

INFRARED REFLECTANCE SPECTROSCOPY FOR ESTIMATION OF MOISTURE OF WHOLE GRAIN

R. A. STERMER¹, Y. POMERANZ, and R. J. MCGINTY², U.S. Grain Marketing Research Center, Agricultural Research Service, U.S. Department of Agriculture, Manhattan, KS 66502

ABSTRACT

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A spectrophotometer employing the tilting-filter wheel principles developed by Karl Norris in the early 1970's was used to study the infrared (IR) reflectance properties of whole-kernel corn and sorghum grain. Reflectance properties were compared with oven moisture content to determine the feasibility of using IR spectroscopy for rapid estimation of the moisture content of whole grain. Spectral reflectance, at two selected groups of wavelengths, was measured with the tilting-filter wheel instrument. The IR reflectance readings were compared with oven moisture analyses by use of multiple regression analysis. Correlation coefficients were 0.959 for sorghum grain and 0.993 for corn for samples ranging in moisture content from 13 to 55%. Results were similar ($R = 0.997$ and 0.974 for

corn and sorghum, respectively) from a commercial IR instrument (Neotec-GQA-41). For native wet samples, standard errors of the estimate ranged from 0.8% for corn to 3.4% for sorghum grain. Electric moisture meters were also used to estimate the moisture contents; standard errors of estimate were reduced by applying a power curve regression analysis ($y = ax^b$) to the data since the readings were nonlinear above about 30%. However, in general, the standard errors of the estimate were higher for the electric moisture meters than for the IR methods. The IR method shows promise for rapid estimation of the moisture content of high-moisture whole grain. Certain modifications in the instrumentation are suggested for improvement of results.

During the early 1960's, the Instrumentation Research Laboratory (IRL), Agricultural Research Service, USDA, Beltsville, Md., under the direction of Karl H. Norris, applied near-infrared spectroscopy to determine moisture content of ground grain (1). That technique was later expanded to include the determination of oil and protein. In 1971, commercial instruments using the principles developed by IRL were introduced to the grain industry (2).

The early versions were designed primarily to measure the oil content of soybeans. Since then, both Neotec Instruments, Inc. and Dickie-john Corp. have extended the scope and capability of the infrared (IR) instruments to encompass a number of grain constituents. The basic operating principles and procedures were described by Trevis (3), and detailed instructions are provided in operators' manuals.

The IR method has been tested extensively for measurement of oil content of soybeans and protein content of wheat (4), and to a limited degree for measurement of moisture in grain over the range of 8 to 15% normally encountered for grain in storage (5). Electric conductance and capacitance-type instruments are readily available and give acceptable results for that range of moisture contents.

In recent years certain grains, particularly corn, have been harvested at high moisture levels and had to be dried rapidly for safe storage. Therefore, it is

¹Present address: Transportation and Marketing Research, P.O. Box ED, College Station, TX 77840.

²Respectively: Research Agricultural Engineer, Research Chemist, and Agricultural Engineer.

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important to have a rapid method of measuring high moisture levels. Electric moisture meters give rather poor results when the moisture content of grain exceeds 25%. Other available methods such as oven-drying and nuclear magnetic resonance or microwave attenuation are time-consuming or require complex equipment. While it is desirable to measure moisture accurately, the degree of accuracy becomes less important at the high moisture levels.

Precise spectral reflectance measurements require relatively flat and uniform surfaces, which are generally achieved by grinding materials such as grain. Grinding high-moisture grain clogs conventional mills, prevents uniform grinding, and generates heat that causes an unknown loss of moisture. Therefore, we have elected to make reflectance determinations on whole grain, even though the relatively small samples contributed to a major source of variability. Sampling error and loss of moisture during handling increase variability and decrease precision. Thus, it appeared that the accuracy of IR reflectance spectroscopy measurements might be acceptable for the high moisture levels. Furthermore, the increasing acceptance of IR instruments for measurement of protein and oil is an incentive to increase the instrument's use by simultaneously measuring moisture.

The objectives of this study were to determine the feasibility and accuracy of IR spectroscopy for estimation of moisture content in corn and sorghum grain containing 13 to 40% moisture and to compare the results with other available methods.

MATERIALS AND METHODS

Spectrophotometer System

Details of the absorption spectra in the 0.9–2.35 μm range were studied with a research model of the high-speed tilting-filter wheel spectrophotometer (TILTFW) based on principles developed by Karl Norris, IRL, described briefly by Rosenthal (6). The monochromator section consists of a paddle wheel in which three narrow-band pass interference filters can be mounted. Collimated light passes through the filters as they move through an angle of about 35°. Since the transmitted wavelength decreases as the angle of incidence from the normal increases, a portion of the spectrum (about 10% of the nominal wavelength) can be scanned with each filter. For example, a filter with a nominal wavelength of 1.0 μm can be used to scan the spectrum from about 0.9 to 1.0 μm . Filters with peak transmittances at 1.0, 1.15, 1.30, 2.0, 2.15, and 2.30 μm permitted scanning from 0.90 to 1.30 μm and from 1.80 to 2.30 μm . Later, a filter peaked at 1.50 μm was obtained so that the H₂O absorption band at 1.45 μm could be scanned. Most of the principal water-, protein-, and oil-absorption bands are in that region. The monochromatic radiation strikes the sample contained in a glass-bottom cell and is reflected back to a unique arrangement of lead-sulfide photodetectors. The analog signal is amplified and converted to a digital signal. An appropriate logic circuit interfaces the spectrophotometer section to a minicomputer. Data are transferred into the memory of the computer by the combined action of an optical encoder coupled to the filter paddle wheel, the logic interface, and "software" program. The instrument reads relative reflectance at approximately 110 wavelengths for each filter, or a total of 330. Resolution is on the order of 1 nm, which permits the detection of minute

differences in the absorbance for samples. A commercial spectrophotometer similar to the research model has been described in detail by Rosenthal *et al.* (7).

Grain Quality Analyzer and Electric Moisture Meters

A Model 41 Grain Quality Analyzer (GQA-41) manufactured by the Neotec Corp. was used to measure native moisture content of grain samples described below. Procedures outlined in Section 2 of the operator's manual were followed, except that the spring compressor used to press ground samples against the window of the sample cell was not used. One layer of corn kernels was placed with the kernels' large flat area against the window to minimize shadows from the relatively large granular material. Sorghum grain was poured into the sample cell with random placement of the kernels. These procedures of kernel placement were also used with the TILTFW instrument sample cell.

The GQA-41 has five "product" settings. Each setting provides reflectance readings at specific wavelengths where the constituent being measured influences the relative reflectance. For example, product 1, specified by the manufacturer for nonprocessed, non-oil bearing grain, emphasizes the influence of H₂O. Four dR/R readings, recorded during calibration of the instrument, are related to the compositional components as follows: the first two for the H₂O absorption band at 1.94 μm ; the third for the protein absorption band at 2.18 μm ; and the fourth for the oil absorption band at 2.305 μm . Thus, four dependent variables were subsequently correlated with oven moisture content. The dR/R results varied presumably from measuring IR reflectance of a small total area for a few kernels. Consequently, the following procedure was used: the cell was filled, the four dR/R readings were taken, the cell was rotated 180°, and four additional readings were taken. After the cell was emptied and refilled, the above steps were repeated. Thus, four observations were recorded for each of the four dR/R readings, and were subsequently compared with oven moisture contents by means of linear multiple-regression analysis.

Moisture was also measured on native wet corn and sorghum with four electric moisture meters. Two of the meters were Motomco Model 919, one was a Steinlite Model RCT, and one a Burrows Model 700. All were calibrated by the procedures recommended by the respective manufacturers. Three independent readings were taken on each sample with each of the instruments. The means were calculated and compared with the corresponding oven moisture contents.

Rewetted Grain

The study was begun prior to the harvest season to determine the general spectral absorption characteristics of grain at various levels of moisture. Several lots of corn and sorghum grain (Groups 1 and 2, Table I), ranging in moisture from 11 to 15% as-is basis (wb), were rewetted to raise moisture contents from 16 to 37% in increments of about 3%. The rewetted samples were equilibrated at about 5°C for 7 to 21 days. Samples were stirred at least once daily to prevent caking.

Native High-Moisture Grain

Corn and sorghum grain were hand-harvested at intervals from the time the grain had reached the soft-dough stage until maturity. For the initial test with native wet grain, corn was harvested and air-dried to the desired moisture levels

(Group 3, Table I). That corn was very immature and shriveled when dried to relatively low moisture levels. Therefore, for subsequent tests, the sorghum grain was selectively harvested to give a range in maturity and moisture contents (Group 3, Table I). The samples were then blended to provide subsamples with the desired range in final moisture content. For the final tests (Group 4, Table I), both corn and sorghum grain were selectively harvested and blended. All samples were equilibrated at 5°C for 1–3 weeks.

Moisture contents of samples (about 20 g) of corn were determined by single-stage heating in an air oven at $103^{\circ} \pm 1^{\circ}\text{C}$ for 72 hr (8). The oven moisture contents of sorghum grain were determined by our procedure, which compares favorably with the AACC two-stage air-oven method (8); samples (about 20 g) were dried by a single-stage heating in an air oven for 19 hr at $120^{\circ} \pm 1^{\circ}\text{C}$.

Sorghum was threshed by hand to minimize kernel damage. Hand threshing of immature sorghum was difficult, so we developed a rub-board procedure. One board, 22×30 cm, was used as a stationary surface; another one, 17×22 cm, and equipped with a handle, was used as the movable rub-board. Both boards were covered with corrugated rubber.

TABLE I
Types of Grain, Treatment, and Number of Samples for Moisture Tests

Sample Group No.	Kind of Grain	Conditioning Treatment	No. of Lots	No. of Sub-samples	Initial Moisture Content %	Range in Final Moisture Content %
1	Corn	Rewetted	1	8	11 to 15	16 to 37
2	Corn	Rewetted	4	32	11 to 15	16 to 37
	Sorghum	Rewetted	2	16	13 to 15	16 to 37
3	Corn	Dried	5	45	59 to 61	14 to 40
	Sorghum	Blended	1	26	25 to 42	25 to 42
4	Corn	Blended	1	14	14 to 47	14 to 47
	Sorghum	Blended	1	26	13 to 55	13 to 55

TABLE II
Wavelengths Used for Moisture Determination of Native Wet Grain

Group	Approximate Wavelength μm	Absorption Band
A	0.97	H ₂ O
	1.09	Near protein
	1.45	H ₂ O
	1.50	Near H ₂ O
B	1.89	Near H ₂ O
	1.96	Near H ₂ O
	2.12	Near starch
	2.34	Starch and oil

All samples were allowed to warm to room temperature in sealed jars to avoid condensation before moisture was measured.

RESULTS

Studies with Rewetted Grain

In preliminary studies on corn from Group 1 (Table I), relative reflectance at $0.970\ \mu\text{m}$ (a water absorption band) was highly correlated with oven moisture content ($r = -0.993$ and $s_{y,x} = 1.13\%$). In subsequent tests with several lots of rewetted corn and sorghum grain (Group 2, Table I), that single reflectance band at $0.970\ \mu\text{m}$ was inadequate, since there were serious interactions (probably chemical compositional differences associated with kind and variety of grain). Three reflectance bands were selected: 1) $1.94\ \mu\text{m}$, an H_2O /absorption band, 2) $2.19\ \mu\text{m}$, adjacent to the protein absorption band at $2.18\ \mu\text{m}$, and 3) $2.33\ \mu\text{m}$, a spectral region where both starch and oil absorb radiation. The relative reflectances at those three wavelengths, when used in a multiple regression equation, provided an estimated moisture that correlated highly with oven moisture content ($R = 0.966$). The standard error of the estimate ($s_{y,x} = 2.0$) was higher than desired. Some of the samples had molded, which could have contributed to variability in the IR spectral reflectance. Probably another source of variability was the altered texture and surface characteristics associated with rewetting the grains.

Native Wet Grain

Use of the three wavebands, previously established for rewetted grain, on the native wet grain (samples from Group 3, Table I) gave poor results ($s_{y,x} = 2.5\%$ for sorghum). All lots of corn were immature, and the corn shriveled when dried, a probable source of error.

A detailed study of the spectral reflectance characteristics of native wet corn and sorghum grain at wavelengths from 0.9 to $1.5\ \mu\text{m}$ and from 1.8 to $2.3\ \mu\text{m}$ indicated that either group A or B of four wavelengths (Table II) selected from the above two ranges would adequately measure moisture content and compensate for interactions from other constituents. The relative reflectance on the slopes of some of the absorption peaks provided more sensitive readings than the reflectance readings directly on the peak.

Both groups of wavelengths produced results (Table III) similar to those for samples from Group 4, Table I. The multiple correlation coefficients for TILTFW-estimated moisture vs. oven moisture were somewhat lower for sorghum grain than for corn. Despite the improved threshing procedure, a large number of stems and glumes that adhered to sorghum grain kernels may have contributed to the lower correlation coefficients. This problem would be reduced with combine harvested grain.

The GQA-41 gave slightly better results than the TILTFW (Table III). Four readings were averaged for the GQA-41 and only three for the TILTFW instrument; that difference could account for the higher accuracy obtained with the GQA-41.

Electric Moisture Meters

In general, results were less accurate from electric moisture meters than from

the IR methods except for results from the Steinlite meter on sorghum (Tables III and IV). The differences in accuracy were particularly apparent above 25% moisture. The deviations from oven determinations increase exponentially with increases in moisture content above about 30%. A regression analysis based on a power curve of the form $\hat{Y} = ax^b$, where x is the electric moisture meter reading and \hat{Y} is the predicted moisture content, compensated for the nonlinearity above 25%. The correlation coefficient and standard errors of the estimate shown in Table IV were obtained by use of the power equations derived by regression analyses.

TABLE III
Summary of Regression Analyses—Native Wet Grain

Instrument	Kind of Grain	Range in Moisture Content %	Multiple Correlation Coefficient ^a	$S_{y,x}$ %
TILTFW (A) ^b	Corn	14.2 to 46.8	0.993	1.30
	Sorghum	13.1 to 54.5	0.959	3.20
	Corn and sorghum ^c	13.1 to 54.5	0.979	2.16
TILTFW (B) ^b	Corn	14.2 to 46.8	0.991	1.44
	Sorghum	13.1 to 54.5	0.951	3.42
GQA-41	Corn	14.2 to 46.8	0.997	0.80
	Sorghum	13.1 to 54.5	0.974	2.50

^aCorrelation coefficients are for IR readings vs. oven moisture contents.

^bWavelength group shown in Table II.

^cData combined.

TABLE IV
Summary of Results—Electrical Moisture Meters—Wet Grain

Meter	Kind of Grain	Range in Moisture Content %	Regression Equation Power Curve	r	$S_{y,x}$ %
Motomco #1	Corn	14.6 to 47.4 ^a	$y = 0.128x^{1.613}$	0.966 ^b	3.03
Motomco #1	Sorghum	13.1 to 42.2	$y = 1.079x^{0.999}$	0.819	4.89
Motomco #2	Corn	14.6 to 47.4	$y = 0.441x^{1.255}$	0.971	2.77
	Sorghum	13.1 to 42.2	$y = 0.889x^{1.061}$	0.886	3.96
Steinlite RCT	Corn	14.6 to 34.4	$y = 0.273x^{1.431}$	0.988	1.04
	Sorghum	13.1 to 33.2	$y = 0.555x^{1.203}$	0.990	0.79
Burrows 700	Corn	14.6 to 44.4	$y = 0.712x^{1.040}$	0.949	2.87
	Sorghum	13.1 to 39.4	$y = 2.000x^{0.775}$	0.936	2.68

^aHighest moisture level that could be measured with the instrument specified.

^bCorrelation coefficients are for adjusted electric moisture meter readings vs. oven moisture content.

The reader is cautioned that the results obtained for the electric moisture meters are for particular meters. Other meters of the same make and model might give somewhat different results.

DISCUSSION

The infrared method of measuring the moisture content of whole grain appears to be promising, particularly in the upper range of moisture where electric moisture meters are unreliable. The results from the GQA-41 are particularly encouraging because rapid and reasonably accurate results can be obtained without grinding the grain. Further research to reduce the standard error of the estimate is warranted. We believe that the sampling error is excessive because of the small number of kernels viewed by the IR devices and because of the irregular surface of the kernels. Reflectance spectroscopy gives best results when a reflective flat uniform surface is measured. Incorporation of an optical integrating sphere would reduce the error associated with both of the above problems. Also, wavelengths other than those we used might be optimum. Further research is needed to verify those theories.

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