Determination of Protein and Moisture in Wheat and Barley by Near-Infrared Transmission

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The Trebor GT-90 is an instrument that measures protein and moisture in wheat and other grains by transmission spectroscopy in the near-infrared area of the spectrum between 900 and 1050 nm through the intact kernels. It accepts a large sample (150–250 g), and the time per test is about 45 s. The GT-90 has been evaluated for the testing of wheat and barley for protein and moisture and by comparison with the Kjeldahl protein and two-stage air oven moisture procedures, and with commonly used near-infrared reflectance instruments, the accuracy and precision are sufficient for the testing of both grains for protein identification and segregation purposes. The effects of several variables on the accuracy of testing have been studied. These include temperature of the sample, tempering, foreign material, seed size, and growing season.

The Trebor GT-90 was introduced in Oct 1980 as the first commercial instrument capable of performing analysis of whole wheat for protein and moisture, without pregrinding the grain. Earlier papers had indicated the feasibility of estimating protein (Stermer et al., 1977) and moisture (Tkachuk, 1981) in intact wheat kernels. Both of these reports concern reflectance spectroscopy. The GT-90 uses transmission spectroscopy in the near-infrared area but at shorter wavelengths than those used by near-infrared reflectance instrumentation, and a silicon detector is used in place of the lead sulfide detector.

The GT-90 uses the principle of near-infrared transmission and employs wavelengths in the region of 900–1050 nm. Energy comes from near-infrared light emitting diodes (NIRLED), and 12 narrow band-pass filters (1 for each NIRLED) complete the optical system. All 12 NIRLEDS are employed in the measurement of protein and moisture. Light at discrete wavelengths passes through the sample and is received in the form of log $(1/T)$ (apparent transmission) by a silicon detection system from which it is amplified and transposed into the second derivative of log $(1/T)$ for computation of protein and moisture, by a microprocessor. A large sample (150–200 g for wheat and 125–150 g for barley) is poured into the hopper at the top of the instrument. This is subsampled by a programmable incrementing device that normally takes 21 small subsamples, each of which are read separately by the instrument.

The residual grain is then swept through into a receiver, and the instrument averages the readings and displays the result. Values are given for moisture, protein "as is" and at constant (programmable) moisture, and dry matter. A system was developed by using an interfaced microcomputer, which enabled the generation of calibrations with large numbers of samples. This led to a significant improvement in the precision and accuracy of analysis. A separate feature of the instrument enables the operator to evaluate and correct differences between calibration slopes and standard data. This paper gives background information on the technology and its development and also presents data for the accuracy and precision of analysis of wheat and barley for protein and moisture. The influences of several variables are discussed. These include grade, temperature, dockage, kernel size, growing season, grain type, and tempering.

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MATERIALS AND METHODS

Spectroscopic Studies. Initial spectroscopic studies were carried out by using the Beltsville Universal computerized spectrophotometer (BUCS). This instrument was assembled and is operated at the Instrumentation Research Laboratory at the USDA Research Centre, Beltsville, MD. It consists of two Cary single grating monochromators, with wavelength capability from 200 to 2600 nm. The BUCS can operate in transmission or reflectance modes, and data are stored and processed with a minicomputer with memory capacity of 128 kbytes. Spectra were recorded for all individual samples from 800 to 1100 nm, an area of the near-infrared where the silicon detector operates at high efficiency. The log $(1/T)$ data were transposed into the second derivative of the log $(1/T)$ for data reduction and wavelength selection using the algorithm $(2A - B - C)$. The curve-fitting procedure was also employed (Hrushka and Norris, 1982). The spectroscopic studies were limited to wheat.

Samples of hard red spring (HRS), hard red winter (HRW), soft white spring (SWS), soft white winter (SWW), soft red winter (SRW), white club (WC), Western white (WW), semidwarf (SD), and durum wheats were accumulated from several areas via USDA Agricultural Research Centers. These, together with 100 HRS samples provided by the Canadian Grain Commission, were used in the computerized spectrophotometer (BUCS) study. Samples (20 g) were ground in a Udy Cyclone sample mill and analyzed for protein by the Kjeldahl procedure (AACC, 1969a) and moisture by an air oven technique (AACC, 1969b). Moisture in the whole kernels was determined by the Motomco moisture meter, and protein was recalculated to the basis of whole kernel moisture. The samples used in the Beltsville study were divided into two series, one for calibration and the other for prediction. These are summarized in Table I. The BUCS was used to scan the samples in transmission mode by using a silicon detector. A circular stainless steel cell 4.5 cm in diameter and 2.1 cm deep was fabricated with a glass bottom (Figure 1). The cell was filled with wheat and "struck" level, to provide a consistent thickness of grain.

For the GT-90 studies, samples of wheat representing 10 growing seasons and many growing areas in Canada included HRS, HRW, SWW, SWS, and SRW types. Barleys (two row and six row) were compiled from Western Canada over three seasons. Some European barleys were

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Table I. Wheat Samples Used on BUCS Study

class	HRS	HRW	SRW	durum	semidwarf	Western white	white club	soft white
				Calibration				
no. of locations	3	7	6	3	1	4	3	4
no. of samples	51	52	49	53	50	49	48	52
mean protein	14.7	12.5	11.8	13.9	11.8	10.7	9.3	10.5
SD protein	1.10	1.17	0.75	1.16	1.66	1.37	1.44	1.57
mean moisture	10.3	10.0	11.2	10.4	9.1	9.3	8.6	9.9
SD moisture	0.74	1.17	1.24	0.90	0.49	0.78	0.87	1.38
				Prediction				
no. of locations	3	7	5	5	1	1	3	4
no. of samples	32	33	30	36	30	31	30	34
mean protein	14.8	12.2	11.8	14.1	11.9	10.5	9.7	10.1
SD protein	0.93	1.11	0.62	1.08	1.89	1.59	1.79	1.31
mean moisture	10.6	10.1	11.0	10.4	9.1	9.4	8.9	9.7
SD moisture	0.98	1.01	0.81	0.78	0.56	0.83	0.99	1.49

Table II. Mean and Standard Deviation of Samples Used in GT-90 NIT Calibration and Prediction of Protein and Moisture

	wheat		barley	
	protein	moisture	protein	moisture
		Calibration		
SD	1.73	1.19	1.13	2.18
mean	13.79	12.69	12.47	12.64
N	278		150	
		Prediction		
SD	2.09	1.18	1.49	1.96
mean	13.57	11.94	12.15	12.71
N	86		75	

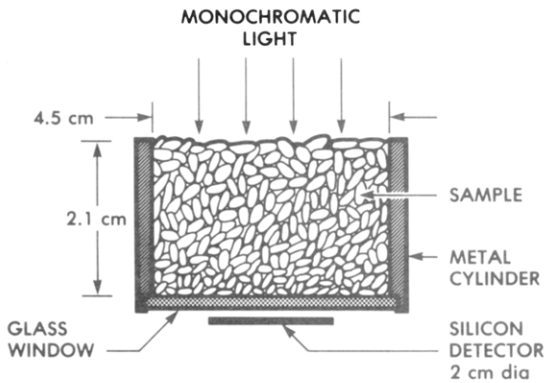

Figure 1. Cell used in transmission measurements by Beltsville Universal computerized spectrometer.

included. Means and standard deviations are given in Table II. These series of samples were distinctly different from those used in the BUCS study. Thousand kernel weight (TKW) was determined by hand counting and weighing duplicate 200-kernel samples. Dockage was determined with a Carter dockage tester, and tempering carried out by adding the requisite amount of water to the wheat, in 4 mil thick plastic bags. The bags were sealed, shaken after 2 and 4 h and overnight, and then stored at 5 °C for 4 days. The samples were then removed from cold storage, agitated again, and left to equilibrate at room temperature before use. For the special tempering–time study, the tempering was carried out in screw-top glass jars by using 300-g samples. Temperatures were attained by leaving samples in 4-mil plastic bags exposed overnight to external temperatures of down to −35 °C in Jan 1981 and 1982, by means of a domestic freezer, a refrigerator, and a forced draught air oven. Throughout all experiments, accuracy was expressed as the standard deviation of differences between GT-90 and standard laboratory results. Precision was evaluated by interposing one of three check samples every fifth test and calculating the standard deviation of these results, following the completion of all experiments.

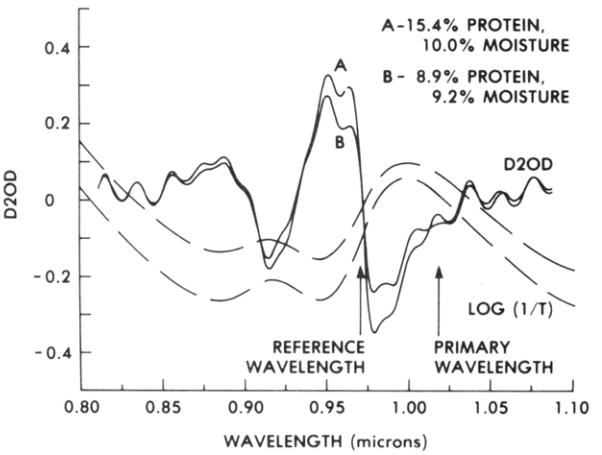

Figure 2. Near-infrared transmission spectra of two wheat samples of different protein contents.

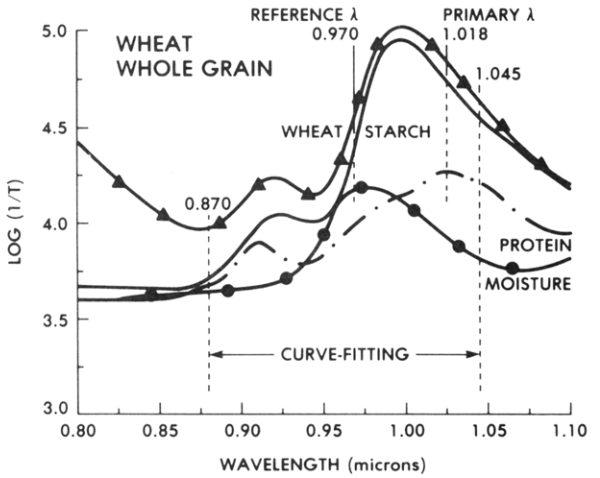

Figure 3. Wavelengths used for measurement of protein in wheat by near-infrared transmission using normalized second derivative and curve fitting.

RESULTS

Computerized Spectrophotometer Analysis of Wheats of Different Types. Figure 2 illustrates typical spectra for two wheat samples differing in protein by 6.5% protein. Figure 3 indicates the primary (1018 nm) and the normalizing or dividing (Norris and Williams, 1984) wavelengths (970 nm) used for the second derivative of log $(1/T)$ [d^2 log $(1/T)$] treatment and for the curve-fitting

Table III. As Is Protein Prediction Errors of One Overall Calibration for All Classes of Wheat

math	SEP, %	bias, %	SD of biases,[a] %
$d^2 \log (1/T)/d^2 \log (1/Tr)$[b]	0.48	0.056	0.40
curve fitting	0.54	−0.035	0.46

[a] Standard deviation of biases for eight individual wheat classes. [b] Tr = reference wavelength.

procedure that employed a wavelength area from 880 to 1045 nm. Multiple linear regression analysis involving several mathematical treatments (Norris and Williams, 1984) revealed that the normalized $d^2 \log (1/T)$ and the curve-fitting mathematical transpositions of the log $(1/T)$ data gave the highest accuracy for testing all types of wheat for protein and moisture. Table III summarizes the results obtained by using a single calibration for all eight classes of wheat. Over 250 individual samples were predicted. The second derivative gave fractionally better predictions for protein. The third column gives the standard deviation of mean deviations from Kjeldahl (biases) for all eight wheat types. Results for individual classes are given in Tables IV and V, using respectively second derivative and curve-fitting treatments. The RPS relates the standard error of performance (SEP) to the standard deviation (SD) of the original data and is given by RPS = SD/SEP. Ideally the RPS should approach a value of 10 or higher. A RPS value that approaches 1 means that predictions are no better than guesswork, since the standard deviations of differences between, e.g., NIR and Kjeldahl data are of the same magnitude as the standard deviation of the original (Kjeldahl) values. Table IV and V indicate that the curve-fitting treatment was slightly superior to the second-derivative treatment for feasibility of analysis of whole wheat kernels for protein and moisture using near infrared transmission.

Trebor GT-90 Analysis of Wheat and Barley. The Trebor GT-90 is a compact low-cost instrument, designed for use in grain elevators, mills, and farms. The instrument employs the same near-infrared transmission principle as was used in the BUCS. The log $(1/T)$ data for the sample is transposed into the second-derivative form before analysis.

Precision. Precision of testing was determined by the repeated analysis of three different check samples, which represented a range of protein and moisture. The overall results are given in Table VI. Precision of protein and moisture testing of both wheat and barley was similar to that of standard analysis. Moisture testing was more precise than protein, and the figures were comparable with those of standard testing. Most of the coefficients of variability were below 2%.

Accuracy. Samples representing a wide range of protein and natural moisture were used for the prediction of protein and moisture in both HRS wheat and barley. Accuracy is reported in terms of the standard deviation of differences between NIT and standard analyses, the

Table IV. Accuracy of Protein Prediction by BUCS: Individual Calibrations for All Wheat Types[a]

class	HRS	HRW	SRW	durum	semidwarf	Western white	white club	soft white
				Curve Fitting				
SD[b]	0.93	1.11	0.62	1.09	1.89	1.59	1.78	1.31
SEP	0.21	0.29	0.23	0.31	0.22	0.29	0.32	0.45
d	−0.02	−0.07	0.05	−0.04	−0.02	±0	−0.01	0.03
RPS	4.4	3.8	2.7	3.5	8.6	5.5	5.6	2.9
				$d^2 \log (1/T)/d^2 \log (1/Tr)$[c]				
SEP	0.32	0.36	0.42	0.29	0.24	0.39	0.42	0.45
d	−0.13	0.04	0.13	−0.17	0.03	0.01	0.05	0.09
RPS	2.9	3.1	1.5	3.8	7.9	4.1	4.3	2.9

[a] Results on "as is" basis with respect to moisture. [b] SD = standard deviation of original data; d = mean deviation (bias); RPS = ratio of prediction standard error (SEP) to standard deviation of original data (SD). [c] Tr = reference wavelength (denominator).

Table V. Accuracy of Moisture Prediction by BUCS: Individual Calibrations for All Wheat Types

class	HRS	HRW	SRW	durum	semidwarf	Western white	white club	soft white
				Curve Fitting				
SD[a]	0.98	1.01	0.81	0.78	0.56	0.83	0.99	1.49
SEP	0.35	0.22	0.43	0.24	0.17	0.31	0.42	0.62
d	0.05	−0.01	−0.12	−0.04	0.06	−0.01	−0.07	0.10
RPS	2.8	4.6	1.9	3.3	3.3	2.6	2.4	2.4
				$d^2 \log (1/T)/d^2 \log (1/Tr)$[c]				
SEP	0.29	0.16	0.45	0.23	0.12	0.32	0.35	0.32
d	0.03	0.20	−0.02	±0	0.03	0.01	−0.04	0.01
RPS	3.4	6.3	1.8	3.4	4.7	2.6	2.8	4.7

[a] Results on "as is" basis with respect to moisture. [b] SD = standard deviation of original data; d = mean deviation (bias); RPS = ratio of prediction standard error (SEP) to standard deviation of original data (SD). [c] Tr = reference wavelength (denominator).

Table VI. Precision of Analysis of Wheat and Barley for Protein and Moisture by GT-90

	wheat				barley			
	standard		NIT		standard		NIT	
	protein	moisture	protein	moisture	protein	moisture	protein	moisture
SD	0.16	0.06	0.17	0.07	0.20	0.18	0.24	0.11
mean	13.86	13.40	13.79	13.30	10.84	14.65	11.00	14.60
CV[a]	1.15	0.44	1.23	0.53	1.85	1.22	2.18	0.75

[a] Coefficient of variability, %.

Table VII. Accuracy of Analysis of HRS Wheat and Barley for Protein and Moisture by GT-90

	wheat		barley	
	protein	moisture	protein	moisture
SEP[a]	0.26	0.20	0.33	0.24
d	0.03	0.0	0.06	0.08
r	0.98	0.97	0.96	0.99
RPS	6.7	6.0	3.4	9.1

[a] SEP = standard error or performance = standard deviation of differences between standard and NIT analyses; d = mean difference; r = coefficient of correlation; RPS = ratio of standard error of prediction to standard deviation of original standard analytical data.

mean differences, and the coefficient of correlation. The results are summarized in Table VII. The accuracy of testing wheat for both protein and moisture was satisfactory for segregating the wheat into sublevels of different protein content commercially. Moisture testing was equivalent to that of most moisture meters for both wheat and barley. Correlations between NIT and standard results were all high. Accuracy expressed in terms of standard deviation of differences is affected by the variance of the precision of the test procedure. If a correction for precision is applied by subtracting the variance of the precision of standard testing from that of the total GT-90/standard analytical comparison, the accuracy of testing wheat and barley for protein become respectively 0.19 and 0.23 in terms of standard deviation of differences. The results for moisture testing likewise become 0.21 and 0.18. This compares with standard (Kjeldahl protein and oven moisture) values of respectively 0.11 and 0.15 for wheat (Williams et al., 1983), which are the corresponding values obtained if the correction for the precision of testing is applied. The chief difficulty in expressing the efficiency of NIR or NIT analysis relative to that of standard analysis is that NIR/NIT measurements are based on absorbances caused by N–H stretching vibrations, in areas of the spectrum defined partly by the absorbance of N–H stretching in the peptide link and amino acid side chains and partly by low interference from other absorbers. Standard methods can be evaluated in many cases by "spiking" with known amounts of pure substances. This is not practicable in the case of protein since pure wheat protein is not available. In the case of NIR or NIT analysis, spiking or similar practices are complicated further by the fact that the form in which proteins, amino acids, etc., occur in natural substances is not necessarily the same as that in which they occur in the isolated state and as such their absorbances usually differ. Accordingly, statistical methods are the most practicable method of expressing accuracy and precision of NIR and NIT analysis of foods, feeds, and commodities in general.

Factors Affecting the Accuracy. *Grade (Weathering).* In Canada all grains are graded on the basis of test weight and by visual examination for damage by weather, fungi, and other factors, all of which affect the analysis. No such effects were detected, provided that samples of all grades were included in the calibration. Samples of 1 CW, 2 CW, and 3 CW wheat were all predicted by using the same calibration. The calibration was derived from over 200 samples of 1–3 CW red spring wheat. The standard deviations of difference and mean differences indicated that when analyzed by using calibrations based on one grade alone, the precision of testing was affected, but no consistent biases were noted. Turning to barley, samples for prediction were predominantly grade no. 1 feed, with blue, white, and yellow aleurone, two-rowed, six-rowed, and mixes present. There was no consistent

Table VIII. Influence of GT-90 In-Built Temperature Correction on Accuracy of Analysis of Wheat

	temperature uncorrected		temperature corrected		temperature,[a]
	protein	moisture	protein	moisture	°C
SEP	2.84	0.49	0.34	0.26	0.14
$\bar{d}$	0.18	0.37	0.04	−0.07	0.40

[a] GT-90 temperature sensing of sample.

Table IX. Typical Results for Analysis of Wheat in the Presence and Absence of GT-90 In-Built Temperature Correction

temperature, °C	corrected		uncorrected	
	protein	moisture	protein	moisture
−14	14.2	10.8	18.4	9.3
−5	14.0	10.6	17.0	9.8
4	14.3	10.6	16.0	10.1
15	14.3	10.5	14.8	10.4
26	14.3	10.4	13.0	10.7
36	14.4	10.2	11.4	11.0
45	14.4	10.3	9.4	11.2

Table X. Mean Results and Significance of Differences in Means from ANOVA Analysis of GT-90 Dockage Study

dockage, %	GT-90 protein, %	moisture, %
0	13.33[a]	11.27
2.5	13.35	11.24
5.0	13.51	11.22
7.5	13.58	11.34
10.0	13.86	11.25

[a] LSD for protein differences (P = 0.05) = 0.17%. LSD for moisture differences (P = 0.05) = 0.14%.

Table XI. Influence of Small Seeds on Accuracy of Analysis of Wheat Using GT-90

initial reading		reading after cleaning		
protein	moisture	protein	moisture	% small seeds
6.5	12.6	14.2	12.8	17
9.2	13.1	13.6	12.8	14
11.1	12.7	12.9	13.1	9
7.1	11.9	15.1	12.2	21
6.9	14.3	11.7	13.9	17
10.1	13.6	13.9	13.7	16

effect of degrading factors on the accuracy of testing.

Temperature. The GT-90 compensates for temperature both of the sample and of the instrument by applying an internally computed correction to the protein and moisture values, based on simultaneous sensing of the temperatures of both sample and sample sensing chamber during the reading of the sample. Results are presented in Table VIII for the analysis of samples ranging in temperature from −23 to +45 °C in the sample. The ambient temperature varied between 18 and 28 °C. Temperature compensations were adequate both for wheat and barley, in that there was no apparent relationship between sample temperature and test error. In the absence of temperature compensation, the influence of temperature was very marked, and Table IX illustrates the influence of temperatures from −14 to +45 °C on the accuracy of testing HRS wheat.

Dockage. The influence of dockage (foreign material) was studied in HRS wheat, with added dockage reaching 10%. The results of the study are summarized in Table X and XI. Essentially dockage of up to 5% exerted no influence on the accuracy of testing. At over 7.5% dockage began to cause erroneous results. Since the average level of dockage in carlots of HRS wheat arriving at both Thunder Bay and Vancouver is less than 3%, dockage is

Table XII. Influence of Growing Season (1980 Samples) on Accuracy of Analysis of Wheat Using GT-40

	1979 calibration		1980 calibration	
	protein	moisture	protein	moisture
N	200	200	200	200
SEP	0.380	0.352	0.312	0.339
$\bar{d}$	0.040	0.060	−0.070	0.120

Table XIII. Statistical Data Comparing GT-90 and Model 919 (Motomco) Meter Results of Testing Wheat for Moisture as a Function of Time after Tempering

		hours after tempering					
		1	2	4	8	24	48
GT-90	SEP[a]	0.68	0.48	0.46	0.26	0.34	0.16
	d	−0.09	0.27	0.40	0.52	0.46	0.41
919	SEP	1.39	0.72	0.69	0.56	0.40	0.38
	d	2.33	1.44	0.56	0.06	0.48	0.38

[a] SEP = standard deviation of differences between GT-90 or Model 919 and two-stage air oven data; d = mean differences.

Table XIV. Typical Data for GT-90 and Model 919 (Motomco) Testing of Wheat for Moisture at Different Intervals after Tempering

	hours after tempering					
	1	2	4	8	24	48
GT-90	14.3	14.6	14.8	14.9	14.9	14.7
919	16.7	15.7	14.9	14.4	14.8	14.8
2SAO[a]	14.4	14.4	14.4	14.4	14.4	14.4

[a] 2SAO = standard AACC two-stage air oven results.

not likely to exert a significant influence at terminal elevators. At country elevators, dockage may be appreciably higher, and cleaning the sample before analysis is recommended, unless the grain arriving at the elevator is obviously fairly low in dockage. One remarkable feature was the influence of small seeds. Lamb's quarters (*Chenopodium album*), foxtail (*Setaria* sp.), wild buckwheat (*Polygonum convolvulus*), and seeds of similar size caused very variable and usually low protein results. If the level of small seeds reached 10%, or more, results of less than 7% protein were recorded, which changed to 13–14% after cleaning (Table XI).

Kernel Size. This factor was studied in wheat and barley. There was no relationship between kernel size (weight) and the discrepancies between NIT and standard results in wheat (r = +0.23), but a significant correlation occurred between kernel weight and protein discrepancies in barley (r = +0.44) although moisture results were not affected. the implications were that HRS wheat analysis by the NIT instrument was not affected by kernel size, but barley was, and a separate calibration woould be advisable for varieties with large kernels, such as the variety Klages. The NIT protein results tended to run higher than Kjeldahl for samples with large kernels (larger than 43 g/1000).

Growing Season. The GT-90 was calibrated with samples representative of 10 growing seasons and a wide range of growing locations. To test the effect of growing season, a set of samples grown in 1980 were tested on two calibrations, the first using 1979 samples only and the second 1980 samples. Table XII summarizes the results. The accuracy of protein testing improved significantly when the samples were analyzed on the 1980 calibration. Moisture results were not so seriously affected. The comprehensive calibration using samples from all 10 seasons stabilized the accuracy of both protein and moisture testing. The influence of growing location, which was of the same order of magnitude, was also corrected by the comprehensive calibration, which included samples from the wheat growing areas of both Canada and the United States.

Tempering. When wheat is freshly tempered for milling, it is desirable to check that the moisture level of the wheat is correct. This is not possible with moisture meters that depend on either capacitance or resistance until the grain has reached equilibrium with the moisture environment by standing for several hours. The testing of tempered wheat for moisture was commenced by using the GT-90 from 30 min after the addition of water to six different samples of HRS wheat that had been tempered respectively to 17 and 14% moisture. Moisture results were compared to those obtained by the two-stage oven (2SAO) method and by a capacitance moisture meter. The GT-90 would not access samples uniformly at 30 min after tempering, but after 1 h testing was possible. The standard deviation of differences between GT-90 and 2SAO methods was acceptable (Table XIII) and less than half that of the 919 meter that, due to high moisture at the outer layers of the kernels, gave results that were over 2% high. Biases in analysis with the capacitance meter were not easily adjustable, and the accuracy of the meter was not reliable until about 8 h after tempering. These results are summarized in Tables XIII and XIV.

Sampling. For most instruments an evaluation of sampling error is of primary importance. In the case of the GT-90 the sample size is large, and no sample preparation is necessary, other than the removal of excessive foreign material. Testing of grain in elevators usually involves subsampling an original envelope sample or a sample taken in a pail or suitable small receptacle. The GT-90 will accommodate an entire envelope sample, which eliminates sampling error and about $^1/_3$–$^1/_2$ of the sample normally taken from trucks or railcars. The sampling error under these circumstances is essentially the same as the error determined during the study of precision.

CONCLUSIONS

Analysis with the BUCS demonstrated the feasibility of measuring protein in whole grains of wheat by near-infrared transmission. Wavelengths were developed for analysis by using the second derivative of the log $1/T$ and a curve-fitting procedure to resolve the log $1/T$ signals into protein and moisture. There was no significant difference in the efficiency of the two methods of data reduction. The second-derivative algorithm was preferred for the Trebor GT-90 mainly because it was simpler to adapt to a mod-

Table XV. Accuracy of Protein and Moisture Testing by GT-90 and NIR Reflectance Instruments

	Technicon						Dickey-john, GAC III		Pacific Scientific				Trebor, GT-90	
	400[a]		300		200				GQA 31EL		101			
	protein	H_2O	protein	H_2O	protein	H_2O	protein	H_2O	protein	H_2O	protein	H_2O	protein	H_2O
SEB[b]	0.183	0.190	0.275	0.204	0.290	0.191	0.218	0.168	0.244	0.297	0.241	0.243	0.264	0.200
$\bar{d}$	−0.04	0.18	−0.22	0.18	−0.15	0.23	0.01	0.12	0.08	−0.20	0.15	0.23	0.03	0.0
r	0.997	0.975	0.994	0.967	0.994	0.975	0.996	0.986	0.992	0.969	0.995	0.957	0.983	0.971

[a] Best data for two possible calibration procedures used in InfraAlyzer 400, 300, and 200. [b] SEP = standard deviation of differences, d = mean difference, and r = coefficient of correlation between standard (Kjeldahl and oven) and NIR or NIT tests.

estly priced commercial instrument.

The Trebor GT-90 whole grain analyzer was used for the measurement of protein and moisture in whole kernels of HRS wheat and barley. Calibrations were based on a very large number of samples. Protein identification and segregation of HRS wheat into protein sublevels are achieved in Canada by tests carried out at terminal grain elevators by near-infrared reflectance instruments such as the Pacific Scientific GQA31EL or the Dickey-john GAC III. A sample is tested by NIR for protein and moisture during the first minute of unloading a car. The protein figure corrected to 13.5% moisture is used for segregation and is compared statistically with a second NIR test carried out in Winnipeg by the ADA (Williams et al., 1978) subsequently on the official sample that represents the total carload. The standard deviation of differences between the "early" and official series of results is normally between 0.35 and 0.40 with essentially no bias. Table XV illustrates data taken from Tables VI and VII of Williams et al. (1983), which described a comparative study of commercial NIR reflectance instruments. On the basis of the current experiments, the Trebor GT-90 appeared to be satisfactory for the testing of HRS wheat for protein identification and segregation purposes. The NIR reflectance instruments routinely used for these operations are the InfraAlyzer 300 and 200, the GAC III, and the GQA 31EL and 101. These gave statistics of the same order of magnitude as the GT-90. The InfraAlyzer 400 is more sophisticated and is not normally employed in such routine situations as testing carlots of wheat. The standard deviation of differences between GT-90 and standard results was slightly higher than with reflectance instruments and powdered samples, but the elimination of grinding and cell-loading errors and the large sample size, with consequent reduction in sampling error, tend to compensate for the difference in the test accuracy. The overall error of the testing process was maintained at, or slightly improved over, customary levels of system accuracy. Temperature compensation was satisfactory, and errors introduced by season, location, kernel size, grade, and grain type could be compensated for by comprehensive calibration. Samples containing above 5% foreign material should be cleaned before testing, and this is of particular importance if excessive quantities of small seeds are present. The GT-90 can be used for testing of wheat for moisture within 1 h of addition of tempering water. The accuracy of moisture testing by the GT-90 was superior to that of protein testing and on the basis of comparison with the AACC 2-stage air oven method was equivalent to the Model 919 (Motomco) meter.

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Polyhydroxydihydrochalcones as Antioxidants for Lard

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Six polyhydroxydihydrochalcones have been evaluated at 120 °C as antioxidants for lard. Eight of the chemically corresponding chalcones and flavanones have been evaluated under the same conditions. The dihydrochalcones show more antioxidant activity than the corresponding chalcones, just as the polyhydroxydihydrocinnamic acids are more active than the corresponding cinnamic acids. Flavanones are significantly less active than the corresponding chalcones.

In recent studies on polyhydroxy aromatic compounds as antioxidants for edible oils, we have reported on flavones and flavanones (Hudson and Lewis, 1983), isoflavones (Dziedzic and Hudson, 1983a), chalcones and flavanones (Dziedzic and Hudson, 1983b), and phenolic acids and esters (Dziedzic and Hudson, 1984a). With favorably located phenolic hydroxy groups, all these chemical classes exhibit marked antioxidant activity.

Particularly marked activity was noted with certain chalcones and phenolic acids or their propyl esters. Perhaps surprisingly, dihydrocinnamic acids were more active than cinnamic acids, which, in turn, were more active than the corresponding substituted benzoic acids. It therefore became pertinent to enquire whether dihydrochalcones were also more active than the corresponding chalcones. The studies reported here were undertaken mainly to settle this question.

A few dihydrochalcones have been reported to be present in plant material as natural products. The best known is the glucoside phloridzin, which occurs in the bark and leaves of the apple. They are largely confined to the Rosaceae and Ericaceae families (Williams, 1966) and can be regarded as precursors of the polyhydroxyflavones (e.g., phloretin, the aglycon of phloridzin, is the precursor of apigenin).

Certain dihydrochalcones also have interesting organoleptic properties. Neohesperidin dihydrochalcone is potentially important as an artificial sweetener (Horowitz and Gentili, 1963).

MATERIALS AND METHODS

As the substrate for the oxidation studies pure dry rendered lard, free from additives and not chemically processed, was used.

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