FAST DETERMINATION OF MATURITY AND FINENESS BY NIR WITH A DIODE-ARRAY HVI. PART 2. REFERENCE METHOD

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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. Micromat calibration PL and PH air flow rates are "transferred" to the HRS. Routine analysis of 36 cottons from Stonveille, MS is presented based on the transferred calibration. Elimination of short-term drift is demonstrated and confirms earlier work.

Introduction

Recent work indicates that the 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, Stark, and Luchter, 1996). The DA HVI was calibrated with cottons analyzed by the FMT (Micromat Model). HRS were used to calibrate and to control the Micromat during routine analysis (Montalvo and Faught, 1996). Elimination of short-term drift in Micromat readings 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. New maturity and fineness equations were derived as a function of PL and PH. This paper focuses on the use of HRS to calibrate and control the Micromat during analysis of an additional set of 36 cottons.

Materials and Methods

Samples. The cottons (N = 36) were grown by William Meredith of the Agricultural Service (ARS) at Stonveille, MS. Each cotton represented a different variety which had been bred in growth areas across the cotton belt in this country. The cottons were ginned by ARS at Stoneville and samples of each were distributed to various researchers. Samples for this study came from John Price of this laboratory.

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. Electronic balance with interface, microprocessor with floppy and hard disk drives, and VDU for displaying results are included.

Prior to Micromat analysis, the 36 cottons were processed at SRRC as follows: opened in an opener-blender, then cleaned in a minicard followed by a Shirley Analyzer. For each cotton, twelve 4.00 g specimens were weighed. Two specimens of each of the 36 cottons were analyzed on the Micromat using the routine analysis procedure described below. The cycle was repeated five additional times to yield 12 reps per cotton. Mean PL and PH readings were computed and were used to calculate the various measures of maturity and fineness.

Headspace Resistance Standards. HRS were created by drawing a predetermined volume of air through narrow diameter copper tubing --- about 4 liters/min (PL) or 1 liter/min (PH), the actual flow rate discussed below. The HRS were connected to a manifold with ports fitted with ON-OFF valves. An HRS manifold was constructed from a 3/8 inch Swagelok union-cross fitting and mounted on plywood. An ON-OFF valve was mounted to each horizontal port of the cross fittings was mounted.

The top of the manifold was sealed and a rubber hose was extended from the bottom of the manifold to the sample chamber lid on the Micromat. To facilitate the connection of the hose to the chamber lid, an inverted funnel was glued to the lid. The wide mouth of the funnel was of sufficient diameter to encompass all of the air holes in the lid. The hose was then connected to the stem of the funnel.

The HRS were constructed from narrow diameter copper tubing (1/4 " O.D. for PL and 1/8 " O.D. for PH) and were connected to the upstream side of the ON-OFF valves. For convenience, the HRS on the left side of the manifold was designated PL resistance, and the corresponding PH resistance was placed on the right side of the manifold.

Here is how the HRS work. With the sample chamber closed and empty, connect the airflow from funnel to manifold. Operate the instrument in the *PAUSE* mode so that air gets drawn through the manifold. With about 4 liter/min (PL) of air drawn through a narrow diameter tubing, the tubing is cut to the proper length to achieve the desired pressure drop, and then carefully coiled and mounted on the manifold. Follow the same procedure at about 1 liter/min for the PH resistance.

Calibration, Calibration Transfer, and Routine Analysis. To calibrate the Micromat, uncouple the air flow from funnel to manifold and follow instructions given in the Micromat instrument manual (Instruction Manual, 1994). Operate in the *PAUSE* mode and use International Calibration Cotton (I.C.C.) # H-3 (declared differential pressures: PL = 122 mm water and PH = 80 mm water). Adjust air flow rates to get the target PL and PH values. Remove the sample from the chamber. Close the chamber

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lid. Connect the air flow from the funnel to a digital flow meter. Measure air flow rates.

Here is how to transfer the calibration to the HRS. Make sure the air flow rates are set to the digital flow rates measured in calibration. With the chamber empty and the lid closed, connect the air flow from funnel to manifold. Open the PL ON-OFF valve. Operate the MIcromat in the *PAUSE* mode and observe the PL HRS value. Open the PH ON-OFF valve and close the PL ON-OFF valve. Operate the Micromat in the *PAUSE* mode and observe the PH HRS value. Calibration is now transferred to the HRS.

A typical routine analysis operational cycle based on HRS calibration and control is outlined in Table 1. The number of specimens analyzed in a cycle is limited to 6 to insure insignificant drift in instrument readings.

Results and Discussion

Precision. Table 2 shows the Micromat statistics on the 36 cottons. The CV of Pl and PH is < 2% and the corresponding CV of the various maturity and fineness units of measure range from o.75% to 2.24%. (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).

Note that maturity ratio, fineness, micronaire, % maturity and % thickness are different combinations of the fundamental measures of maturity and fineness, namely, wall thickness and perimeter. To improve the precision of the combinational units of maturity and fineness, it is necessary to improve the precision of the perimeter estimation.

Drift and Accuracy. If the Micromat PL and PH readings drift over time, then the resultant cotton maturity and fineness values are unreliable and, therefore, 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 I.C.C. # H-3 as an unknown sample at a high frequency over the six week period required to analyze the 36 Stoneville 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 replicated Micromat data on each of the 36 Stoneville cottons was used to compute a coefficient of determination that we refer to as R square-maximum regression, and denoted as R_{AXREG}, for each measure of maturity and fineness. This test statistic is an estimate of the upper limit in the correlation (or maximum possible correlation) between mean values of a cotton property and NIR (Watkins, Montalvo, Grimball, Vinyard, and Buco, 1996). R_{AXREG} was > 0.95 for all measures of maturity and fineness, indicating mean values with significantly reduced random error and free from drift as demonstrated above.

References

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Table 1. Micromat routine analysis based on HRS to maintain calibration, one operational cycle.

1. Connect air flow from funnel to manifold.
2. In *PAUSE* operational mode, verify PL = 122 and PH = 80 across needle valves. Adjust flows if needed.
3. Uncouple airflow from funnel to manifold.
4. Connect air flow from funnel to the digital flow meter. Verify that the digital flow rates match the calibration flow rates. Uncouple the digital flow meter.
5. Analyze 6 cotton specimens.
6. To analyze more specimens go to STEP (1).

Table 2. Micromat statistics on 36 Stoneville cottons.

1995 crop year; Samples run in six sets: 2 reps/sample/set x 6 sets = 12 reps/sample

Property	Mean	Std. Dev.	CV,%
PL (mm water)	193.1	2.332	1.21
PH (mm water)	137.0	2.175	1.59
Maturity ratio	0.9547	0.01720	1.80
Fineness(mtex)	171.2	3.328	1.94
Micronaire	4.281	0.0359	0.84
% Maturity	84.06	1.315	1.56
Perimeter (:m)	50.68	0.9148	1.81
Thick.(:m)	2.660	0.01983	0.75
% Thickness	33.01	0.7396	2.24

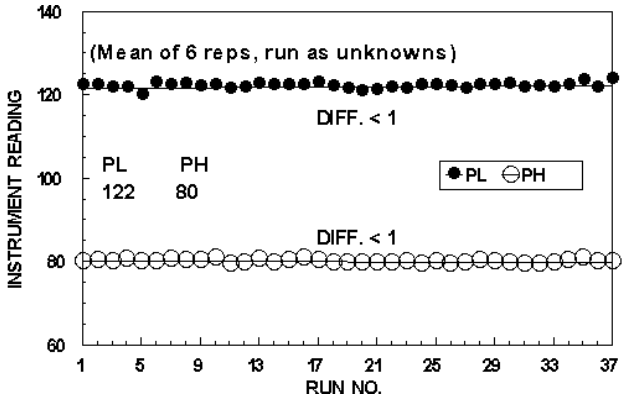

Figure 1. I.C.C #H-3 analysis over a 6-week routine analysis period based on tranferred calibration.