# INDICATING MOISTURE IN COTTON WITH NEAR INFRARED LIGHT IS FAST, ACCURATE AND COMPATIBLE TO HVI TESTING

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## Abstract

Five different moisture instruments were considered for use as candidates to integrate into HVI systems. Three measured electrical properties of cotton (capacitance, and two conductivity methods) and two measured reflectance of near infrared light. All were compared with weight loss by oven drying. The two infrared light systems differ in illumination geometry, light wavelength and reflectance detector material. One reflectance method was nearly as accurate as oven measurements (±0.2%). Moisture content in 1500 cottons covering the full range of grade and trash content are reported for the best near infrared method at 60% RH.

## Introduction

Inspection of cotton is required to grade the commodity according to quality. Within the grading system, physical measurements of fiber length, strength, fineness, color and trash content are needed to establish its basic quality grade. For improved marketing the USDA is developing high volume instruments (HVI) which rapidly measure cotton fiber quality. HVI now measures several physical characteristics on nearly half of the 12 to 14 million bales of U. S. Cotton. To ensure measuring accuracy, cotton samples must be pre-conditioned to within a specific range of moisture content and testing must be performed in a controlled environment.

Some HVI fiber measurements are greatly affected by changes in moisture content. Fiber strength will increase nearly 10% with a 1% increase in moisture content [4]. Therefore, extensive sample conditioning is essential to assure reliable strength results. However, different cottons reach a different moisture level when conditioned in the same environment. Equilibrium moisture content may differ as much as 1.5% between cottons (at 60% RH). Moisture will also drop with length of storage following harvest. At present, it is not clear how much of these moisture differences occur in the molecular structure and contribute to changes in fiber strength or how much is in surface contaminants or extraneous material and only affect the strength measurement. Therefore, moisture measurements made during HVI strength testing would be beneficial when comparing test results between locations and in cotton acceptance or arbitration.

Quantitative analyses with NIR reflectance has provided a means to accurately estimate concentrations of certain organic compounds in agricultural products [2]. Early investigations with cotton demonstrated the feasibility of measuring fiber fineness [3] and several organic contaminants [5]. NIR applications in cotton have not been widely adopted because of two main factors; 1) Cotton is very variable and cannot be readily homogenized, 2) Cotton contains more than 90% cellulose which makes accurate NIR measurement of minor compounds difficult. However, owing to the strength of water absorption in region II (1100 to 2500 nm), calibration for moisture should be very accurate. Early NIR moisture calibrations (using oven data) indicated a changing bias which depended on the amount of cleaning (removal of vegetable matter) and the length of sample storage [6]. However, wavelength selection by calibrating with weight loss by desiccation eliminated trash and aging biases [7]. After proper wavelength selections , calibrations were adjusted to indicate oven moisture. The purpose of this research was to compare the accuracy of NIR methods with other conventional moisture instruments and report moisture measurements for 1500 bales tested in equibilibrium at 60% RH.

## Experimental Materials

### Cotton Lint Samples

Two independent groups of cottons were used to examine the characteristics of moisture measurement in ginned lint. One group included 13 cottons which covered the full range of color and trash content occurring in lint cottons (Figure 1). This group was used because of their wide range of visible color and trash characteristics. They were extensively measured in a "round robin" trashmeter evaluation program. The second group consisted of 1500 cottons selected for several gin locations across the belt. Both groups included a wide range of quality but the second group more closely represented typical production qualities (Table 1).

### Instruments

Laboratory equipment used in the experiment included: bell jar type desiccators with stop-cock grease seals and calcium sulfate as the desiccant; individual sample drying bottles (40 ml) with ground glass covers; a precision laboratory balance with one milligram accuracy; and a thermostatically controlled electric over. A Hart moisture meter which measures the electrical conductivity through cotton samples compressed between two flat circular electrodes (1.9 inches in diameter) was included for comparison. A Strandberg moisture meter which also measures electrical conductivity was used for comparison. The Strandberg meter measures conductivity between two sharp pointed electrodes pressed into the cotton sample surface (one inch apart). Capacitance measurements were made in the precision dialectric measuring cup used by Lyons [8]. The cup has circular electrodes measuring 1.9 inches in diameter. However, a modern electronic instrument was used to measure cup capacitance (Continental Specialties Corporation Model 3001). This instrument used a bridge method at two killohertz. The NIR spectrometer was Pacific Scientific Model 6350 which includes a grading type monochrometer. Sample observation cups fitted with quartz windows were used (two inches in diameter). Samples were rotated off center at 10 rpm to improve large area averaging. Calibrations were developed and tested with the Infra-Soft International forage computer software routines.

## Experimental Methods

All moisture comparisons were made with samples that had been exposed at least 24 hours to circulating air in a temperature and humidity controlled laboratory (±2% RH). Equilibrium conditioning was needed because the moisture in cotton changes when exposed to a different environment. Additionally, surface and bulk measurements made by different instruments will agree best with weight loss from oven drying when equilibrium samples are used. Two experiments are described: 1) The 13 round robin cottons were conditioned at two room humidities (55% and 75%) for moisture testing by all five methods. 2) The best NIR method was used to measure the moisture in 1500 cottons of known color grade and trash content.

### Reference Method

The accepted method of testing raw cotton for moisture content involves drying samples in a thermostatically controlled oven at 105 ±, 2°C with forced air circulation [1]. Samples are dried until their weight change between successive weighings drops below 0.1% of the sample weight (usually overnight). Samples are allowed to cool in a desiccator before their final dry weight is recorded. The stated standard error of repeat sampling for moisture content using the oven method is 0.2%. Under carefully controlled conditions used in our experiments the standard error of repeating the oven moisture measurement was 0.07%.

### Instrument Methods

Instrument measurements of moisture (except the Strandberg hand held probe) were made by weighing a 1.0 ± 0.05 gram specimen without blending or plucking to prevent moisture changes. Two such specimens were tested by each instrument and combined in a weighing bottle for each oven drying replication. NIR Spectrometer procedures used the same (one gram) specimen from each cotton used by the Hart Meter and the capacitance measurements. They were uniformly packed into a sample observation cup which contains a spring loaded pressure plate to produce a uniform sample pressure against the observation window. Sixty-four reflectance spectra (log 1/R) were measured and averaged to improve the signal-to-noise ratio.



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Spectra were measured for two seperate NIR regions. Region I covered the wavelength range from 680 to 1235 nm and region II covered the range from 1100 to 2500 nm. Wavelength scales for each region were calibrated with special filters which exhibit several sharp absorption bands (Figures 2 and 3). Computer routines were used to precisely locate peaks and compare their location with known wavelength values. The entire NIR sample observation process requires about 2 minutes per cotton per region (one minute for detector reference calibration and one minute for sample measurement). Calibration samples were also preconditioned at two room humidities to force a wide range of moisture content.

## Moisture Wavelength Selection

Capital cost and slow testing speed make spectrometers impractical for use in HVI lines. However, they can be used to specify the best wavelengths for fixed filter designs and they can estimate design accuracy before new instruments are built. Fixed filter instruments are faster and cheaper than their spectral counterparts. For example a colorimeter is a three filter design equivalent of a visible spectrometer.

The spectral response to changes in cotton moisture was used to select the best wavelength for filters. Examples for one cotton sample (round robin set) are shown in Figures 4 and 5. Water absorption bands can be seen near 940, 1450 and 1940 nm wavelengths. Selecting filter wavelengths is a three step process:

- Four spectra were measured and stored in the computer for each of 13 cottons, at two moisture levels and two spectral regions (208 spectra per cotton).
- Oven moisture was immediately measured in each sample and it's value (percent) entered into the computer for calibration.
- Statical correlations were performed at each spectral wavelength between measured reflectance values (log 1/R) and oven moisture to select the best wavelengths for correlation (Table 3).

The first two wavelengths in the region II calibration were forced to match those identified in previous experiments as best for moisture measuring stability [7].

## Spectrometer Calibration Checking

A standard practice in calibrating other cotton testing instruments involves establishing "reference" or "known" values of the desired measurement with one or two cottons which are evaluated in well controlled laboratories. Subsamples of the standard cotton with its known values are distributed to field locations for use in checking calibrations of field instruments. The feasibility of using a reference cotton in sealed cups to calibrate NIR field systems for moisture was investigated. Very low standard error for repeat instrument measurements with the sealed sample was observed (Table 2). NIR predicted values are also recorded for reflectance, yellowness and micronaire which are indirectly correlated to the other physical cotton measurements. Wavelengths used in these other calibrations are unrelated to moisture but checking their response gives a more complete picture of instrument performance.

## Results and Discussion

Long sample conditioning is not practical for HVI field measurements of incoming cotton. Since individual cottons reach different moisture levels (at equilibrium) it is impossible to establish the amount of conditioning achieved with one measurement. Therefore, cotton moisture measured during testing would be beneficial to instrument users. Measurements which provide the desired moisture reading should be free of errors due to cotton color, grade, growing location and trash content for a wide range of cotton sources.

## Precision and Accuracy Compairsons

Statistical comparisons between each instrument method and oven moisture (Tables 4 and 5) showed NIR region I produced the largest errors (0.59 and 0.67%) while the two wavelength design using NIR region II produced the smallest error (0.19%). Currently used hand probes (Strandberg meter) produced two times the of error (0.40%) compared to the best NIR method. We observed that there were considerable operator errors and techniques involved in recording moisture with the hand probe. Some new moisture instruments are based on measuring capacitance. Our data did not find the capacitance method very accurate (0.47%) This result was consistant with the findings of Lyons Et. al. [8] who showed that capacitance in cottons at 6% moisture could differ by 25%.

## Moisture in Graded Cottons

In general, moisture in conditioned cotton increased as its quality decreased. This trend was true except for the best grades (grade 11 and trash grade 1). The same moisture results were averaged by classers grade (Table 6) or by trash grade (Table 7). Because grade and trash are interrelated, the data did not indicate a moisture preference.

During the experiment to characterize moisture in conditioned cottons, one hundred seventeen random samples were collected in sealed drying bottles to use as a running check with oven moisture. Average moisture for the full population and the check samples compared extremely well (Table 8). However, the usual standard error between the NIR and oven check measurements remained (0.25%). A comparison of the extreme NIR check values with measured oven values suggest a slope error in the calibration. Errors in calibration can be reduced as methods and experience are developed.

## Conclusion and Recommendations

NIR spectrometer calibrations for moisture in cotton are very accurate because of the strength of the water absorption band. A direct comparison with other moisture instruments showed one NIR method had two times the accuracy of it's closest competitor. However, caution should be used during calibration to include cottons representing the full range of fiber fineness, lint color (reflectance and yellowness), and extraneous material which may affect the reflectance spectra in samples.

The development of a low cost field instrument employing NIR reflectance is recommended. Moisture measurements can be easily integrated into the current cotton colorimeters.

## Literature Cited


1. "American Society for Testing Materials", Committee D-13 Textile Materials, ASTM Designation: 2495-75

2. K. H. Norris, R. F. Barnes, J. E. Moore, and J. S. Shenk. Predicting Forage Quality by Infrared Reflectance Spectroscopy, 1976. J. of Animal Science 43 (4): 889-897

3. H. H. Ramey, Jr., Estimating Quality Components of Natural fibers by Near-Ingrared Reflectance. 1982. Textile Res. J. Vol. 52: 20-25

4. R. A. Taylor, Cotton Tenacity Measurements with High Speed Instruments, 1986. Textile Res. J. Vol 56: 92-101

5. R. A. Taylor, Cotton Fiber Quality and Contamination Measurements Using Near Infrared Reflectance, Akademiai Kiado, Budapest, Hungary, 1987, 253-239

6. R. A. Taylor, Measuring the Moisture Content of Cotton with Near Infrared Reflectance, 5th Annual NIR Users Conf. 1987, Silver Spring, MD

7. R. A. Taylor, Predicting the Moisture Content of Materials containing Oven Sensitive Volatiles by Near Infrared Spectroscopy, 1987 Intl. Proc. Royal Socety of Chemistry, Norwich, England

8. D. W. Lyons, J. A. Seabolt, J. Graham, The Dielectric Properties of Various Cotton Varieties, Text Res. J. Vol 43: 110-115

Table 1. Summary of cotton measurements for evaluation of indirect moisture indication with electrical and optical reflectance.

| Measurement | Observations per Cotton | Mean | Min | Max |
|---|---|---|---|---|
| ----------round robin cottons (N=13)---------- | | | | |
| Non-Lint Content | | | | |
| Visible (%) | 2 | 5.48 | 0.63 | 11.22 |
| Total (%) | 2 | 6.90 | 1.31 | 13.00 |
| Reflectness (%) | 8 | 65.6 | 54.1 | 79.7 |
| Yellowness (+b) | 8 | 10.4 | 7.5 | 15.0 |
| Oven Moisture (%) | 2 | 7.42 | 6.22 | 8.94 |
| Hart Meter (%) | 4 | 6.88 | 5.35 | 8.30 |
| Strandberg Mtr.(%) | 4 | 7.43 | 5.50 | 9.32 |
| Capacitance (pf) | 4 | 32.06 | 9.63 | 69.75 |
| NIR Region I(2t;%)* | 4 | 7.43 | 6.60 | 9.02 |
| NIR Region I(4t;%) | 4 | 7.41 | 6.52 | 9.21 |
| NIR Region II(2t;%) | 4 | 7.28 | 6.34 | 8.24 |
| NIR Region II(3t;%) | 4 | 7.36 | 6.28 | 8.44 |
| ----------graded cottons (N=1500)---------- | | | | |
| Non-Lint Content | | | | |
| Visible (%) | 2 | 3.35 | 0.69 | 18.04 |
| Total (%) | 2 | 5.05 | 1.43 | 20.78 |
| Reflectance (%) | 8 | 71.4 | 48.2 | 82.0 |
| Yellowness (+b) | 8 | 9.9 | 6.7 | 15.6 |
| Grade** | 1 | 50.6 | 11 | 85 |
| Trash** | 1 | 4.07 | 1 | 8 |
| NIR Region II(3t; %) | 2 | 6.26 | 5.00 | 7.54 |

*t indicates the number of terms or wavelengths
** indicates visual classing

Table 2. Summary record of spectrometer predictions using a sealed check cotton sample.

| Date | Color RD | +B | H2O | MIC |
|---|---|---|---|---|
| 31-JUL | 63.44 | 12.27 | 5.42 | 3.34 |
| 3-Aug | 64.78 | 13.49 | 5.49 | 3.29 |
| 4-Aug | 65.05 | 14.24 | 5.39 | 3.40 |
| 5-Aug | 65.75 | 14.31 | 5.35 | 3.35 |
| 7-Aug | 65.97 | 14.40 | 5.40 | 3.33 |
| 10-Aug | 65.69 | 14.87 | 5.31 | 3.33 |
| 28-Aug | 62.98 | 14.30 | 5.44 | 3.34 |
| 31-Aug | 62.28 | 14.05 | 5.55 | 3.33 |
| 1-Oct | 65.68 | 13.37 | 5.41 | 3.32 |
| 9-Dec | 66.09 | 14.29 | 5.37 | 3.37 |
| 9-Dec | 66.10 | 14.25 | 5.39 | 3.37 |
| 9-Dec | 65.64 | 14.60 | 5.30 | 3.35 |
| 17-Dec | 67.06 | 15.09 | 5.34 | 3.36 |
| 18-Dec | 66.13 | 13.77 | 5.30 | 3.33 |
| Mean | 65.62 | 14.19 | 5.39 | 3.42 |
| St. Dev. | 2.13 | .78 | .07 | .32 |
| Today's Check | | | | |
| 22-Dec | 64.95 | 13.14 | 5.43 | 3.31 |
| Error* | -.31 | -1.35 | .58 | -.38 |

* Error is the number of standard deviations today's check differs from the mean

Table 3. Wavelengths and accuracy of selected spectrometer calibrations for moisture using round robin cottons.

| NIR Region | terms (n) | wavelengths (nm) | correlation $R^2$ |
|---|---|---|---|
| I | 2 | 1142,1152 | 0.673 |
| I | 4 | 1142,1152,959,969 | 0.768 |
| II* | 2 | 1966,2054 | 0.785 |
| II | 3 | 1966,2164,2296 | 0.822 |

* From [7]

Table 4. Oven moisture measured in round robin cottons during instrument testing (percent)

| Condition | RH | Mean | Max* | Min** |
|---|---|---|---|---|
| Low | 55 | 6.63 | 7.06 | 6.22 |
| High | 75 | 8.21 | 8.94 | 7.52 |

* Tinged cotton with 11.2% non-lint content and 3.4 micronaire
** Spotted cotton with 2.4% non-lint and 5.1 micronaire

Table 5. Moisture content precision and accuracy comparison between instrument methods and weight loss in oven drying (percent).

| Method | $R^2$ | Slope | Intercept | Standard Error |
|---|---|---|---|---|
| Hart Meter | 0.81 | 0.719 | 2.47 | 0.40 |
| Strandberg Meter | 0.81 | 0.500 | 3.71 | 0.40 |
| Capacitance | 0.73 | 0.037 | 6.23 | 0.47 |
| NIR Region I(2t)* | 0.45 | 0.987 | 0.08 | 0.67 |
| NIR Region I(4t) | 0.60 | 1.011 | -0.07 | 0.59 |
| NIR Region II(2t) | 0.96 | 1.300 | -2.04 | 0.19 |
| NIR Region II(3t) | 0.95 | 1.162 | -1.13 | 0.21 |

* t indicates the number of terms or wavelength

Table 6. Average cotton moisture at 60% RH by grade*

| Trash | Color Plus 0 | White 1 | Lt. Spot 2 | Spot 3 | Tinged 4 | Stained 5 |
|---|---|---|---|---|---|---|
| 1 | | 6.22 | | | | |
| 2 | | 6.03 | 5.74 | 6.16 | | |
| 3 | | 5.94 | 6.05 | 6.17 | | |
| 4 | 5.92 | 5.95 | 6.10 | 6.30 | 6.66 | |
| 5 | 5.92 | 6.07 | 6.21 | 6.36 | 6.65 | |
| 6 | 6.20 | 5.93 | 6.36 | 6.47 | | |
| 7 | | 6.65 | | | | |
| 8 | | | 6.63 | 6.72 | 6.76 | 6.62 |

* all data in weight percent of conditioned lint

Table 7. Average cotton moisture at 60% RH by trash grade*.

| | Trash Grade 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 |
|---|---|---|---|---|---|---|---|---|
| Moisture | 6.10 | 6.02 | 6.16 | 6.23 | 6.38 | 6.48 | 6.59 | 6.84 |
| Number | 10 | 108 | 344 | 555 | 326 | 117 | 25 | 15 |

Table 8. Summary of moisture content measurements in graded cottons.

| Samples | N | Mean | SD | MIN | MAX | SE(oven) |
|---|---|---|---|---|---|---|
| Lint | 1500 | 6.26 | 0.39 | 5.00 | 7.54 | NA |
| Oven | 117 | 6.27 | 0.37 | 5.46 | 7.29 | NA |
| NIR | 117 | 6.28 | 0.29 | 5.53 | 7.07 | 0.25 |

NA (not analyzed)

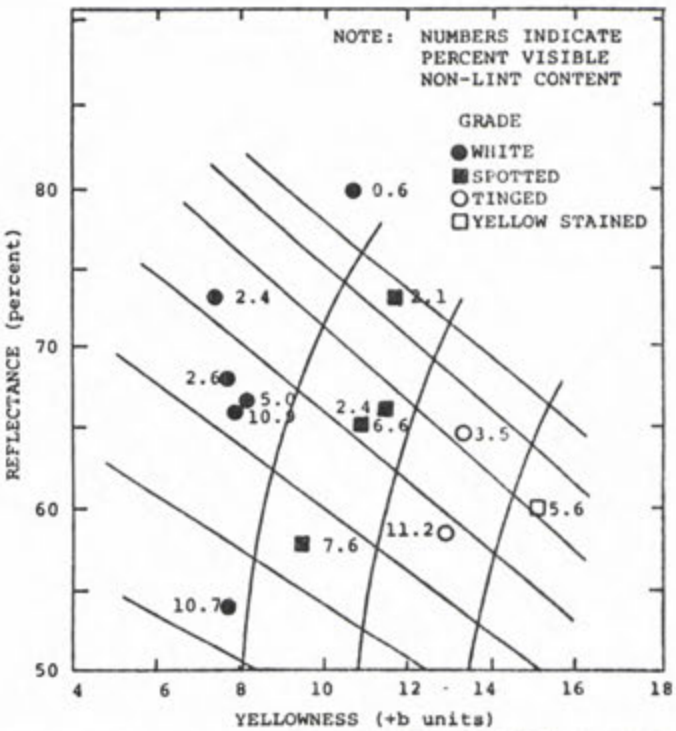


FIGURE 1. COLOR AND NON-LINT CONTENT OF ROUND ROBIN COTTONS.

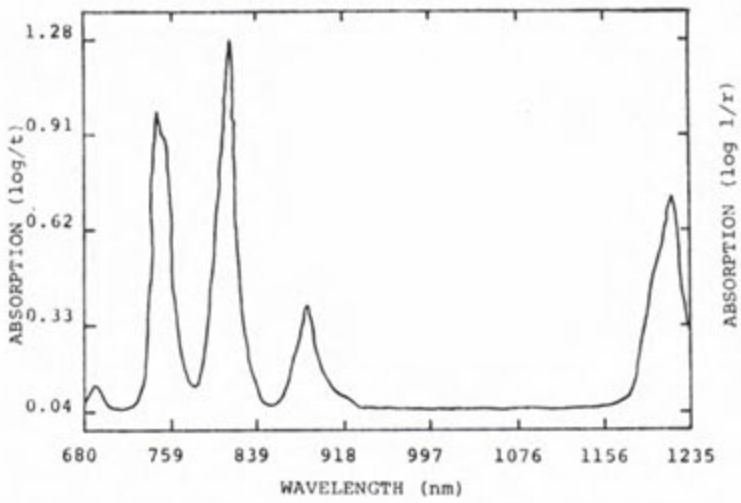


FIGURE 2. SPECTRAL ABSORPTION OF DIDYMIUM FILTER USED TO CALIBRATE THE WAVELENGTH SCALE FOR NEAR INFRARED REGION I.

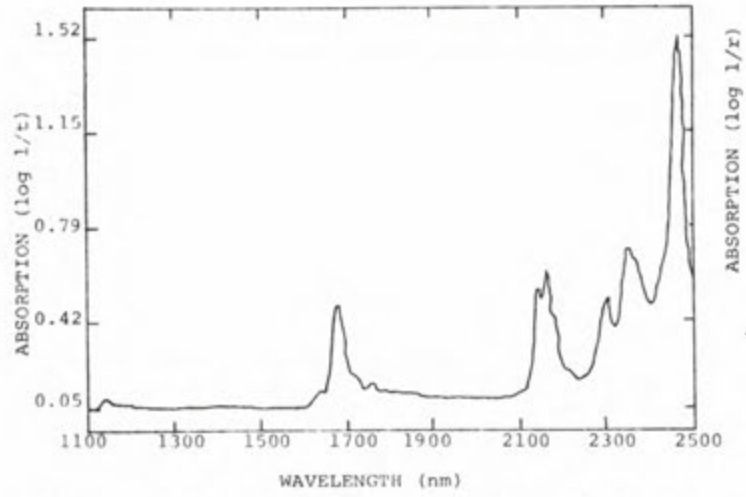


FIGURE 3. SPECTRAL ABSORPTION OF POLYSTYRENE FILTER USED TO CALIBRATE THE WAVELENGTH SCALE FOR NEAR INFRARED REGION II.

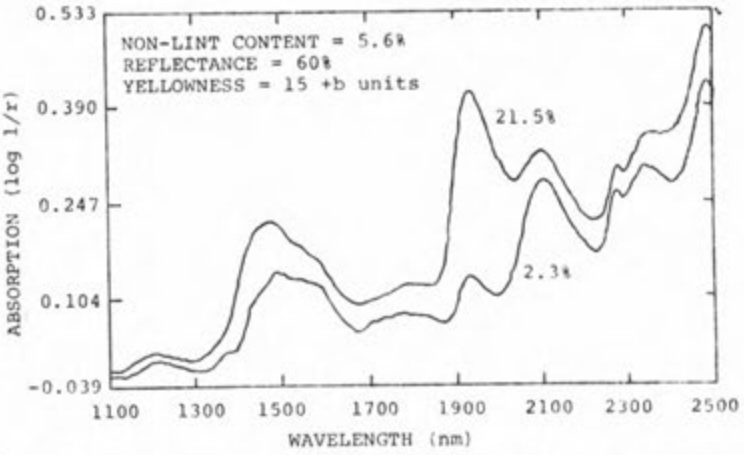


FIGURE 5. SPECTRAL ABSORPTION IN NEAR INFRARED REGION II FOR A YELLOW STAINED COTTON AT TWO LEVELS OF MOISTURE CONTENT.

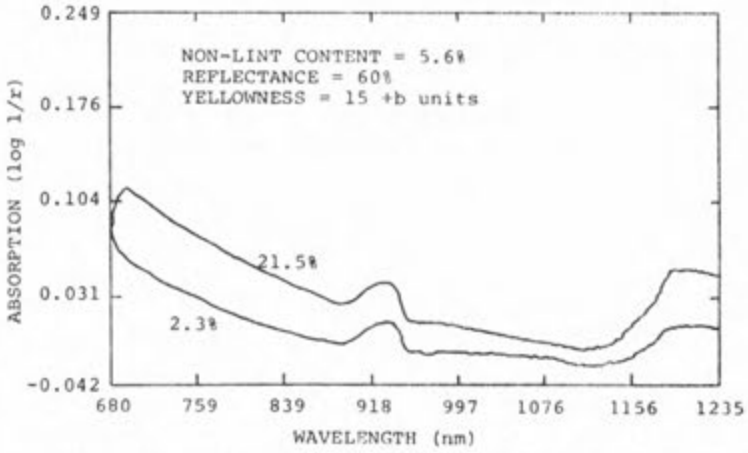


FIGURE 4. SPECTRAL ABSORPTION IN NEAR INFRARED REGION I FOR A YELLOW STAINED COTTON AT TWO LEVELS OF MOISTURE CONTENT.