

## IMPROVED SPECTROPHOTOMETER FIBER SAMPLING SYSTEM FOR COTTON FIBER COLOR MEASUREMENTS

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### Abstract

Cotton in the U.S. is classified for color using the Uster® High Volume Instrument (HVI), and the parameters Rd and +b are used to designate color grades for cotton fiber. However, Rd and +b are cotton-specific color parameters, and the need existed to demonstrate the relationships of Rd and +b to  $L^*a^*b^*$  if “standards” are to be developed for cotton color measurements. The relationships of Rd and +b from the HVI to the globally recognized  $L^*a^*b^*$  color system from color spectrophotometers was verified and validated previously. A program was implemented to 1) determine the impact of applied pressure on spectrophotometer fiber measurements and 2) to develop an improved, pressurized fiber sampling system that can precisely measure large cotton fiber samples (e.g., standard AMS bricks). The pressure impact results on small fiber “fluff” samples indicated that applied pressure impacted primarily fiber  $L^*$ . The optimum applied pressure for small “fluff” fiber color measurements was 30 psig (pounds per square inch gauge). For large fiber samples (e.g., Agricultural Marketing Service/AMS cotton bricks), a prototype sampling system was developed. Protocols for pressurized fiber measurements on spectrophotometer color measurements were developed.

### Introduction

Cotton production and usage is a global enterprise, and color measurements of cotton fiber and cotton textile products are important quality parameters. Domestically produced cotton is with the Uster® High Volume Instrument (HVI), including cotton color. Cotton color by the HVI is presented by two cotton-specific color parameters—Rd (fiber’s diffuse reflectance) and +b (fiber’s yellowness)—through the use of two broad-band filters. These two filters cover only 2 specific regions of the visible spectrum. Two types of HVI color standards are provided by the Agricultural Marketing Service (AMS) of the United States Department of Agriculture (USDA)—a set of 5 ceramic tiles and a set of 12 uniform cotton fiber “bricks” (Figure 1). The AMS uses a master colorimeter (HVI) to obtain the Rd and +b for each fiber and tile standard.



Figure 1. Examples of AMS Color Standards, ceramic tiles and cotton fiber bricks.

A vast majority of the globally recognized color systems are based on 3-dimensional tristimulus color, such as  $L^*a^*b^*$  or CIELAB, which covers the entire visible spectral region from 400nm-700nm. In  $L^*a^*b^*$ ,  $L^*$  denotes the

lightness or darkness of the sample,  $a^*$  denotes the greenness or redness of the sample, and  $b^*$  denotes the blueness or yellowness of the sample. (Billmeyer and Saltzman, 2000; Harold, 1992; Judd and Wyszecki, 1975). Color differences between  $L^*a^*b^*$  results can be expressed for each parameter by the difference between a reference unit and the unit being compared for  $L^*$  ( $DL^*$ ),  $a^*$  ( $Da^*$ ), and  $b^*$  ( $Db^*$ ). The primary expression for color difference between samples or color units evaluates all 3 dimensions at the same time and is often expressed by  $DE^*$  (equation 1), which is the square root of the sum of the squares of  $DL^*$ ,  $Da^*$ ,  $Db^*$ . In general, a color difference is considered to be significant when  $DE^* > 1.0$ . (Berger-Schunn, 1994)

$$DE^* = \sqrt{(DL^*)^2 + (Da^*)^2 + (Db^*)^2} \quad (1)$$

In addition, the most commonly used and well-recognized color space systems that are used for National Institute of Standards and Technology (NIST)-like traceability and standards verification use 3-dimensional color space, not 2-dimensional. Thus, the AMS standard tiles and standard cotton bricks measured on the master HVI colorimeter have no “NIST-like” traceability or means to certify/verify the color results. These HVI color standards limitations (2-dimensional color space, no traceable/verifiable standards) offer the opportunity for significant improvements in fiber color measurements could lead to a much improved overall color analysis system for cotton fibers, to include standards traceability or certification, and these improved systems and protocols could be used to complement and strengthened the present HVI cotton color system.

Previous evaluations on both AMS tiles and cotton bricks established strong  $L^* \leftrightarrow R_d$  and  $b^* \leftrightarrow +b$  correlations between bench-top and portable spectrophotometers and the HVI. (Thibodeaux, et. al., 2008; Rodgers, et. al., 2008a; Rodgers, et. al., 2009) Color unit agreement ( $DE^*$ ) was often  $< 1.0$  (not significant) for tiles when HVI glass (6 mm thick) was not used in the spectrophotometer color measurement (tile placed directly against the measurement port), but color unit agreement was often significant ( $DE^* > 1.0$ ) for tiles when glass was used in the spectrophotometer color measurement (tile placed against the glass placed against the measurement port; Figure 2). The color parameter most impacted by glass use was  $L^*$ . The feasibility of developing “traceable” color measurement standards for the HVI and color spectrophotometers was demonstrated, using metal or ceramic tiles without glass use and  $L^*a^*b^*$  color space.



Figure 2. Examples of HVI glass use, tile and fiber color measurements.

Glass use is often required for cotton fiber color measurements in order to present a smooth surface for measurement and to prevent potential contamination of the spectrophotometer. Due to its impact on  $L^*$ , evaluations were performed to ascertain the extent of the impact of glass use on color measurements. (Rodgers, et. al., 2008b) Glass impact could be significantly reduced with the use of specific instrumental conditions and correction factors.

With the establishment and validation of strong  $L^* \leftrightarrow R_d$  and  $b^* \leftrightarrow +b$  correlations and procedures to minimize glass impacts, two additional areas were addressed for cotton fiber color measurements on spectrophotometers. A program was implemented to 1) determine the impact pressure on spectrophotometer fiber measurements (small and large/bulk samples) and 2) to develop an improved, pressurized fiber sampling system for large/bulk cotton fiber samples, with initial emphasis on AMS cotton bricks. The program was a joint project between the AMS, Cotton Incorporated, and the Cotton Structure & Quality (CSQ) research unit with the Southern Regional Research Center (SRRC) of the Agricultural Research Service (ARS).

### **Experimental**

The samples used in this evaluation consisted of one box of AMS standard cotton bricks (12 bricks per box) and a set of 5 domestic and 5 international cotton fiber samples. The cotton fiber samples were measured “with glass” (glass was placed between the cotton sample and the spectrophotometer port) on the Gretag Macbeth CE7000A color spectrophotometer at SRRC. The spectrophotometer settings were illuminant D65, 10° observer, and large area of view. Each sample was measured 5 times by specular component included (SCI).

For small, “fluff” samples of cotton, fiber was placed in a HunterLab Associates fiber compression cell (Figure 3). Color measurements were made on the 5 domestic and 5 international samples from 10 to 40 psig (pounds per square inch, gauge), in increments of 10 psig.



Figure 3. HunterLab fiber compression cell.

For large/bulk samples (e.g., AMS cotton bricks), a prototype pressurized large sample fiber sampling system was developed for the SRRC Macbeth CE7000A. Measurements were made on both the 12 AMS cotton bricks and the 5 domestic and 5 international cotton samples. Color measurements performed from 10 to 40 psi, in 10 psi increments, with both 6 mm thick HVI glass and 1 mm thick thin glass. All color results for  $L^*a^*b^*$  and within standard deviation for each color parameter were compared at each pressure and versus the typical hand-applied pressure method.

### **Results and Discussion**

#### **Pressure Impacts, Small “Fluff” Fiber Samples**

The color of small quantities (“fluffs”) of fiber samples can be measured by placing the sample into a HunterLab Associates fiber compression cell, in which a plunger compresses the fiber at a specified pressure against a glass surface (“with glass” fiber color measurement). Color measurements were performed from 10-40 psig (pounds per square inch, gauge), in 10 psig increments. The color parameter most impacted by increasing applied pressure was  $L^*$ , with only minor (not significant) color differences observed with increasing pressure for  $a^*$  and  $b^*$  (Figure 4). As the applied pressure is increased, a color “plateau” is reached for  $L^*$  at 20-30 psig, after which only minor  $L^*$  changes are observed for most samples. The evaluations demonstrated that consistent fiber color measurements could be obtained for small cotton samples using the HunterLab fiber compression cell at 30 psig.

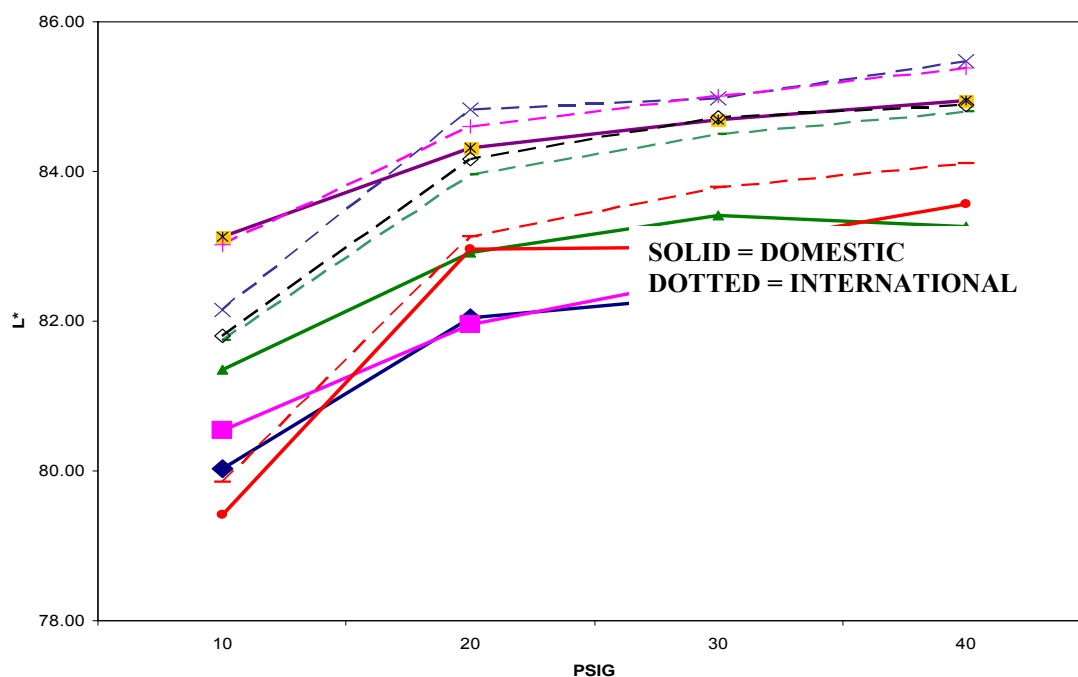


Figure 4. Impact of applied pressure on  $L^*$ , domestic and international cotton samples (HunterLab fiber compression cell, “fluff” cotton samples).

#### **Pressure Impacts, Large “Bulk” Fiber Samples**

The HunterLab fiber compression cell was not acceptable for large, “bulk” fiber samples such as the AMS cotton bricks. A program was implemented to develop and fabricate a fiber sampling system that would be acceptable for both small fluff samples and large bulk samples. A prototype system was developed, with applied pressure up to 50 psi available. The AMS cotton bricks were measured on the spectrophotometer, in the boxes in which they were placed by AMS, from 10 to 40 psi in 10 psi increments. For the domestic and international cotton samples (“regular” cottons), the fiber was placed in an AMS cotton brick box and were measured on the spectrophotometer from 10 to 40 psi in 10 psi increments. In addition, all large samples were also measured by hand pressure (Figure 2). The color results and within standard deviation (5 reps) for  $L^*$ ,  $a^*$ , and  $b^*$  were determined for each pressure and condition.

For all systems,  $L^*$  was the color parameter most impacted by increasing applied pressure, with only minor differences (not significant) observed for  $a^*$  and  $b^*$ . With HVI glass, the AMS bricks exhibited a higher average  $L^*$  value with the large sample pressure system compared to the conventional hand pressure method (Figure 5). The average  $L^*$  values with the large sample pressure system were overall very consistent, with only a small increase from 10 psi to 20 psi. In order to be consistent with the pressure evaluations with the small fluff cotton samples, 30 psi was selected as the operational pressure for large bulk samples and the SRRC large sample pressure system. The  $L^*$  within standard deviation (SD) for each sample at each pressure and condition was obtained ( $n=5$  reps), and the average SDs compared. Significant improvements in  $L^*$  SD were observed with the large sample pressure system compared to the hand pressure method, especially at  $\geq 30$  psi (Figure 6). Similar results were observed with thin glass. For the domestic and international “regular” cotton bulk samples, the results were similar to those observed for the AMS cotton bricks, but the observed higher  $L^*$  results and improved  $L^*$  SDs with the large sample pressure system were much greater.

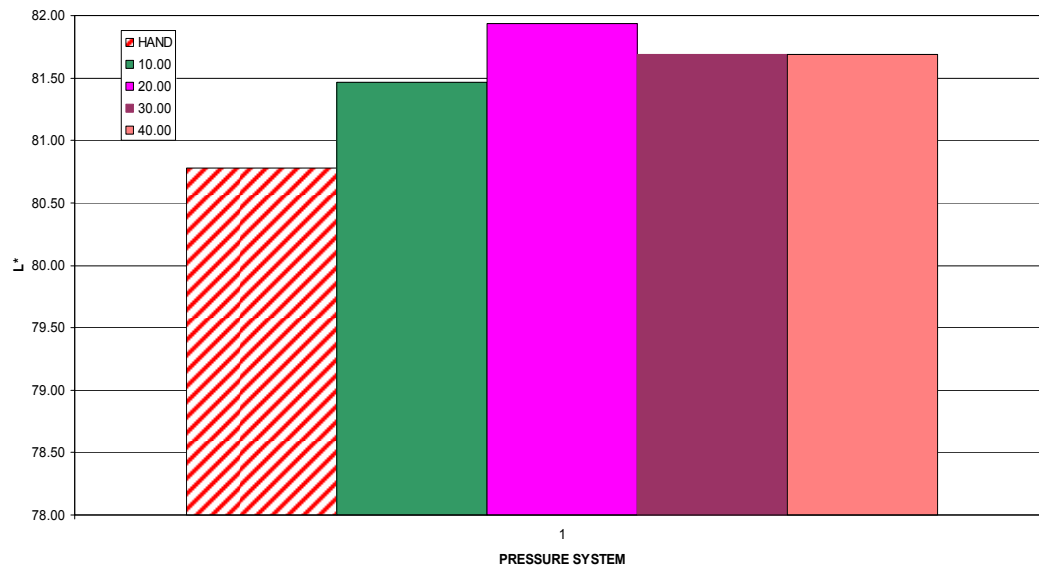


Figure 5. Average L\*, AMS cotton bricks measured by hand pressure and the SRRC large sample pressure system, HVI glass.

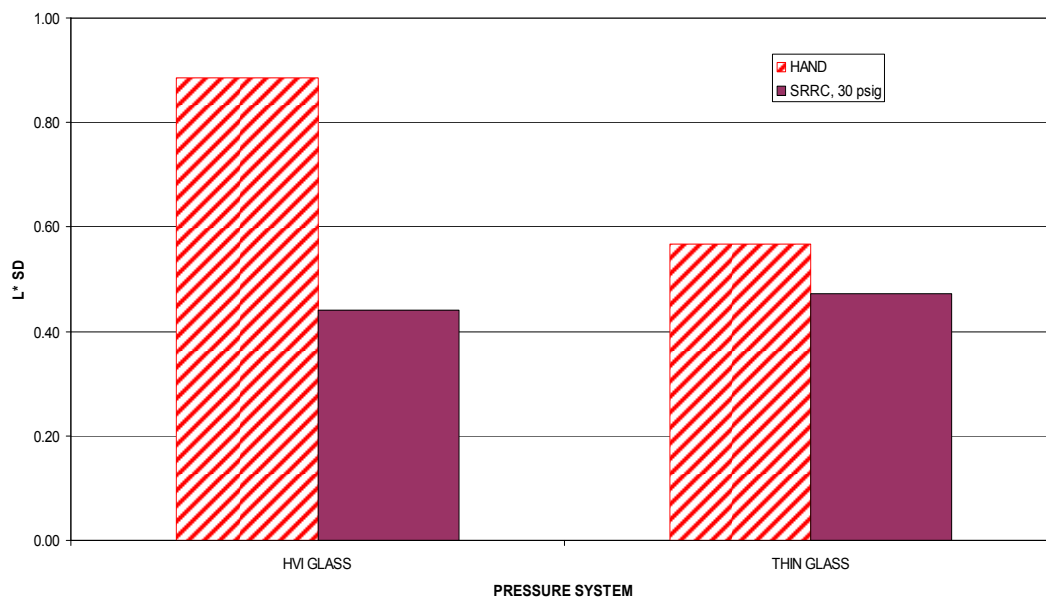


Figure 6. Average L\* Standard Deviation, AMS cotton bricks measured by hand pressure and the SRRC large sample pressure system, HVI glass and thin glass.

### **Conclusions**

A program was implemented to 1) determine the impact of applied pressure on spectrophotometer fiber measurements and 2) to develop an improved, pressurized fiber sampling system that can precisely measure large cotton fiber samples (e.g., standard AMS bricks and “regular” cottons). For both domestic and international cottons, the pressure impact results for small fiber “fluff” samples indicated that applied pressure impacted primarily fiber

L\*. As the applied pressure is increased, a color “plateau” is reached for L\* at 20-30 psig, and 30 psig was selected as the optimum applied pressure for small “fluff” fiber color measurements. For large fiber samples (e.g., Agricultural Marketing Service/AMS cotton bricks), a prototype sampling system was developed. As observed for the small “fluff” samples, L\* was the color parameter most impacted by increasing applied pressure. Higher average L\* values were observed for the large sample pressure system compared to the conventional hand pressure method, and overall very consistent average L\* results were observed, even at 10 psi. Significant improvements in L\* SD were observed with the large sample pressure system compared to the hand pressure method, especially at  $\geq 30$  psi. Protocols for pressurized fiber measurements on spectrophotometer color measurements were developed. In order to be consistent with the small fluff cotton samples results, 30 psi was selected as the operational pressure for large bulk samples and the SRRC large sample pressure system.

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