectance	XX.X	Rd		
9. Yellowness	XX.X	+b		
10. Color Grade	XX-X	C Grade		
11. Trash Count	XXX	Tr Cnt		
12. Trash Area	XX.XX	Tr Area		
13. Trash Grade	XX	Tr ID		

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10.2. Instrument Preparation / Maintenance

Instruments should be thoroughly checked at the beginning and end of each continuous testing period (e.g. season).

Always install and use the latest given manufacturer's software as soon as possible, as the modifications may affect the test results.

For maintenance, follow the instrument manufacturer's procedures as published in their instructions.

Each instrument should be rechecked for operation and accuracy after any corrective action / modification / update has taken place.

10.3. Operation / Testing

Unless otherwise defined, each test (=result line) should consist of at least

- → 1 Micronaire measurement = 1 specimen
- \rightarrow 2 combs for the length/uniformity index/strength measurement = 2 specimens/beards
- \rightarrow 2 color readings for Rd and +b = 2 specimens

For bale samples forming a lot, unless otherwise defined, one test per Upland cotton sample is carried out. In the case of extra fine cotton or roller ginned cotton or of non homogenous cotton, the number of tests or the number of measurements per test shall be doubled.

The instrument should be checked in terms of its condition and functioning at least at the beginning of each testing shift in accordance with the manufacturer's instructions.

The surrounding area has to be checked at the beginning of each testing day.

- → Power supply
- → Compressed air (e.g. sufficient pressure, clean filter, empty water trap)
- → Air management system
- → Atmospheric conditions (current and during conditioning time)

The instrument should be kept "on" 24h / 7 days during the testing period, or else, the instrument must be warmed up for a sufficient period prior to the commencement of calibration and testing.

Tests should be performed according to the manufacturer's instructions.

When starting testing and periodically during testing, the operators must

- → Check the current atmospheric conditions
- → Check the calibration (see section 11)
- → Organize their working space
- → Organize the sample supply

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10.3.1. Micronaire Module

For the bale sample, Micronaire is reported to the nearest 1/100 of a unit.

Any large foreign particles, like large pieces of trash, seeds or large leaf, are to be removed manually from the sample before testing.

Fluff the fibers of the test specimen to eliminate dense clumps of fibers or knotty balls.

10.3.2. Length/Strength Module

For the bale sample,

the Upper Half Mean Length is reported to the nearest 1/100 of an mm or 1/1000 of an inch, the Length Uniformity Index is reported to the nearest 1/10 of a unit,

and the strength is reported to the nearest 1/10 of a gram force per tex unit.

10.3.3. Color/Trash Module

The surface of each subsample should be large enough to cover the instrument measurement area and thick enough to be opaque (no light transmitted through the sample). An uncompressed minimum thickness of 50 mm and a minimum measurement surface area of 100 cm² of each subsample are required.

For a bale sample, Rd and +b are reported to the nearest 1/10 of a unit.

11.Calibration

11.1. Calibration Standards

Only calibration with the following calibration material is allowed:

- →Universal HVI Calibration Cotton Standards (U-HVI-CCS) for length, uniformity index and strength parameters should be used. For testing of all Upland varieties, it is recommended to use the Upland Short/Weak standard combined with the Upland Long/Strong standard. For testing Extra Fine varieties, it is recommended to use the Upland Short/Weak standard combined with the ELS Long/Strong standard.
- → Universal HVI Micronaire Calibration Cotton Standards for Micronaire: One low Micronaire cotton and one high Micronaire cotton (or USDA orifice calibration method). The standards have to cover the entire range of cottons being tested and need to have a Micronaire difference of at least 1.5.
- → USDA Color and Trash Calibration Materials for Rd / +b and for trash percent area and particle count.
- \rightarrow The above mentioned calibration material may be obtained from USDA-AMS (order at www.ams.usda.gov/cotton \rightarrow Standardization).

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Calibration cottons for length and strength have an expiration date that must be observed. Calibration cottons should not be used for calibration after these dates.

Calibration cottons need to be replaced when they have been used very frequently ("overused").

Calibration cottons must be replaced when there is any chance that they have been mixed up.

Calibration cottons must be conditioned within the same laboratory and under the same conditions as the test samples and where they will be tested. The moisture content should be between 6.75 and 8.25% (dry basis) when fully conditioned. The calibration material must be kept in an atmospherically conditioned space at all times.

Color tiles are adapted to the different colorimeter types / light sources (e.g. incandescent, Xenon). The tile set assigned with the SITC should stay with this instrument. Never try to use a tile set other than the one assigned to your instrument, or, if ordering new tiles, strictly choose a tile set adapted to the colorimeter type / light source of your instrument. The type of the color tile is encoded in its serial number (e.g. "X2" for Uster HVI 1000).

Color tiles should be returned to the USDA every 2 years for re-evaluation to ensure accurate colormeter calibration.

11.2. Internal Check Material

Internal standard material can be used for check testing, but not for calibration.

(*Recommendations*) When using internal check material, the instrument should also be frequently checked with Universal Standard Material.

11.3. Calibration / Calibration Check

Calibration contributes to the accuracy of the instrument testing levels by using the internal software to adjust for variations in such things as mechanical, electrical and cotton moisture influences. In fact, the instrument results are adjusted to a specific level of measurement set at an internationally agreed level. Calibration is not a substitute for maintaining the equipment in good operating condition or maintaining properly adjusted and controlled atmospheric conditions.

Calibration in this document means that the instrument parameters are adjusted to come to a specific measurement level. Calibration check means that compliance with the specific measurement level is checked. Typically, the instrument software combines a calibration check with an automatic calibration in the case of out of tolerance deviations from the expected level.

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Calibrations should be performed according to the manufacturer's instructions for each of the fiber property measurements.

Calibrations may be done on an "as needed" basis, given that these detailed check procedures are fully implemented.

Calibration checks must be performed frequently in order to ensure the accuracy of the data.

- → For Micronaire and length/strength, at least at the beginning, middle and end of each shift.
- → For color/trash, the frequency depends on the light system used in the instrument. With incandescent bulbs, the calibration check should be conducted at least every 2 hours. For flash light, the calibration check can be synchronized with the checks for the other instrument modules.

Records of calibration results and of calibration check results must be maintained systematically for each instrument within the laboratory. The results should be examined for trends.

(*Recommendations*) When doing calibration checks <u>on cotton samples</u> independently from calibration, recommendations for tolerances (based on the average of 4 tests) are:

- Micronaire +/- 0.10 units
- Strength +/- 1.5 g/tex
- Length +/- 0.015 inch
- Length Uniformity +/- 1 unit
- Rd +/- 1.0 units
- +b +/- 0.5 units
- Trash area +/- 0.1 %
- Particle count +/- 5 counts

When finding out of tolerance deviations, possible reasons for the deviations must be identified before calibrating.

If the laboratory operates multiple instruments, then a procedure should be adopted which ensures that instruments are operating on the same level based on calibration checks.

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12. Variability of Data / Measurement Uncertainty

Test result data must be sufficiently reproducible for commercial or scientific use.

The following data set has been extracted from CSITC Round Trials 2007-1 to 2013-4 for some 112 US Upland cotton samples and with an average of 106 participating instruments. All the given results are averages for the 112 cotton samples. For the results, 6 tests on 5 consecutive days were conducted with each instrument, hence, in total 30 tests per sample. Outliers according to Grubbs' algorithm were excluded from the calculation.

Within-Instrument Variations (Average of the Median of the within-instrument SD for 112 US Upland cotton samples)							
Characteristic	Mic	Str	UHML	UI	Rd	+b	
Unit		g/tex	inch	%		-	
between different days	0.027	0.40	0.0059	0.29	0.24	0.12	
between single test on one day	0.039	0.58	0.0101	0.51	0.24	0.11	
between 30 tests over 5 days	0.048	0.70	0.0116	0.58	0.35	0.17	

Inter-instrument variations (Average of the inter-instrument SD for 112 US Upland cotton samples)						
Characteristic	Mic	Str	UHML	UI	Rd	+b
Unit		g/tex	inch	%		-
based on 30 tests per instrument	0.071	0.99	0.0118	0.53	1.03	0.36
based on 6 tests per instrument	0.076	1.08	0.0134	0.62	1.05	0.38
based on single tests	0.086	1.24	0.0169	0.81	1.08	0.41

(*Recommendations*) Besides using the variation found in inter-laboratory round trials, it is important for cotton testing laboratories to consider the measurement uncertainty of the test methods based on its basic influences. Only by knowing the influences on the tests and by estimating their significance, is it possible to systematically reduce the measurement uncertainty.

13. Round Trials / Reproducibility Check

Participation in the International CSITC Round Trials is necessary for commercial trading of cotton. The results of the round trials should be used to detect and reduce systematic deviations in the inter-laboratory test result averages.

Where there are more than one instrument in a laboratory, conduct comparisons between the instrument results based on Round Trial results as well as based on specific tests carried out for comparison.

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14.Data Recording / Reporting / Export

The data which is saved on the instrument's hard drive must be copied to a remote and safe place to avoid loss of data.

(Recommendations)

A laboratory test result database, independent of the instrument data storage, is recommended for compiling all the necessary information. The laboratory test result database should be designed to fulfill the requirements for the use of the testing data, such as module averaging or delivery of one result from several to the customer.

15. Commercial Use of the Data

The given variation inside the bales and the measurement uncertainties have to be regarded with appropriate limits in order to ensure proper trading with cotton.

16.Personnel

For instrument testing of cotton, all quality relevant tasks should be defined and listed.

Each person involved in Instrument Testing of Cotton should be competent to perform the assigned quality relevant tasks.

A laboratory representative must be designated and must have the necessary responsibility and authority.

A key testing competent person is mandatory.

Documentation needs to be prepared, which assigns the authorization of each person to each quality relevant task (authorization matrix). Only the persons that are authorized to do a quality relevant task may be assigned to this task / may conduct this task.

17.Laboratory Management

The laboratory management should document and prove how it ensures that all means are available and used before, during and after the performing of the testing of cotton samples and the corresponding reporting in accordance with the quality expected by its customer.

Suitable sample identification, combined with the corresponding documentation of all test related information, should be given, so that tracing of all information is possible.

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18.Additional Topics to be Included in Later Versions

19. Acknowledgements

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