ESTABLISHING HVI CALIBRATION STANDARDS

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Abstract

The main objective of the Cotton Program's production of High Volume Instrument (HVI) calibration standards is to meet the annual demand while maintaining year to year establishment consistency. Each year, over 40,000 pounds of HVI calibration cotton and over 200 sets of HVI color and trash tile calibration standards are produced. Consistency in value establishment of all HVI calibration standards is achieved by maintaining links to reliable reference standards. Through careful planning and execution of detailed procedures, the Cotton Program's Standardization and Quality Assurance Branch supplies practically all HVI users throughout the world with accurate HVI calibration standards.

Introduction

High Volume Instruments (HVI) serve the cotton industry as the primary means for obtaining accurate cotton fiber quality information. In order to ensure accurate and repeatable HVI measurements of cotton quality, calibration to established HVI standards is necessary. The USDA Cotton Program has been producing calibration standards specifically for the HVI since the early 1980's when the utilization of HVI's started to increase. Calibration standards for the various HVI measurements were not developed at the same time. Since HVI development involved the integration of various types of individual instruments, some of the measurement standards existed before the HVI. For example, calibration standards for the stand-alone colorimeter and micronaire testers were well established prior to these instruments being integrated into the HVI. Today, HVI calibration standards are produced by the USDA Cotton Program for HVI calibration of micronaire, strength, length, length uniformity index, color reflectance (Rd) and yellowness(+b) and trash (percent area and count). These HVI standards are recognized worldwide as official standards for HVI calibration.

Production of HVI Standards

The Standardization and Quality Assurance (S & QA) Branch, of the USDA, Agricultural Marketing Service, Cotton Program, is responsible for the production of the HVI calibration standards. Ninety percent or more of the HVI standards produced are used by Cotton Program classing offices. The remaining ten percent of HVI standards are used by foreign and domestic HVI users.

Annual consumption of "Universal HVI Calibration Cotton" (used for strength, length and length uniformity index calibration) has been stable at just over 30,000 pounds per year for the past two years. Usage per HVI has decreased over the years with improvements made in HVI measurement accuracy and stability. Improved and refined calibration routines have also contributed to lower usage. Annual consumption of micronaire calibration cottons has averaged about 10,000 pounds per year. Production of new color and trash calibration tiles is typically required only for equipping new HVI's and replacing broken tiles. The number of color and trash tile sets produced last year was just over 200. An annual Cotton Program reviewing procedure requires classing offices and the Quality Assurance Unit to return all HVI color and trash tile sets (approximately 250 sets) to the Standardization and Quality Assurance Branch for inspection, cleaning and reestablishment of values.

Concepts Utilized in the Value Establishment of HVI Standards

Any reliable calibration standard must be traceable to a reference that is as absolute as possible. Calibration standards for fundamental measurements such as length, mass, temperature and time should be traceable to absolute standards maintained by standards organizations such as the National Institute of Standards and Technology (N.I.S.T.). Traceability is defined as the ability to trace the accuracy of a standard back to its ultimate source (Doebelin, 1990). Applying the concept of traceability to HVI calibration standards requires that each and every established value be traceable to a single reference standard.

All HVI calibration standards that are produced by the USDA Cotton Program are traceable to HVI reference standards that are maintained by the Standardization and Quality Assurance Branch in Memphis, Tennessee. Thoroughly tested cottons are held in long-term storage to serve as reference standards for all HVI calibration standards. The only HVI calibration reference standard that is not cotton is the HVI color reference which is maintained by master sets of color tiles. Given the importance of the HVI reference standards, ensuring their safe and secure storage is critical. Backups of all the HVI reference standards are securely stored in a classing office away from Memphis in the unlikely event that the reference standards in Memphis are destroyed.

Traceability ends at the physical HVI reference standards. The original measurement levels of these standards were established by classical or other appropriate methods that provided the closest estimate of an absolute measurement. Since those times, the HVI reference standards have stood

on their own as absolute standards. A more absolute reference method for any HVI measurement has yet to be developed.

Establishing Universal HVI Calibration Standards

HVI measurements for strength, length, and length uniformity index are calibrated to two "Universal HVI Calibration Cotton Standards". One of these calibration cottons is short and weak and the other is long and strong. Universal HVI Calibration Cotton Standards are produced for HVI calibration for both Upland and Pima cotton testing. In 1995, McNabb reported on the value establishment procedure for HVI Calibration Cottons. The procedure has changed considerably since that time, given such things as the gradual phase-out of the MCI HVI and improvements in methology and instrumentation.

The value establishment procedure begins during the classing season by studying HVI classification data to identify cotton bales with HVI measurement properties ideal for candidate calibration cottons. All candidate bales must have a color grade of Middling Light Spotted (32) or better, a leaf grade of 3 or better and a micronaire close to 4.0. Candidate bales for short-weak calibration cotton must have an upper-half-mean length between 0.94 to 0.99 inches, length uniformity index between 77 and 80 percent, and a strength between 20 to 25 grams per tex. Candidate bales for long-strong calibration cotton must have an upper-half-mean length between 1.14 to 1.18 inches, length uniformity index between 82 and 85 percent, and a strength between 30 to 34 grams per tex. In most cases, candidate bales are obtained by purchasing an entire module of bales (10 to 20 bales).

Candidate bales are purchased and shipped to the Standardization and Quality Assurance Branch in Memphis, Tennessee where they are held in non-air conditioned warehouse storage for a year before values are established. The storage period is used as a precaution to ensure that the fiber properties have stabilized prior to value establishment.

Value establishment studies involve making up sample sets each containing eighteen candidate bales. In addition, each sample set contains samples from two "Ringer" bales and two "Benchmark" bales. The two ringers are a short-weak bale and a long-strong bale. Ringers provide a basis for monitoring continuity from study to study. The two benchmarks are a short-weak bale and a long-strong bale. Benchmarks serve as the reference for value establishment on the candidate bales.

The first step involves obtaining representative samples from each bale required in the value establishment study. In order to properly sample a bale, half of the bale bands are removed and ten samples are taken from across the bale fanhead. Given that ten samples are taken from each of

eighteen candidate bales, two ringer bales and two benchmark bales, the total number of samples in a study set is 220.

Currently, there are five HVI laboratories that have been designated for value establishment. These laboratories are the Memphis, Lubbock and Macon classing offices, Quality Assurance in Memphis and Cotton Incorporated in Raleigh, North Carolina. For each value establishment study, a complete sample set is made up for each of the designated laboratories. Each laboratory then tests the sample sets on two HVI systems.

Each designated laboratory conditions their sample sets in the standard atmosphere (65% RH, 70° F) for 72 hours prior to testing. The HVI's are calibrated to the benchmark cottons and the samples are tested once a day for two days. Since 1997, values have been established on Zellweger Uster 900 Automatic HVI's.

Testing results are sent to Memphis for analysis. All data are averaged by HVI and by day. The measured values for the benchmark bales are compared to their established values. All data from an individual HVI is rejected if that HVI's measured values on the benchmark bales are not within set tolerances of the established benchmark values. The within tolerance differences are used as the basis for adjusting the obtained values of the candidate and ringer bales to the established levels of the benchmark bales. All adjusted values are then averaged. These averages are then used as the established values for the strength, length and length uniformity index for the candidate bales.

Candidate bales are rejected if test results indicate unacceptable variability within the bale. Bale rejection is based on bale measurement standard deviations that exceed established tolerances. These tolerances are 0.012 inches for length, 1.0 for length uniformity index, 1.25 g/tex for strength above 30 g/tex and 1.00 g/tex for strength less than 30 g/tex.

Maintaining the Reference for Universal HVI Calibration Cotton

Maintaining a reference for Universal HVI Calibration Cotton is complicated by the destructive nature of the HVI measurements on cotton during testing and calibration. Supplies of HVI calibration cottons must continually be replenished on active HVI's. As a result, value establishment studies must be continually performed to meet the demand for calibration cottons. Since value establishment studies require benchmark cottons to serve as the reference for all assigned values, bales of benchmark cottons also require replenishing. Two benchmark bales, (one short-weak and one long-strong) on average, will provide enough cotton for six value establishment studies. When they are depleted, another set of benchmark bales is selected. On average, two to three sets of benchmark bales

are depleted each year. Maintaining long-term value consistency for each new set of benchmark bales requires a special value establishment procedure.

Benchmark cotton bales are selected from previously approved calibration cotton bales that are above average in measurement uniformity. Benchmark bales are selected in groups of at least ten bales that were ginned in sequence and were grown by the same producer. Calibration bales selected to be benchmarks already have established values. However, the practice of utilizing these values as benchmark values would result in a propagation of error in the reference over time. In other words, a lack of an absolute reference through time would lead to drifting measurement levels. Therefore, benchmark bales are referenced to a special set of bales known as "HVI Reference" bales. In practice, whenever more benchmark bales are needed, a special value establishment study is conducted using the HVI reference bales as the benchmark for establishing benchmark values on the candidate benchmark bales. Currently, there are five short-weak and five long-strong HVI reference bales.

The value establishment program for Universal HVI Calibration Cottons references calibration cottons to benchmark cottons and benchmark cottons to HVI reference cottons. Under this program, a supply of accurate and consistent calibration cottons should be provided for more than forty years based on present HVI calibration cotton consumption. The system would not have to come to an end even beyond this time. Two of the HVI reference bales (a short-weak and a long-strong) are designated as the "stake" bales. If ever necessary, in theory the stake bales could be used to produce enough reference bales to continue HVI calibration cotton establishment for hundreds of years.

Establishing HVI Micronaire Calibration Cotton Standards

Two micronaire calibration cottons are required for calibrating the HVI micronaire measurement. One cotton has a low micronaire (3.5 or less) and the other has a high micronaire (5.0 or higher). There are a total of six micronaire calibration standards available ranging from 2.6 to 5.5.

The established operational framework by which the HVI micronaire calibration standards are provided is governed by the International Calibration Cotton Standards Committee (ICCS Program, 1998). This committee is made up of foreign and domestic members representing six sponsoring organizations. The concept for establishing values on micronaire calibration cottons is similar to that of the Universal HVI Calibration Cotton Standards. The laboratories that provide value establishment are designated by the sponsoring organizations. There are currently five designated laboratories.

As with the Universal HVI Calibration Cottons, reference bales for micronaire standards are required for sustained and traceable value establishment. Currently there are three high micronaire reference bales and three low micronaire reference bales. Two of these bales are designated as "Micronaire Stake" bales to be used for establishing more reference bales if ever needed. Unlike establishment of Universal HVI Calibration Cotton Standards, micronaire value establishment consumes a relatively small amount of candidate cotton. Therefore, the current reference standards should be adequate for many years.

Establishing HVI Color Calibration Standards

Calibration of practically all HVI colorimeters is achieved with the use of a set of five color tiles. These tiles are referred to as the brown, yellow, gray, white and central tiles. All color tile values are established on a master colorimeter maintained by the Standardization and Quality Assurance Branch in Memphis. The master colorimeter instrument utilizes the same colorimeter as late model Zellweger Uster HVI's. Four master sets of color tiles serve as the reference for all HVI color calibration standards. One of the master sets is designated as the "working set" while the other sets are designated as backup sets. The working set is used for daily calibration of the master colorimeter. The backup sets have two purposes. One is to replace the working set in the event that one or more tiles became unusable due to breakage or some other reason. The second purpose of the backup sets is to provide a routine check of the level stability of the working set. Once a month, color readings are obtained from the master colorimeter on the backup sets. The color readings on the backup sets are compared to their originally established values to verify stability in the working set. In addition to the master color tile sets, a working check set is measured on the master colorimeter immediately following calibration to the working set. The purpose for the daily reading of the check set is to verify that the working set is holding the reference level on a day to day basis.

The color tile establishment process begins by thoroughly washing the tiles with mild soap and water. The tiles are then inspected for defects and allowed to dry overnight. The next day the tiles are measured on the master colorimeter. Four Rd and +b color measurements are made on each tile. After each measurement, the tile is turned ninety degrees. The differences between the four Rd readings and between the four +b readings must be 0.4 or less or the tile is discarded for lack of color uniformity. If the tile differences are within the tolerance, then the average of the four measurements becomes the established value for the tile.

Establishing HVI Trash Calibration Standards

Traditional calibration of the HVI trashmeter was achieved primarily by the use of a trash tile. However, as the HVI

trashmeter evolved, the necessity for more calibration materials was realized (Knowlton, 1997). Today, the primary HVI trashmeter calibration materials are the trash tile and a set of six cottons, varying in the amount of trash, mounted under glass. As trashmeter technology continued to change, it was realized that a tile calibration standard was not adequate for maintaining a constant cotton trash measurement level from year to year (Randle, 1992). In other words, the relationship between trash tile measurements and actual cotton trash measurements was different for different trashmeters. As a result, sets of cottons mounted under glass were developed to serve as the reference for the HVI trash measurements of percent area and particle count. In order to ensure proper calibration of the HVI trashmeter, a set of cottons-under-glass is measured on the instrument and the readings must be within the tolerances for the established values. No other HVI trash reference material investigated has more closely mimicked the measurement characteristics of actual cotton.

All HVI trash measurement tile standards and cottons-under-glass standards are established on a late model Zellweger Uster HVI trashmeter that serves as a master instrument. The master trashmeter is calibrated to the level of a master set of twelve cottons-under-glass.

Conclusion

Maintaining cotton fiber measurement consistency from year to year is critical for accurate marketing and proper utilization of cotton. Achieving this goal requires continued establishment of accurate HVI calibration standards. A continuing objective of the Cotton Program's Standardization and Quality Assurance Branch is to continue to supply accurate HVI calibration standards for meeting the calibration demands of HVI testing in the United States and throughout the world.

References

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