# 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)



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# 1. Preamble

Standardized high volume instrument testing of cotton is carried out widely today and is becoming more and more the basis for cotton trading instead of manual classing. The aim of the ICAC Task Force on Commercial Standardization of Instrument Testing of Cotton (<u>CSITC Task Force</u>) is to facilitate instrument testing for commercial use. For this, it is important to obtain reliable and comparable test results from all involved laboratories worldwide.

The findings of the 6<sup>th</sup> Breakout Session – Best Practices in Instrument Testing – of the 68<sup>th</sup> International Cotton Advisory Committee (ICAC) Plenary Meeting in Cape Town, South Africa, in 2009 confirmed the need for designing a universally acceptable and comprehensive manual covering best practices for commercial instrument testing of cotton fibers from sampling to data reporting.

The CSITC Task Force and the International Textile Manufacturers Federation (ITMF) International Committee on Cotton Testing Methods (<u>ITMF-ICCTM</u>) agreed to jointly work on this important topic, together with representatives from the United States Department of Agriculture, Agricultural Marketing Service (USDA-AMS) and the instrument manufacturers. The CFC/ICAC/33 project, funded by the Common Fund for Commodities and the European Commission, served as a framework to develop this Guideline and to acquire some of the relevant knowledge.

The Guideline combines into an operational guide information from:

- The ASTM Standard Test Methods
- The ITMF HVI User Guide
- The USDA Guidelines for HVI Testing
- Manufacturers' instructions
- The recommendations from the CSITC Task Force and the ITMF International Committee on Cotton Testing Methods
- And up-to-date knowledge.

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# 2. Introduction

For the production, trading and processing of cotton, including the prediction of its processing performance and product quality, it is important to know the quality of the fibers. Instrument testing offers the opportunity to rapidly measure the most important characteristics of each single cotton bale, and many countries include the test results in commercial cotton trading. As cotton is traded worldwide, test results need to be obtained and expressed in the same standardized way and on the same result level, no matter where in the world the tests are carried out.

After sampling in a standard way, samples should be tested in a standardized way, which includes the following steps:

- Standardization utilizing approved physical calibration standards and standardized calibration and test procedures
- Verification utilizing approved methods to validate testing levels
  - Inter-laboratory Round Trials
  - Instrument qualification (ASTM D7410)
  - Within-laboratory verification

For the CSITC purpose, standardized instrument testing may be defined as:

- Testing, according to a standardized method (ASTM D5867) and on a common scale, for any one or more of the following characteristics as defined in ASTM D5867 and currently recommended by the CSITC Task Force:
  - Micronaire
  - o Strength
  - o Upper Half Mean Length, Length Uniformity
  - Color Reflectance (Rd) and Yellowness (+b)
- Calibration with Universal Standard Materials as currently provided by USDA
- Comparison and verification of instruments in CSITC Round Trials, which may be accompanied by re-tests in an independent laboratory

Definition is not confined to a specific instrument manufacturer, model or technology, and is not dependent on the speed of testing of the instrument.

The testing instruments usually measure other characteristics in addition to the above mentioned CSITC parameters. ASTM D 5867 also additionally includes Trash Area and Trash Particle Count and Elongation. Besides these, instruments may also measure or derive other characteristics, such as Short Fiber Index, Maturity, Color Grade, Trash Grade and CSP.

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.

Any process output or effect can be defined as a function of its various inputs, which might be categorized for testing of cotton samples as:

- Test material (see sections: Sampling, Conditioning, Sample Handling)
- Environment (see sections: Laboratory Environment, Atmospheric Conditions, Conditioning)

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- Test method (see sections: Calibration, Testing)
- Instrument (see sections: Testing Instruments, Instrument Service, Maintenance)
- Personnel (see section: Personnel)
- Management (see sections: Laboratory Management, Sample Handling, Data Recording)

The objective of this Guideline is to cover all inputs in order to assist cotton testing laboratories in obtaining accurate test results, with testing costs only a secondary focus. The various inputs will be detailed in the sections below.

As the topic is very complex and at the same time laboratories need an easily understandable guide, each topic in the text is divided up into:

- Explanations
  → in order to understand the subject
- Requirements
  → that must be met
- Recommendations →to improve testing reliability
- More information
  → for a deeper understanding

(marked in a box)

(marked as "Recommendations")

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# **3.** Necessary Basic Documents

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

 $\rightarrow$  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)

 $\rightarrow$  Manufacturers' instrument manual(s)

→ ASTM D 1776 "Practice for Conditioning and Testing Textiles (current version: 2016)

→ ASTM D7410 "Standard Practice for Qualification of Cotton Classification Instruments for Cotton Marketing" (current version: 2007, reapproved 2012)

(*Recommendations*): Besides the above, it is recommended that there is access to the latest versions of the following:

- ISO/IEC 17025 "General Requirements for the Competence of Testing and Calibration Laboratories" (version 2005)
- USDA Guidelines for HVI Testing (based on version June 2005)
- ISO 139 "Textiles Standard Atmospheres for Conditioning and Testing" (version 2005 + Amd. 1: 2011)
- "The Classification of Cotton" USDA AMS Agricultural Handbook 566 / Cotton Incorporated 2013 (available at <u>http://www.cottoninc.com/fiber/quality/Classification-Of-Cotton/Classing-booklet.pdf</u>)

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.

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# 5. CSITC Requirements for Cotton Testing

The objective of the CSITC Task Force is to facilitate instrument testing for commercial use by creating confidence in instrument testing results. This is mainly achieved by agreeing on the various requirements in a completely transparent process.

The following requirements have been specified by the CSITC Task Force.

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

→ Micronaire

 $\rightarrow$  Strength in g/tex

 $\rightarrow$  Length UHML in mm or decimal inches

 $\rightarrow$  Uniformity Index UI in %

 $\rightarrow$  Color Reflectance Rd

 $\rightarrow$  Color Yellowness +b

#### Sampling

 $\rightarrow$  Mechanical sampling at gin/press

 $\rightarrow$  Samples of not less than 200 g

 $\rightarrow$  Identify samples clearly (gin ID, bale number).

(Recommendations) Aim to achieve 100% sampling of all bales.

Additionally, the origin could be mentioned on the label.

Only calibration with the following calibration material is allowed

 $\rightarrow$  Universal HVI Calibration Cotton Standards (U-HVI-CCS) for length and strength parameters. For testing Extra Fine varieties<sup>1</sup> the USDA Extra Long Staple Standards shall be used as described in section 11.

 $\rightarrow$  Universal HVI Micronaire Calibration Cotton Standards for Micronaire shall be used.

 $\rightarrow$  USDA Color and Trash Calibration Materials for Rd / +b and for trash percent area and particle count

→ The aforementioned calibration materials are available from USDA-AMS (order at <u>www.ams.usda.gov/cotton</u> → Standardization) or from the instrument manufacturers.

Only for specific instrument types and customers, alternatively 2 USDA Calibration Orifices and USDA Chamber Calibration Cottons can be used for Micronaire calibration, strictly following the relevant procedure. Instrument's setup 4.0 orifice must not be used for this purpose (contact USDA-AMS for more information).

Testing shall be done according to ASTM D5867

<sup>&</sup>lt;sup>1</sup> 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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(*Recommendation*) ISO 17025 offers an appropriate framework for assuring suitable testing conditions and laboratory management. Laboratories are encouraged to acquire ISO 17025 accreditation or at least to follow its technical requirements.

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.

Adhering to the given CSITC requirements and assessing the accuracy in the CSITC Round Trials will ensure test results at the CSITC recognized level.

More information can be obtained from the CSITC Task Force Reports. Information is also given on csitc.org or icac.org. More details about each topic are given in the specific section which follows.

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# 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.

#### (*Recommendations*)

- Sampling to be done mechanically (mechanical bale press knives "cookie cutters" or warehouse mechanical saws)
- Sampling to be done at the stage when the bale is formed (or being formed) in the gin
- Draw samples from both sides of each bale for forming a "two portion sample" per bale.
- Alternatively, take the appropriate number of samples from each bale, to accurately represent the quality of the bale and to meet the allowed trading tolerances.

(*Recommendations*) In the case of control samples, remove 1 or 2 bands from near the center of the bale. Cut the covers to expose the surface of the baled cotton. Knives at the gin bale press may have already made the cut into the bale. If not, mechanical saws may be used at the warehouse to cut into the bale. Reach into the pre-cut hole and insert the fingers into the layers of cotton and draw fibers across the bale in a rolling motion, removing a large flake (layer) of approximately 100g. This should be repeated on the other side of the bale. When sampling, ensure that the outer layer of cotton is firstly removed, as this layer may be dirty.

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

If the seed cotton is consistent within a seed cotton module, then module averaging across multiple bales can be considered.

(<u>Recommendations</u>) Samples should be packed immediately after sampling without any other kind of handling. Packages and samples should be clearly identified by gin, optionally lot reference, and bale numbers. Samples should be wrapped in packages of no more than 100 samples per package. Samples should only be packed in heavy paper, cotton covers or heavy duty plastic. Packing of single samples in plastic bags is not permitted.

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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 manual.

(*Recommendations*) The equipment in the laboratory should be protected by separate circuit breakers.

(*Recommendations*) A separate electrical line to be used which should be free from transient voltage.

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

(*Recommendations*) For the UPS, the minimum requirement is to protect the instrument's computer. With an adequate UPS capacity, the whole machine can be protected. The UPS has to be such that it at least allows the computer/instrument to be shut down safely. At least 10 minutes is considered necessary.

(<u>Recommendations</u>) The UPS should include a "Line Interactive" or "AVR = Automatic Voltage Regulation" for maximum protection against under/reduced voltages (brownouts) and over/excessive voltages (spikes).

Emergency power generators can allow continued work in the laboratory independently from the grid, but a UPS is still required. In the case that testing is to be continued with an emergency power generator, the UPS has to cover the period up to the start of the power generator.

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

The instruments require

- An air pressure within the range specified by the manufacturer
- Clean air by means of a suitable filter
- Dry air by means of a suitable air drier / water trap
- Oil free compressed air
- Sufficient air volume of the compressor
- Sufficiently wide air tubes

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

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For defining the air supply, the number of instruments and a safety margin shall be considered.

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.

- For the instrument, besides the instrument size itself, at least a 70 cm space should be provided on each side to allow for instrument maintenance.
- For the operator, sufficient space has to be provided to move and operate the instrument as well as to handle the samples being tested.
- Space is also required for conditioning the samples. This is considered in the sections dealing with sample conditioning.

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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.

(*Recommendations*) Alternatively, ISO 139 Textiles Standard Atmosphere for Conditioning and Testing can be applied. For testing,

- The allowed standard temperature is fixed at 20°C, with a tolerance of +/-2°C minus the measurement uncertainty of the sensor so in practice a conformity zone of not more than +/-1°C is allowed
- The allowed standard relative humidity is fixed at 65%RH with a tolerance zone of +/- 4%RH minus the measurement uncertainty of the sensor- so in practice a conformity zone of not more than +/- 2%RH is allowed

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.

The monitoring can be done either with an electronic system (logger), or with a mechanical thermo-hygrograph, or by manually recording temperature and humidity periodically. The sensors need to have a sufficient sensitivity and resolution, suitable to detect and record short term fluctuations.

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

(*Recommendations*) An electronic monitoring system is preferred. Measurements should be done at least every 2 minutes.

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

(*Recommendations*) A psychrometer, ventilated by aspiration, or a similar measuring device, can be used for verifying the recorded relative humidity and avoiding systematic deviations.

Whereas ASTM D 1776 does not give any information about the time period of a moving average of the temperature / humidity for approving, ISO 139 defines a period of not longer than one hour for the moving average in order to exclude short term fluctuations.

(<u>Recommendations</u>) For cotton fiber testing, it is useful to apply a moving average to the climate data of each sensor for a maximum period of 5 to 15 minutes. Nevertheless the individual readings should be inspected frequently for any short term fluctuations. The overall aim should be to avoid short term variations, which are responsible for most of the cotton measurement variations, as well as drifts over longer periods.

As the temperature and humidity may vary at different positions in the laboratory, a sufficient number of sensors has to be used to cover all testing relevant zones of a laboratory. At least two sensors have to be used even in small laboratories for covering the samples and the instrument(s). The best position for the sensors is close to the instrument as well as close to the samples.

(*Recommendations*) ISO 139 requires one sensor for at maximum every 50 m<sup>3</sup>. Locations near the middle of the room at heights approx. 1.5 to 2.5 m from the floor are generally desirable.

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

 $\rightarrow$  the climate conditions do not exceed the allowed tolerances

 $\rightarrow$  and did not exceed the allowed tolerances during conditioning.

#### 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.

- The best insulation is obtained by surrounding the conditioned laboratory and conditioning rooms with other rooms, thereby avoiding outside walls. At least there should be no doors to the outside.
- Windows usually do not provide good insulation and allow direct radiation and consequently allow heat to pass through, and should definitely be avoided.
- To reduce heating of the walls, direct solar radiation has to be avoided. This can be achieved by having large awnings on the East and West sides of the building. In locations further from the equator, the laboratory has to be protected from midday sun.

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- Good heat and vapor barriers (insulation) will help in maintaining constant atmospheric conditions in the laboratory. Any investments in insulation will reduce daily energy costs and will stabilize laboratory conditions.
- Insulation should also be provided for the floor and the ceiling.
- The room size / volume influences the required capacity of the air management system and the daily energy costs. For this reason, the room area and height should not be larger than necessary.

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  $150 \text{ m}^2$ ), air locks for every door leading to unconditioned areas are highly recommended. For all laboratories, the doors should close automatically.

(*Recommendations*) A positive air pressure in the laboratory will minimize outside impacts.

For conditioning the samples, a preconditioning room is not essential.

- For relatively moist samples, a preconditioning room might nevertheless be desirable or necessary for conditioning the samples to the dry side without using an oven. For this, the relative humidity of the preconditioning room should be kept at a maximum of 50% RH.
- For samples coming from relatively dry conditions, the preconditioning room, although not essential, can be beneficial. The room should have a relative humidity similar or slightly below the humidity of the testing room.
- With sufficient time for conditioning in the testing room, the required precision of the preconditioning room might be lower, saving costs.

#### 8.3. Ambient Air Management System and its Design

To achieve accurate climatic conditions, the temperature as well as the relative humidity shall be controlled. Since the temperature and relative humidity of the air interact in terms of the absolute moisture content of the air, it is not possible to control temperature and relative humidity independently.

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.

An integrated AMS consists of the following components with an interconnected control:

- Cooling system
- Heating system
- Steam humidifying system
- Drying system (optional)
- Control/regulation system, including sensors and comparator/regulator and command system
- Air flow components
- Air distribution

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For achieving constant conditions, the integrated AMS should have a sufficient capacity to allow sufficient impact of the AMS components and a good homogenization of the air for its control.

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.

The basis for the design includes:

- Historic distribution data of the outside temperature and humidity (or dry bulb and wet bulb temperatures) (for the relevant testing period)
- Typical daily maximum and minimum temperatures (relevant to the testing period)
- Extreme temperature and humidity levels (relevant to the testing period)
- General building design, position of the room(s) to be conditioned
- Room volumes
- Wall construction/insulation: material, thickness and dimensions / insulation of internal walls, external walls, floor and ceiling
- Roof construction/insulation
- Windows, shadings, doors, air locks
- Instruments involved and their power consumption
- Any system using the conditioned air of the room
- Minimum value of fresh air per minute, acceptable max. air speed
- People, lights, other heat sources
- Amount of moisture absorbing material (daily sample weight) and its moisture content

(For more information, see e.g. British Standard 4194: Recommendations on the design requirements and testing of controlled-atmosphere laboratories (withdrawn in 1992) or similar sources).

(<u>Recommendations</u>) In order to maintain constant conditions in the entire testing room it is important to distribute the conditioned air evenly. This can be done, for example, by suitable ventilation ducts with several outlet vents. Additional ventilators may be used. Care must be taken that there are no air drafts disturbing the measurements (e.g. balance), cross-contaminating the samples, or distributing dust.

(*Recommendations*) The total room air exchange rate should be at least 1 air exchange every four minutes.

(*Recommendations*) In addition to maintaining constant atmospheric conditions, adequate fresh air has to be supplied to the rooms.

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

A log book is an indispensable tool to store all relevant maintenance and service related information.

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#### 8.4. Passive Conditioning of the Samples

According to ASTM D 5867, the only requirement is to bring the laboratory samples to <u>moisture equilibrium</u> 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<sup>2, 3</sup>.

Unfortunately different cottons exhibite different moisture content despite their exposure to the same standard atmosphere.

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.

(<u>Recommendations</u>) This can either be done in an oven having a temperature not higher than  $50^{\circ}$ C or in a preconditioning room with a humidity not higher than 50%.

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].

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.

(*Recommendations*) To ensure the minimum conditioning time, the starting time for conditioning should be recorded.

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.

(<u>Recommendations</u>) Forced conditioned air moving across the surfaces of the samples is preferable. Open-wire shelves are preferred; plastic mesh baskets or suitable cardboard trays can be used when stored in mesh wire racks.

(<u>*Recommendations*</u>) When the samples are laid <u>on</u> the packing, more space around the samples has to be allowed for sufficient air penetration.

<sup>&</sup>lt;sup>2</sup> An immature cotton cannot absorb as much moisture as a mature one.

<sup>&</sup>lt;sup>3</sup> Extra fine / *Barbadense* cottons typically condition with a slightly lower moisture content.

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Figures: Storage of samples for conditioning [Uster]

(<u>Recommendations</u>) It is important to undertake regular checks of the moisture content of the cotton samples. For Upland cottons, the moisture content should not exceed the range of 6.75 to 8.25% (dry basis) and should not vary by more than 1 percentage point from that of the Calibration Cottons. Out of range samples should be allowed additional conditioning time. If the range is still not achieved, then the sample should be marked as exceptional.

(<u>*Recommendations*</u>) Moisture content should be measured using the "oven dry" method or moisture meters (like the Strandberg Model 200D or equivalent), calibrated strictly according to the oven dry method.

#### 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).

Rapid or active conditioning of cotton samples is done in laboratories equipped with Rapid Conditioning Units and may replace passive conditioning of the samples.

A Rapid Conditioning System cannot, however, replace appropriate laboratory climatic conditions during testing.

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The principle of Rapid Conditioning Systems is that conditioned air is drawn through the cotton until equilibrium with the surrounding atmosphere is reached. The time of conditioning is usually less than one hour. It depends on:

- The rate of air flow
- Obstructions to the air flow (samples laid on sample wrappers)
- The moisture differential between the current sample moisture and the moisture of the sample at equilibrium
- The direction of conditioning (conditioning from the high moisture content side is much slower than from the low side).

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  $\sim 15$  minutes.

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

Care has to be taken that air penetrates to the inner portion of the samples, too, so that the whole cotton sample reaches equilibrium moisture content.

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.

#### (<u>Recommendations</u>)

- At any time, abnormalities or deviations from normal or specified conditions should be recorded.
- Lots / groups of samples should be kept together.
- The testing conditions, results and storage details should be recorded and stored. This data should be traceable to the physical sample.
- For possible re-tests, samples should be kept for a fixed period.

The identification, with all associated documentation, can best be achieved with a recording form accompanying the lot / group of samples.

(*Recommendations*) For best practice and efficiency, the sample handling should be organized in detail, so that it is followed at all times and known by all the relevant laboratory staff.

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# **10.Standardized Instruments for Testing of Cotton (SITC)**

#### 10.1. General

<u>Standardized</u> Instruments for <u>Testing</u> Cotton, often referred to as High Volume Instruments or HVI (abbreviation protected by Uster), from here on called "<u>SITC</u>"<sup>4</sup>, 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)

The above is not confined to a specific instrument manufacturer or model, and is not dependent on the speed of testing of the instrument.

The recommendations and comments in this guideline are based upon the experience with the following instruments:

- Uster HVI 1000, HVI Spectrum, HVI 900 types
- Premier ART, ART2 and HFT types

This guideline applies to stand-alone instruments, too, as far as they are designed to provide the CSITC Task Force defined characteristics.

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

<sup>&</sup>lt;sup>4</sup> Another suitable abbreviation is e.g. HVCT for High Volume Cotton Tester

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#### **10.2.** Instrument Preparation / Maintenance

Instruments must 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.

(*Recommendations*) Instruments must be serviced at least at the beginning of each testing season or once a year.

(*Recommendations*) Before being taken into service, the equipment, including support tools, should be checked to establish that it meets the laboratory's specifications and complies with the relevant standard specifications.

(<u>Recommendations</u>) The instrument should be qualified at the beginning of each testing season according to ASTM D 7410 "Standard Practice for Qualification of Cotton Classification Instruments for Cotton Marketing". Verification material is available at <u>cotton.standards@usda.gov</u>. / <u>www.ams.usda.gov/cnstandards</u>. Records of the annual verification results must be maintained.

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

(*Recommendations*) Run the maintenance according to an instrument specific maintenance plan and check-list.

(*Recommendations*) A thorough mechanical check is recommended on a regular scheduled basis, particularly for SITCs with high daily testing volumes.

(*Recommendations*) The color/trash module will show deviating results with a scratched color window. This should be checked frequently, putting a white paper on it and looking at the camera image.

(*Recommendations*) Use a log book to record all events that may help in detecting or solving problems.

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

(*Recommendations*) For major corrective actions, relevant requalification procedures (ASTM 7410) should be performed. Records for the corrective actions and the subsequent verification should be maintained.

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# **10.3.** Operation / Testing

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

- $\rightarrow$  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, roller ginned cotton or non homogenous cotton, the number of tests or the number of measurements per test shall be doubled.

(*Recommendations*) The number of measurements per test or the number of tests per sample should enable results of an acceptable accuracy to be achieved in accordance with the internationally recognized tolerances (see section 12).

(*Recommendations*) In order to identify and address outlying results, define and apply rules for repeating tests and for replacing or averaging test results. This might e.g. be lot limits or variation thresholds.

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.

Items to check include the condition of the instrument:

- General
  - State of the instrument (e.g. cleanliness, cotton residues, unusual sound)
  - Trash bin (empty)
  - o Filters
- Length/Strength Module
  - Sampler (e.g. cleanliness, card cloth, homogenous cotton distribution on the comb)
  - Combs (e.g. missing teeth)
  - Brush (e.g. cleanliness, bent bristles)
  - Clamps (e.g. smooth surface, cleanliness)
  - Pressure at the clamps
  - Vacuum at the length/strength module
- Color/Trash Module
  - Color window (e.g. cleanliness, scratches)
  - Plate pressure
  - Light bulb / illumination
  - Micronaire Module
    - o Balance
    - o Cleanliness

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

 $\rightarrow$  Power supply

→ Compressed air (e.g. sufficient pressure, clean filter, empty water trap)

 $\rightarrow$  Air management system

 $\rightarrow$  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

 $\rightarrow$  Check the current atmospheric conditions

- $\rightarrow$  Check the calibration (see section 11)
- $\rightarrow$  Organize their working space
- $\rightarrow$  Organize the sample supply

#### 10.3.1. Micronaire Module

A predetermined mass of raw cotton is placed in the measurement space and compressed. For measuring, a constant air pressure method is used.

Take one specimen from the bale sample and place the specimen into the instrument's micronaire measurement space for testing. For two portion samples, the specimen can be taken from either one portion or can be a combination of equal amounts from each portion.

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

Any large foreign particles such as large pieces of trash, leaf and seeds must be removed manually from the sample before testing.

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

- Recommended sample size, as specified by the instrument manufacturer, should be strictly followed during testing.
- If the bale sample consists of 2 portions, the Micronaire specimen should represent both portions.
- The sample weighing balance should be properly calibrated and maintained according to the specifications of the manufacturer.
- Care must be taken not to lose any of the weighed material.
- The sample density should be as uniform as possible. Do not for example "poke" a finger through the middle of the sample when inserting the sample.
- External air disturbances around the Micronaire module and weighing balance should be strictly avoided.

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# 10.3.2. Length/Strength Module

The length and length uniformity index of cotton fibers in a tapered beard are derived from the measured length distribution of the cotton fibers. Fibers are caught at random along their lengths to form a tapered beard. The tapered beard is scanned from base to tip to generate the fiber length distribution. The breaking tenacity (strength) is measured, based on breaking the tapered beards using 3.2 mm (1/8 inch) clamp spacing.

In the case of two portion bale samples for Upland cottons, take one specimen from each portion of the sample. In the case of extra fine or roller ginned cottons, take two specimens from each portion.

For the bale sample,

the Upper Half Mean Length is reported to the nearest 1/100 of a 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.

- The recommended sample size for the sampler, as specified by the instrument manufacturer, should be strictly followed during testing.
- In semi-automatic specimen preparation
  - The amount of fibers in the beard can be influenced by the pressure on the sample as well as by the number of turns. The specimen preparation technique during testing should be as close as possible to the technique used during calibration and checking. The sample should be placed so that it is evenly spread over the width of the sample drum.
  - Take care that the beard does not show large holes or gaps without fibers.
  - Take care that the amount of fiber in the beard does not vary too much from comb to comb.
  - Clean the card clothing on the sampler periodically.
  - Take care that the card clothing is not damaged.
- Automatic sample preparation
  - Monitor the cleanliness of the card clothing.
- Check the combs frequently to detect any problems like missing teeth.
- Check that the combs are brushed out at every test.
- Monitor the brush in order to avoid previously attached fibers.
- Check the strength clamps routinely for dirt / particles / sticking fibers.

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# 10.3.3. <u>Color/Trash Module</u>

A smooth representative surface of a cotton sample is placed in the color measurement area and pressed flat with a minimum force of 0.6 kg per square centimeter.

In the case of two portion bale samples, perform at least one measurement on each portion of the sample.

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  $100 \text{ cm}^2$  of each subsample are required.

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

For a bale sample, the percent area (trash), given in decimal form, is reported to the nearest 1/100 of a unit, and the particle count to the nearest whole number.

- The recommended sample size, as specified by the instrument manufacturer, should be strictly followed during testing.
- Take care to cover the full window for each measurement. This can be checked by the control monitor, too.
- The sample has to be thick enough to be opaque (no light transmitted through the sample). The thickness of the sample should be uniform.
- Select a smooth surface of the laboratory sample that is judged to be representative for color, avoiding lumps or folds.
- Check the color window frequently for cleanliness and scratches.

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# 11.Calibration

#### **11.1.** Calibration Standards

Only calibration with the following calibration material is allowed:

 $\rightarrow$ 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.

 $\rightarrow$  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.

 $\rightarrow$  USDA Color and Trash Calibration Materials for Rd / +b and for trash percent area and particle count.

→ The above mentioned calibration material may be obtained from USDA-AMS (order at <u>www.ams.usda.gov/cotton</u> → Standardization).

(<u>*Recommendations*</u>) Micronaire Only Calibration Cotton Standards (ICCS), provided by the USDA, offer a choice of 6 cottons in the Micronaire range. They are recommended for Micronaire Calibration checking, but should not be used for calibration.

For testing Upland cottons						
	UHM Length, In.	gth, Uniformity Strength, M Index, % g/tex		Micronaire		
Upland Short Staple	below 1.01	77 – 81	22 - 26	3.6-4.4		
Upland Long Staple	1.13 – 1.22	83 - 90	30 - 35	3.6-4.4		
For testing ELS / Extra fine cottons						
	UHM Length, Uniformity Strength, Micron In. Index, % g/tex					
Upland Short Staple	below 1.01	77 – 81	22 - 26	3.6-4.4		
ELS Long Staple	1.30 +	84 - 90	37+	3.6 – 4.4		

Generally the approximate test values for the calibration cottons are [USDA]: <sup>5</sup>

Calibration Cotton	Micronaire Level
Low Micronaire	approximately Mic 2.6
High Micronaire	approximately Mic 5.5

The standard deviation of the values of the Universal calibration cottons can be requested from the USDA. The following table gives typical examples for the Standard Deviations

<sup>&</sup>lt;sup>5</sup> ELS Short Staple should not be used anymore.

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[ITMF] and are generally representative of all calibration cottons obtained from USDA. The variations for ELS Standards can be significantly higher. This table can help in calculating tolerances / measurement uncertainties.

Examples of Universal HVI Calibration Cottons						
<b>D</b> .	Short-Weak Long-Strong			Strong		
Property	Designated Values	Standard Deviation (SD)	Designated Standard Values Deviation (S			
Micronaire	4.04	0.08	4.32	0.08		
Strength (g/tex)	23.2	0.74	33.9 0.94			
UHM (inch)	0.975	0.012	1.167	0.012		
UI (%)	79.8	0.64	84.0	0.71		

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.

(*Recommendations*) The more the calibration cottons are used, the earlier they must be replaced, independently of their expiration date. An annual replacement should be considered. In the case of non-frequent use, the calibration cottons should nevertheless be replaced after the expiration date or, when no expiration date is given, after no more than 4 years.

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.

(*Recommendations*) The surface of the color tiles must be clean to ensure accurate calibration. An effective procedure for cleaning the tiles is to spray a diluted non-abrasive liquid detergent on the tile surface, followed by wiping with a clean cloth or tissue. Detergents containing bleach, abrasive or other harsh cleaning agents should not be used.

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 appropriate for 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 colorimeter calibration.

(*Recommendations*) Labs should at least have two color tile sets to ensure continuity of testing whenever a tile set becomes unavailable for use.

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USDA additionally offers calibration check cottons for verification of color and trash measurements using actual cotton. For color, a color check box, consisting of 6 or 12 cottons, is available. Color grade boxes include an expiration date due to the natural change in cotton color over time. Care must be taken that the Color Grade Boxes are used within the specified one (1) year of their validity.

For trash, a set of 6 or 12 cotton samples, mounted under glass with established percent area and count values, is available.

# 11.2. Internal Check Material

In addition to the Universal Calibration Standards, there is an option to use an internal check material for verification of testing levels. The advantage of internal checks is the reduced consumption of Calibration Standards and the ability to utilize cottons for check testing that are similar to those that are generally tested.

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

- Select bales of homogenous, even running cotton with low variation of SITC values. Saw ginned cotton is highly recommended. The check cotton must be clean and without any preparation.
- The properties of the bale should be representative of the general type of material that is tested routinely.
- Two bales are actually preferred over one one of relatively long-strong and one of relatively short-weak cotton.
- Establish the mean and standard deviation by testing at least 60 samples with x specimens per sample; the samples being taken throughout the bale. The value of x should be the same as that which will be used for routine check testing.
- These tests should be made at a time when it is known that all systems, including the conditioning, are functioning correctly. It is advisable that the samples be conditioned for at least 48 hours before testing. Take care that during testing, the instrument is regularly checked with Universal Standard Material.
- Compare the obtained standard deviation with the standard deviation of the Universal Calibration Standards. At most, the obtained standard deviation should not exceed the standard deviation of the Universal Calibration Standards by much. With this, the tolerances that are applied for calibration checks with Universal Calibration Standards can be applied for the internal standards as well.

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

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### **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.

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.

For example, in the case of:

- Deviations from the expected level in the calibration check procedure
- Consistent deviations found (e.g. in independent checks or in interlaboratory comparisons)
- Change of the calibration material
- Changes in the instrument mechanical setup
- Repair / corrective maintenance
- Changes in the laboratory environment

Calibration tolerances are instrument type specific. Typical tolerances are given in the table below<sup>6</sup>:

Instrument	Micronaire	Strength, g/tex	UHML inch / mm	UI %
HVI 1000 HVI 900 HVI Spectrum	$\pm 0,1$	± 1,0	± 0,013 / 0.33mm	± 1,0
Premier ART Premier ART 2 Premier HFT	±0,1	±1,0	± 0,013 / 0.33mm	±1,0

- The average of the Micronaire specimens used to calibrate the Micronaire reading must be within +/- 0.1 Micronaire units of the values established for the standards.
- The average of the test results of the specimens tested to calibrate for length, length uniformity index and strength must be within
  - +/- 0.013 inch / 0.33mm UHML
  - +/**-** 1% UI

<sup>&</sup>lt;sup>6</sup> Tolerances can be set in the instrument software. Do not change unless advised to do so by the manufacturer.

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- +/- 1 g/tex Strength
- The colorimeter Rd and +b values must calibrate within +/- 0.4 of the established values for each of the colorimeter tiles.
- Acceptable trashmeter calibration requires calibration within +/- 0.05 percent area of the established trash tile percent area.

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

 $\rightarrow$  For Micronaire and length/strength, at least at the beginning, middle and end of each shift.

 $\rightarrow$  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 (0.4 mm)
- Length Uniformity +/- 1 unit
- Rd +/- 1.0 units
- +b +/- 0.5 units
- Trash area +/- 0.1 %
- Particle count +/- 5 counts

The level of testing can be operator sensitive on semi-automatic instruments, therefore this should be calibrated/checked when the operator changes.

There are at least three possible approaches for calibration checks:

a) Using the manufacturer's software menu for the <u>internal Calibration/Calibration</u> <u>Check routine</u>. This has to be started for each module of the instrument. The routine will involve testing the relevant calibration material, and will detect compliance with the standard level ("pass") or deviations larger than the allowed calibration tolerances ("fail"). Based on the measurements, the system will in the case of deviations calculate a new calibration. With this approach it is easy to conduct the calibration check, but it is dependent upon Universal calibration material and cannot detect small, but consistent deviations.

b) Conducting an <u>independent test in system testing mode</u>. Suitable cotton samples are tested in the usual system testing mode. The user has to compare the results of the tests to the established results of these cotton samples. If the deviation between the tested results and the established results exceed given limits, then the same follow-up activities as for calibration have to be conducted. This approach allows the use of internal check material and enables small but consistent deviations to be detected. Nevertheless, as each step has to be initiated manually, it is only suitable for users with a good background in data interpretation.

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Control charts, in which the test results are plotted, will help to detect constant deviations, trends, or sudden discrepancies.

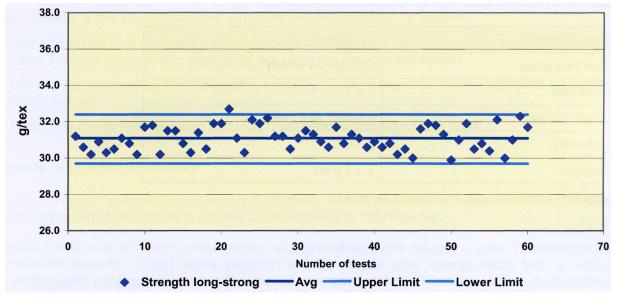


Figure: Control chart [Uster]

When conducting solely independent tests in system testing, the number of measurements per sample should be equal to, or higher than, the number of measurements in calibration mode. With an equal number of measurements, the calibration tolerances can be applied for the test. With a different number of measurements, the tolerances must be adapted accordingly. The number cottons should at least be two, covering the usual range of the properties.

c) <u>Combining approaches a) and b</u>). Besides using the internal Calibration/Calibration Check routine with Universal standard materials, additional independent tests in system testing can be undertaken on the same or other cottons during the day. This intense approach allows combining the advantages of both approaches. In this case, a lower number of tests per sample and only one sample for the independent tests are suitable.

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 CSITC Task Force selected 6 characteristics to be sufficiently reliable for commercial purposes:

- Micronaire (Mic)
- Strength (Str)
- Length (UHML), given in mm or decimal inches
- Uniformity (UI)
- Color Reflectance Rd
- Color Yellowness +b

For these 6 characteristics, suitable data can be obtained from the CSITC Round Trials.

The following data set has been extracted from CSITC Round Trials 2017-1 to 2017-4 for in sum 16 US Upland cotton samples and with an average of 137 participating instruments. All the given results are averages for the 16 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

The within-instrument variations are defined as the Median of the Standard Deviations of all participating instruments on a similar sample:

- Median of the within-instrument variation between different days with 6 tests on each day; this variation includes mainly between-day variability and additionally sample variability.
- Median of the within-instrument variation between 6 tests on the same sample on the same day; this variation includes mainly sample variability and short term fluctuations, but not between-day variability.
- Median of the within-instrument variation between 30 tests on the same sample; this variation includes sample variability as well as short term fluctuations and between-day variability.

Within-Instrument Variations (Average of the Median of the within-instrument SD for 16 US Upland cotton samples)							
Characteristic Mic Str UHML UI Rd +							
Unit		g/tex	inch	%			
between different days	0.024	0.30	0.0053	0.27	0.15	0.09	
between single test on one day	0.035	0.50	0.0098	0.50	0.18	0.09	
between 30 tests over 5 days	0.044	0.58	0.0109	0.56	0.25	0.14	

(*Recommendations*) Each laboratory should compare its within-instrument variation with the averages given here in order to detect influences that reduce the repeatability of its data.

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#### Inter-Instrument Variations

The inter-instrument variations are defined as the Standard Deviations between the results of all participating instruments. This evaluation is done after deleting outliers.

- The inter-instrument variation based on 30 tests. It reflects the systematic deviations between instruments/laboratories.
- The inter-instrument variation based on 6 tests.
- The inter-instrument variation based on single tests. It reflects the actual variation in daily commercial practice, as usually only one test per sample is done.

Inter-instrument variations (Average of the inter-instrument SD for 16 US Upland cotton samples)						
Characteristic Mic Str UHML UI Rd +t						
Unit		g/tex	inch	%		-
based on 30 tests per instrument	0.057	0.71	0.010	0.46	0.52	0.27
based on 6 tests per instrument	0.063	0.82	0.012	0.54	0.55	0.28
based on single tests	0.072	0.96	0.015	0.73	0.60	0.32

The inter-instrument variations can be taken as a basis for fixing commercial trade limits. For this, a litigation risk based on tests on different samples of the same bale, conducted in two different laboratories has to be considered. Additionally, it is important to recognize that the given variations are solely based on US Upland cotton samples. For other origins of the cotton, different variations may exist e.g. based on the variety, production, harvesting or ginning.

(*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 a knowledge and understanding of the various factors which influence the measurements and their values, and their significance. Only by knowing the influences on the tests and by estimating their significance, is it possible to systematically reduce the measurement uncertainty.

#### Preliminary Inter-Instrument Variations on Other Characteristics

For other characteristics, measured with the standardized instruments for testing of cotton, the inter-instrument variability is significantly higher, so that they were not considered by the CSITC Task Force for commercial use. The typical inter-instrument variations for Trash and Short Fibre Index are given in the following table, again based on the CSITC Round Trials 2017-1 to 2017-4 (16 US Upland samples).

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Inter-instrument variations (Average of the inter-instrument SD for 16 US Upland cotton samples)							
Trash Trash							
Characteristic	Count	Area	SFI				
Unit		%	-				
based on 30 tests per instrument	6.4	0.052	1.06				
based on 6 tests per instrument	6.7	0.057	1.09				
based on single tests	7.2	0.065	1.23				

For elongation, the inter-instrument variation can be seen based on results in the ICA Bremen Cotton Round Trials. Based on 6 Round Trials (2016-1 to 2017-3) with in sum 6 samples from different origins and based on an average participation of 95 instruments, the inter-instrument variation for elongation is (based on typically 12 tests per sample)

- SD 0.95%
- CV 15%

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# **13.Round Trials / Reproducibility Check**

Laboratories should participate in regular inter-laboratory Round Trials.

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.

The CSITC Round Trial is the most comprehensive international testing programme offered for standardized instruments for testing of cotton (SITC). It is conducted 4 times a year, each with 4 cotton samples, each sample to be tested 30 times. Information: <u>csitc.org</u>. Registration: <u>csitcsecretariat@icac.org</u>.

(*Recommendations*)

- Compare the evaluation results of the properties for your instrument in order to determine, which modules / measurements have to be improved.
- Analyze the diagnostic graphs for each measurement in order to find possible reasons for deviations and to improve the accuracy.
- Analyze the diagnostic graphs and the precision table for improving the over-time variability of the data.
- Analyze the results of subsequent round trials in order to find trends.
- Compare the result deviations found in CSITC Round Trials with those from other round trial programmes.
- Document the Round Trial results and the relevant follow-up actions.

Besides the CSITC Round Trials, the following Round Trials can be considered for participation:

- The USDA HVI Checktest Programme allows monthly comparisons on each of 2 cotton samples. Contact: <u>cotton.standards@usda.gov</u>.
- The Bremen Cotton Round Test allows participation free of charge, and enables the SITC results to be compared with the SITC results of other laboratories as well as with the results of different cotton testing methods. Contact: <u>drieling@faserinstitut.de</u>.
- Regional Round Trials allow inter-laboratory comparisons using locally grown cottons. Information: <u>csitc.org</u>

Where more than one SITC instrument are operated in a laboratory, each instrument should be checked on the basis of its Round Trial results. In addition, the instruments should be compared on the basis of tests carried out specifically for comparative purposes between the instruments.

(*Recommendation*) Round Trials do not allow a daily check of the accuracy of the instrument results. For the purpose of daily verification, a Reproducibility Check is recommended. For a Reproducibility Check, a representative subset of all daily samples is sent to an independent laboratory, retested utilizing methods that provide a better accuracy/precision, and the results compared.

• USDA AMS is offering a non-periodic Checklot Program, re-testing single samples sent by any laboratory.

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- In some regions, Regional Technical Centers offer a Reproducibility Check program under CSITC control for their surrounding countries.
- Laboratories can assign another independent laboratory for running reproducibility checks, if the assigned laboratory can prove
  - That it fulfills the requirements of this CSITC Guideline
  - $\circ$  and that it provides a better accuracy/precision.

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# 14.Data Recording / Reporting / Export

The data which is saved on the instrument's hard drive must be copied to remote data storage device(s) to avoid loss of data.

## (*Recommendations*)

- A routine for periodic data storage should be developed and applied.
- It is recommended to follow the manufacturer's instruction manual to best transfer the data in the appropriate format to other medias (disk, cable, USB memory sticks, etc.).
- The transfer from the instrument hard drive to the laboratory database can be facilitated by choosing the appropriate export format from the instrument; follow the manufacturer's instruction manual
- On yearly basis, during manufacturer's maintenance visit, it is recommended to clean the instruments hard drive from old data, as long as an external database is used to store the historical data from previous seasons.

# (*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.

The database should be permanently copied to a remote and safe place to avoid loss of data.

A procedure should be in place for continuously copying the data from the instrument's data storage to the database.

For any sample tested, in order to trace back all information, the database should store:

- $\rightarrow$  All information relevant to the sample history
  - Origin
  - Processing gin
  - Customer/provider name
  - Sample type (gin or control)
- → All information relevant to the applied method and / or settings applied for the testing of the samples
  - The name and type of the instrument used
  - The number of tests per samples per module of the instrument
  - The applied method (testing made on portion of samples or representative samples),
  - Technician and operator names
- → All information relevant to the conditions of testing of the samples, such as:
  - Calibration of the machine at the moment of testing of this sample (reference material names, expiry dates, results of the calibration verification
  - Temperature and relative humidity conditions
  - Any remarks

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 $\rightarrow$  All information relevant to the testing of the samples

- Results
- Remarks (for low sample mass or dirty cottons for instance)

Reporting is usually made from the laboratory test result database; it should respect rules given in ISO 17025 as well as the abbreviation and the format as given in Section 10.1 for better understanding between cotton stakeholders.

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# **15.Commercial Use of the Data**

The overall objective of this guide is to achieve accurate and repeatable results on instruments testing at high speed, so that the cotton spinner is able to accurately evaluate the raw material in order to be sure of the correct performance, not only in spinning, but through out the complete cycle of the cotton textile transformation process, including dyeing and finishing.

However there is also a commercial aspect of valuing the cotton in accordance with the characteristics as determined by the instruments, which can assist the seller, the farmer or ginner, and the final consumer, the spinner, to negotiate the price within the context of the overall market value at a given time.

Since a cotton lay down at the spinner's factory is made up of a large number of bales, the single bale test data is used to achieve an average of the mix, whilst still achieving the predetermined distribution of the characteristics or parameters.

On the production side, the cotton being a natural product, it is virtually impossible for each bale to have the same identical characteristics, therefore during the testing process some slight variations will occur from bale to bale. Also, at the spinners laboratories, such slight variations will become apparent, but this should not be considered a defect or inconsistency of the instrument, but rather an acceptable "commercial" tolerance or range of results, which has been agreed upon before hand between the buyer and seller. This commercial use, or "tolerances", of the data is defined in the Trade Rules of the Cotton Associations. However without accurate and repeatable instruments the cotton will fall outside of such variations or tolerances, and therefore prejudice the spinner's quality and the financial return to the seller.

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.

Additionally the cotton properties vary between the bales. This can for example be considered by not trading based on single bale results, but on sales lot averages and allowed variations. Due to the statistical background, sales lot averages and variations with significantly lower tolerances than the single test results can be agreed upon.

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#### 16.Personnel

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

Quality relevant tasks include calibration, testing, checking and signing test reports, maintenance of instruments, procurement etc. The necessary competence for the tasks has to be defined.

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

Competence can be imparted by appropriate education, training, experience and/or demonstrated skills, as required.

(*Recommendations*) It is recommended that the laboratory maintains records of the relevant competence / training of personnel.

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

A key testing competent person is mandatory.

The typical personnel involved in instrument testing is:

- A laboratory head / key testing competence person
- Instrument operators
- Assisting personnel
- Instrument maintenance technician.

#### (*Recommendations*)

It is recommended to not only train internally, but to also provide external competence training at least for the key personnel.

Operators should be trained to work on all the positions / modules of the test instrument and should periodically rotate. They should also be able to perform calibration, handle samples, use correct specimen preparation and testing techniques, and recognize instrument malfunctioning and errors.

For maintaining and improving the know-how it is useful to exchange knowledge with other cotton testing laboratories.

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.

#### (Recommendations)

The laboratory management should ensure that a sufficient number of qualified and authorized personnel are always available to perform the required tasks.

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# **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.

(*Recommendations*) The laboratory should:

- Establish and maintain sample identification from collection to disposal as well as a method to ensure the security and confidentiality of the collected information in a system that stores the original information, derived data and any information to facilitate any research for easy traceability of the information.
- Have defined well trained managerial and technical personnel designated for realizing the required testing analysis in accordance with the quality demanded by the customer.
- Develop and apply procedures for the selection and the purchasing of services and supplies that affect the quality of the tests.
- Have and apply a policy that should be implemented when any aspect of its work or results of its work do not conform to the requirements agreed upon by the customer. This policy should include the overall description for implementing corrective actions and / or preventive measures.

ISO 17025 defines the corresponding requirements.

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# **18.Additional Topics to be Included in Later Versions**

- Other test instruments
- Requirements and rules for module averaging
- Bale tagging recommendations

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#### **19.Acknowledgements**

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