# DETERMINATION OF WALL THICKNESS AND PERIMETER BY FMT AND DIODE-ARRAY HVI. PART 2. FMT HEADSPACE RESISTANCE STANDARDS

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## Abstract

High precision headspace resistance standards (HRS) are developed for the Micromat model of the fineness and maturity tester (FMT). Micromat calibration PL and PH mean readings are "transferred" to the HRS. Routine analysis of 80 cottons is presented based on the transferred calibration. Elimination of short-term drift is demonstrated.

## Introduction

Recent work using an improved operating procedure for the Micromat model of the FMT to calibrate the fast diode-array high-volume instrument (DA HVI) indicates that the DA HVI has the potential to measure cotton maturity and fineness in an on-line application (Montalvo, Faught, and Grimball, 1995; Buco, Montalvo, Faught, Grimball, Stark, and Luchter, 1995). Micromat procedure modification included defining an acceptable sample weight range and controlling instrumental drift and other contributing factors which may lead to biases and affect precision. New maturity and fineness equations were derived as a function of PL and PH. Part 2 of this series focuses on headspace resistance standards to calibrate the Micromat.

## Materials and Methods

**Samples.** Cotton samples (N = 80) were from the 1993 National Cotton Variety Test (NCVT) program. These cottons represented four varieties (Acala, DP-50, DP-90, and Paymaster) from 16 growth areas in the U.S.

**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 the results are included.

Prior to Micromat analysis, the 80 cottons were cleaned in a Shirley Analyzer. For each cotton, twelve 4.00 g specimens were weighed. Four specimens were analyzed on the Micromat for each of the cottons using the routine analysis procedure described below. The cycle was repeated two 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. Outliers (between FMT and the DA HVI, see part 1) were reanalyzed on the FMT.

**Headspace Resistance Standards.** HRS were created by drawing either 4 liters/min of air (PL) or 1 liter/min of air (PH) through narrow diameter copper tubing, metal frits or high precision needle valves. The HRS were connected to a manifold with ports fitted with ON-OFF valves.

A HRS manifold was constructed from ten 3/8 inch Swagelok union-cross fittings and twenty ON-OFF valves. The union-cross fittings were connected together to form a linear manifold which was mounted vertically on plywood. To each horizontal port of the cross fittings was mounted an ON-OFF valve.

The top of the manifold was sealed and a rubber hose 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 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), metal frits or high precision needle valves and were connected to the upstream side of the ON-OFF valves. For convenience, the HRS on the left side of the manifold were designated PL resistances and each corresponding PH resistance was placed on the right side of the manifold.

Here is how the HRS work. With the sample chamber closed and no sample in the chamber, connect the airflow from funnel to manifold. Operate the instrument in the *PAUSE* mode to get air drawn through the manifold. With either 4 liter/min or 1 liter/min of air drawn through: (1) a narrow diameter tubing, the tubing is cut to the proper length to achieve the desired pressure drop and then carefully coiled, (2) the metal frit is manufactured to give the desired pressure drop, and (3) the needle valve is closed to achieve the correct pressure drop.

**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* and use the H3 calibration cotton (declared values: 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. 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, specifically, to the needle valves. Make sure air flow rates

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are set to the digital flow rates measured in calibration. Connect the air flow from the funnel to the manifold. Select the PL needle valve and open its ON-OFF valve. Operate the MIcromat in the *PAUSE* mode. Adjust the PL needle valve until PL = 122. Select the PH needle valve. Open the PH ON-OFF valve and close the PL ON-OFF valve. Adjust the PH needle valve until PL = 80. Calibration is now transferred to the HRS.

A typical routine analysis operational cycle based on HRS calibration is outlined in Table 1. The number of specimens analyzed in a cycle is limited to 12 to prevent drift in instrument readings.

## Results and Discussion

**Precision**. Table 2 shows that the statistical variation of HRS data is significantly less than that of cotton (Table 3). The tubing precision data is based on 15 replicates run with: sample chamber closed, no cotton in chamber, air flow connected from the funnel to the manifold, and running the HRS as "cotton samples". This is possible by turning the ON-OFF valves to coincide with activation of the piston in the sample chamber to the PL and PH positions. The CV of Pl and PH is < 0.2% and the corresponding CV of the various maturity and fineness units of measure are < 0.35%. (In Table 2, MR is maturity ratio, FIN is fineness in millitex units, and T and P are wall thickness and perimeter, respectively, in microns. MR, FIN, T, and P were computed from PL and PH using SRRC advanced software for the FMT -- see Montalvo and Grimball, 1994.)

Table 3 documents the precision of PL and PH readings when separate specimens of the same cotton sample were analyzed without and with HRS calibration and control. To provide a more rigorous discrimination of variation in the data, the specimens were blended prior to cleaning in the Shirley Analyzer for Micromat processing without HRS. Even so, the unblended specimens run with HRS calibration and control gave more precise instrument readings.

**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. Note the sudden change in mean PH values for the third set of runs (Table 3, without HRS control). Without the HRS, we were never able to maintain extended analysis periods on the Micromat because severe drift problems were always encountered.

To test for drift with HRS calibration and control, we periodically ran the H3 calibration cotton over the three week period required to analyze the 80 NCVT 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.

From the 12 PL and PH readings on each of the 80 NCVT cottons, wall thickness and perimeter, micronaire, maturity ratio, and fineness (in millitex) were computed using SRRC advanced software for the FMT (Montalvo and Grimball, 1994). The replicated data was used to compute a coefficient of determination that we refer to as R square-maximum regression, and symbolized as $R^2_{MAXREG}$, 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). $R^2_{MAXREG}$ 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 12 cotton specimens.
6. To analyze more specimens go to STEP (1).

Table 2. Example precision of HRS (tubing).

| | PL | PH | MR | FIN | T | P |
|---|---|---|---|---|---|---|
| mean | 205. | 145. | 0.959 | 160. | 2.58 | 48.9 |
| std dev | 0.164 | 0.259 | 0.003 | 0.467 | 0.00245 | 0.154 |
| CV(%) | 0.080 | 0.189 | 0.309 | 0.202 | 0.0948 | 0.315 |

Table 3. Micromat statistics on NCVT cottons.

80 cottons, 1993 crop

Samples run in three sets: 4 reps/set·sample x 3 sets = 12 reps/sample

| | **PL** | | | **PH** | | |
|---|---|---|---|---|---|---|
| SET # | MEAN | STD DEV | CV(%) | MEAN | STD DEV | CV(%) |
| BLENDED SPECIMENS RUN WITHOUT HRS CALIBRATION AND CONTROL | | | | | | |
| 1 | 189.8 | 1.86 | 0.98 | 126.9 | 2.12 | 1.67 |
| 2 | 189.2 | 2.03 | 1.07 | 126.4 | 2.41 | 1.91 |
| 3 | 189.2 | 1.74 | 0.92 | 129.8 | 1.56 | 1.28 |
| UNBLENDED SPECIMENS RUN WITH HRS CALIBRATION AND CONTROL | | | | | | |
| 1 | 190.4 | 1.77 | **0.93** | 135.2 | 1.69 | **1.25** |
| 2 | 190.2 | 1.98 | **1.04** | 134.3 | 1.80 | **1.34** |
| 3 | 189.6 | 1.70 | **0.90** | 134.5 | 1.65 | **1.23** |

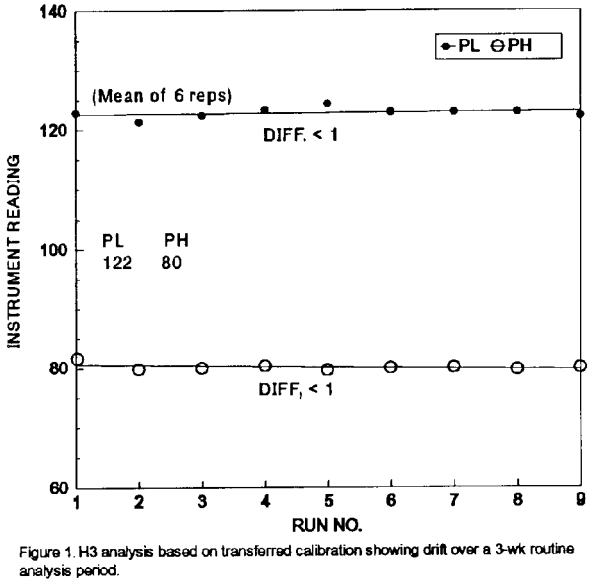


Figure 1. H3 analysis based on transferred calibration showing drift over a 3-wk routine analysis period.

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