ANALYSIS OF COTTON MATURITY AND FINENESS BY MULTIPLE NIR HVIS PART II: REFERENCE METHOD

J.G. Montalvo, Jr. and S.E. Faught
USDA, ARS, Southern Regional Research Center
New Orleans, LA

Abstract

High precision headspace resistance standards (HRS) were developed at SRRC for the Micromat model of the fineness and maturity tester (FMT). HRS are stable, physical standards used to create precise differential pressures when air is drawn through the device. FMT calibration PL and PH air flow rates are "transferred" to the HRS resulting in declared HRS PL and PH values. Recalibration and control of the FMT during routine analysis is performed by adjusting the flow controllers to maintain the declared HRS PL and PH values within specification. A leak detector module (LDM) was installed to test for leaks. Routine analysis of 100 cottons is presented based on HRS recalibration of the FMT. Elimination of drift in instrument readings over a 3-month period is demonstrated and confirms earlier work.

Introduction

Recent work indicates that fast diode-array high volume-instrumentation (DA HVI) has the potential to measure cotton maturity and fineness in an on-line application (Buco, Montalvo, Faught, Price, Meredith, Stark, and Luchter, 1997; and Stark, Luchter, Jamil, and Montalvo, 1997). The DA HVI was calibrated with cottons analyzed by the FMT (Micromat Model). HRS were used to recalibrate and control the Micromat during routine analysis (Montalvo and Faught, 1997). Elimination of drift in Micromat readings over a 6-week period was demonstrated. Other improvements to the Micromat operating procedure included defining an acceptable sample weight range and controlling temperature changes and other contributing factors which could lead to biases and affect precision. A leak detector module was installed to test for leaks. New maturity and fineness equations were derived as a function of PL and PH. This paper focuses on the use of HRS to recalibrate and control the Micromat during routine analysis of an additional set of 100 cottons in 1997.

Materials and Methods

Samples

The cottons were provided by the Agricultural Marketing Service (AMS) in Memphis, TN as part of the 1996 quality assurance check lot program. More than 600 samples were shipped to SRRC, but only 200 met our criterion for this study of ≥ 80 g of fiber. The 200 samples were numbered sequentially in the order they were received from the AMS and divided into four groups (I, II, III, and IV) of 50 each by selecting every 4th sample. Samples from groups I and II (N = 100) are included in this series of papers. Eighteen of the 100 samples were Pima cottons.

For each of the 100 samples, two 40 g subsamples (labeled A and B) from each sample were weighed. Each subsample was cleaned in a Shirley Analyzer. Eight 4.00 g specimens were weighed from each cleaned subsample. The total number of specimens analyzed on the Micromat was: 100 samples x 2 subsamples/sample x 8 specimens/subsample = 1600 specimens.

Micromat

The Shirley Developments Limited (SDL) 089 Micromat Tester is the latest in the series of FMT instruments developed to measure the maturity and fineness of cotton. An electronic balance with interface, a microprocessor with floppy and hard disk drives, and a VDU for displaying results are included.

The Group I samples were analyzed first. Two specimens from each of the 50 “A” subsamples were analyzed on the Micromat using the routine analysis procedure described below. The steps were repeated three additional times to yield 8 FMT replicates per subsample or one replicate for each 4.0 g specimen. The PL and PH readings were collated and mean subsample values computed and used to calculate the various measures of maturity and fineness.

The cycle was repeated with the 50 “B” subsamples. Then the entire procedure was continued with the Group II cottons.

Leak Detector Module (LDM)

The LDM was constructed from a series of valves mounted on plywood which was attached to one side of the FMT. The LDM was then connected to the FMT air flow system. (Details on the design of the LDM will be presented elsewhere.) The LDM allows for three operational modes: *leak detection*, *headspace resistance standards recalibration*, or *routine analysis.*

Leak Detection Mode

Set the LDM to *leak detection*. Use the FMT vacuum pump to evacuate the system to 350 mm water. Next, the leakage rate is measured with a stopwatch. Specifications were < 0.35 mm water reduction in vacuum in 10 seconds (the dwell time in both PL and PH piston stroke positions). If the leakage exceeded specifications, the system was shut down and the leak found and sealed.

Headspace Resistance Standards (HRS)

(See Montalvo and Faught, 1977, for construction of the HRS from narrow diameter copper tubes.)

Reprinted from the *Proceedings of the Beltwide Cotton Conference*
Volume 2:1625-1628 (1998)
National Cotton Council, Memphis TN

***Headspace Resistance Standards Recalibration* Mode**

Set the LDM to *HRS Recalibration*. Close the lid of the empty sample chamber and connect the flexible hose from the funnel (glued to the lid) to the HRS manifold. Open the HRS PL ON-OFF valve making sure that the HRS PH ON-OFF valve is closed. Operate the instrument in the *AUTOMATIC* mode and observe the HRS PL value on the digital pressure guage. When the piston stroke changes to PH, open the HRS PH ON-OFF valve and close the HRS PL ON-OFF valve. Observe the HRS PH value. If the observed pressure drops across the HRS are not within the declared specifications, operate the instrument in the *PAUSE* mode and readjust the flow controllers as necessary. Confirm that the FMT is in recalibration by operating the instrument in the *AUTOMATIC* mode while observing the HRS PL and PH values.

***Routine Analysis* Mode**

For this practice, the LDM remains in the HRS recalibration position, but the flexible hose from the funnel to the HRS manifold is disconnected. A typical routine analysis operational cycle is outlined in Table 1. The number of cotton specimens analyzed in a cycle is limited to 6 to insure against significant drift in instrument readings.

Results and Discussion

Precision

Table 2 shows the Micromat statistics on the 200 subsamples. The CV of Pl and PH are < 2.6% and the corresponding CV of the various maturity and fineness units of measure range from 1.22% to 2.48%. (All of the various units of measure in the table were computed from PL and PH using SRRC advanced software for the FMT -- (see Montalvo and Grimball, 1994).

The coefficients of variation (Table 2) are higher than in previous studies. There are two sources of variability that may have contributed to the observed error.

When the samples were received from the AMS, we noted that, generally, ten samples were placed in a large paper bag. Each sample had been rolled into the shape of a solid cylinder with the bale tag in the center of the cylinder. Unfortunately, there were no paper or cloth surfaces to ensure separation of the samples in a bag. When the samples were removed from a bag, sometimes the rolls were cleanly separated, particularly with rolls that had been wound up tight. However, this was not always the case and therefore, to minimize cross contamination of the samples, we were forced to rely on the differences in color between adjacent samples.

Another source of significant variability in the FMT data is the eighteen Pima samples. Comparison of the standard deviations for each sample showed that the largest errors are generally associated with the Pima cottons. We do not know at this time if this is characteristic of Pima cottons.

Sample Heterogeneity

Two 40 g subsamples were taken from each of the 100 raw cottons. There were eight specimens per subsample and one FMT observation per specimen (see Table 2). If the cottons were homogeneous with respect to maturity and fineness within a sample, then the mean FMT instrument readings for each cotton (mean PL and PH values symbolized as $\bar{x}$)

$$\begin{array}{rcccc} & \bar{x}_A = \bar{x}_B = \bar{x}_{A+B} = \bar{x}_{AB} & & & \\ & 100_8 & 100_8 & 200_8 & 100_{16} \\ \text{\# of specimens:} & 800 & 800 & 1600 & 1600 \end{array} \quad (1)$$

can be averaged across: the 100 "A" subsamples, the 100 "B" subsamples, the 200 "A+B" subsamples, and the 100 "AB" samples.

The above notation is explained below with two examples. First example: With the 100 "A" subsamples, the data was first averaged over the 8 specimens for each subsample; then the means were averaged across the 100 subsamples. Also, the standard deviation of each subsample mean was computed followed by computing the pooled standard deviation across the 100 subsamples.

Second example: With the 100 "AB" subsamples, the data was first averaged over the 16 specimens for each sample, then averaged across the 100 samples. The standard deviation of each sample mean was computed followed by computing the pooled standard deviation across the 100 samples). Note: The results reported in Table 2 refer to across the 200 subsamples (200_8).

Assume that the cottons are homogeneous with respect to maturity and fineness within a sample and that the only errors in the FMT readings are random errors. According to statistical theory, the expected variability in sample means will decrease by the square root of 2 if the number of replications is doubled:

$$\begin{array}{rccc} & PSD_{AB} & = & PSD_{A+B}/2^{0.5} \\ & 100_{16} & & 200_8 \\ \text{\# of specimens:} & 1600 & & 1600 \end{array} \quad (2)$$

where PSD = pooled standard deviation. Equation 2 can be expressed as an inequality:

$$PSD_{AB} < PSD_{A+B}. \quad (3)$$

Now assume that the cottons are heterogeneous with respect to maturity and fineness within a sample. The two subsamples for any sample in the set may not be equivalent so that the expected inequality could reverse if the difference in subsample means contributes significantly to the pooled variability:

$$PSD_{AB} > PSD_{A+B}. \quad (4)$$

Thus, by comparing the pooled standard deviation of the 100 samples against that of the 200 specimens allows for discrimination of sample heterogeneity and homogeneity.

The results are as follows (in mm water):

	PSD_{AB}		PSD_{A+B}
	100_{16}		200_{8}
PL	4.13	>	3.71
PH	3.92	>	3.54.

The pooled variability is in reverse order compared to the expected variability for homogeneous samples. This implies that the cotton samples are heterogeneous with respect to maturity and fineness.

Drift and Accuracy

If the Micromat PL and PH readings drift over time, then the resultant cotton maturity and fineness values are of questionable accuracy. Before using the HRS, we were never able to maintain extended analysis periods on the Micromat because drift was always experienced.

To test for drift with HRS calibration and control, we ran the International Calibration Cotton (I.C.C.) AM-13 as an unknown sample at a high frequency over the 3-month period required to analyze the cottons. A plot of the PL and PH readings (Figure 1) demonstrates the elimination of short-term drift. The differences in the initial and final values, computed from the lines of best fit, are less than one unit for both PL and PH.

The increased scatter in the data beginning at about data point # 42 is due to problems with temperature and humidity control in our laboratory. After the problem was rectified, the points again follow the line of best fit.

References

Buco, S. M., J. G. Montalvo, Jr., S. E. Faught, J. B. Price, W. Meredith, E. Stark, and K. Luchter. 1997. Fast determination of maturity and fineness by NIR with a diode-array HVI. Part 1. Data analysis. Proceedings Beltwide Cotton Conferences. I, 552-554.

Montalvo, J. G., Jr. and S. E. Faught. 1997. Fast determination of maturity and fineness by NIR with a diode-array HVI. Part 2. Reference Method. Proceedings Beltwide Cotton Conferences. I, 554-555.

Montalvo, J.G., Jr. and R. Grimball. 1994. SRRC maturity and fineness equations, version 1.0 software.

Stark, E., K. Luchter, M. Jamil, and J. G. Montalvo, Jr. 1997. Fast determination of maturity and fineness by NIR with a diode-array HVI. Part 3. HVI operation. Proceedings Beltwide Cotton Conferences. I, 556-557.

Table 1. Micromat *routine analysis* mode based on HRS to maintain calibration - one operational cycle.

1. Connect air flow from funnel to manifold.
2. Switch the LDM to *HRS Recalibration* mode and verify instrument is in calibration. If recalibration is necessary, adjust flows as described in the section on *Headspace Resistance Standards Recalibration* Mode.
3. Uncouple airflow from funnel to manifold.
4. Analyze 6 cotton specimens.
5. To analyze more specimens go to STEP (1).

Table 2. Micromat statistics on the 200 subsamples.

100 samples x 2 subsamples (coded A and B)/sample = 200 subsamples
8 specimens/subsample with 1 FMT observation/specimen
"A" subsamples analyzed before "B" subsamples
"A" subsamples run in four sets: 2 reps/set x 4 sets = 8 reps/subsample
"B" subsamples run in four sets: 2 reps/set x 4 sets = 8 reps/subsample
TOTAL FMT REPS = 8 reps/subsample x 200 subsamples = 1600 reps

Statistical data below is across the 200 subsamples.

Property	Mean	Pooled Std. Dev.	CV,%
PL (mm water)	192.9	3.713	1.92
PH (mm water)	136.8	3.541	2.59
Maturity ratio	0.9722	0.01908	1.96
Fineness(millitex)	170.9	3.437	2.01
Micronaire	4.332	0.05274	1.22
% Maturity	85.14	1.411	1.66
Perimeter (µm)	50.22	0.9140	1.82
Wall Thick.(µm)	2.697	0.03047	1.13
% Thickness	33.84	0.8396	2.48

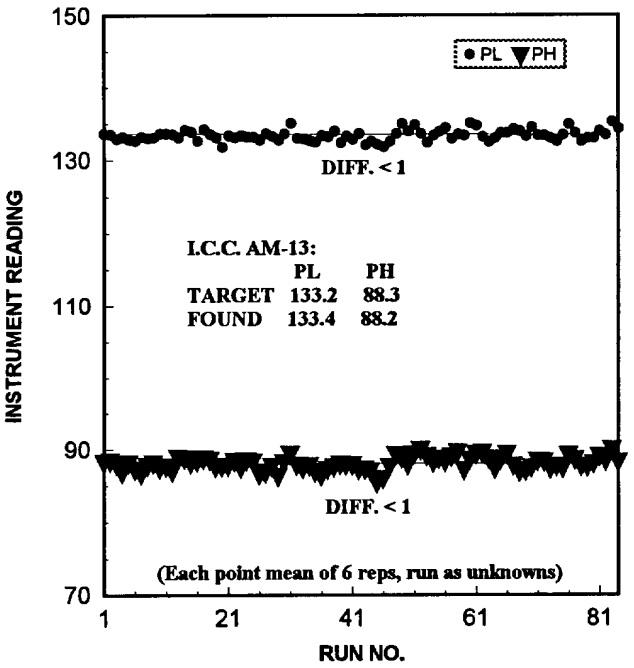

Figure 1. I.C.C. Am-13 analysis over a 3-month routine analysis period based on HRS calibration and control.