

Near-Infrared Reflectance Analysis of Bread¹

KENJI SUZUKI, C. E. McDONALD, and B. L. D'APPOLONIA²

ABSTRACT

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The possibility of analyzing fresh bread for constituent content or quality factors by the near-infrared (NIR) reflectance method was investigated. Laboratory analyses of two loaves of bread showed moisture was highest at the center of the slice, whereas protein and lipid content (dry basis) were evenly distributed throughout the loaf. Thus, constituent content of a loaf on dry moisture basis could be calculated from NIR-determined moisture

and "as is" constituent content measured on center disks cut from fresh bread slices. NIR calibrations for moisture, protein, sugar, and lipids were developed and tested on center disks cut from fresh slices of white bread. Statistics indicated moisture and protein could be accurately determined by NIR reflectance, whereas the sugar and lipid analyses were less accurate.

The use of near-infrared (NIR) reflectance analysis for determining protein and moisture in grain has been widely accepted. More recently, NIR analysis has been shown to be feasible for determining a number of other constituents in food products.

Usually samples are finely ground before presentation to the NIR instrument; bread for example would be ground after air drying. However, Osborne et al (1983a) reported on the use of NIR reflectance to directly analyze biscuit and biscuit dough without drying and grinding. Osborne et al (1984) also published on the NIR determination of protein, fat, and moisture in fresh slices of English white bread.

In the present study the feasibility of using NIR reflectance for the analysis of bread without prior drying and grinding was investigated. Bread constituents or quality parameters investigated were moisture, protein, total sugar, and crude fat.

MATERIALS AND METHODS

Flour Samples

A wheat composite from hard red spring (HRS) wheats collected in a four-state survey in 1982 and Waldron variety wheat were milled on an experimental Buhler mill; the protein content of the flours was 13.1 and 13.9%, respectively. Commercial wheat gluten and starch from Henkel Corp. (Keokuk, IA) was used for adjusting protein content of flour.

Baking Methods

One-pound loaves of white bread were baked by straight-dough, sponge-dough, and no-time-dough procedures using the baking formulas shown in Table I.

Protein in flour was varied by adding wheat gluten or starch to the flour. In a completely randomized block design, nine levels of flour protein from 8 to 16% were selected. Then for each level of protein, nine levels of sugar (1-9% of the flour) and nine levels of shortening (1-5% of the flour) were randomly selected for each

TABLE I
Baking Methods for White Bread Loaves

Method/Ingredient	Amount (%)
Straight dough	
Flour + gluten or starch	100.0 ^a
Salt	2.0
Yeast	2.5
Shortening	1.0-5.0
Sugar	1.0-9.0
Water	50-70
Sponge dough	
Sponge stage	
Flour + gluten or starch	60 ^a
Yeast	2
Water	42
Dough stage	
Flour + gluten or starch	40
Shortening	1.0-5.0
Sugar	1.0-9.0
Water	15-23
Salt	2
No-time dough	
Flour + gluten or starch	100.0 ^a
Yeast	3.0
Salt	1.5
Sugar	3.0
Shortening	2.0-5.5
Absorption	60-65
Ascorbic Acid	50 ppm
KBrO ₃	20 ppm
L-Cysteine	5 ppm

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²Project research assistant and professors, respectively, Department of Cereal Science and Food Technology, North Dakota State University, Fargo 58105. Present address of Mr. Suzuki: 20-4 Meieki Z-chome, Nishi-ku, Nagoya, Aichi, 451, Japan.

protein level. In a factorial design, used only with the composite flour baked by the straight-dough procedure, the protein, sugar, and shortening were added at high, medium, and low levels in all combinations. The high levels were 14.0% flour protein, 7.0% sugar, and 5.0% shortening (sugar and shortening on flour basis [fb]); medium levels were 12.0% flour protein, 5.0% sugar (fb) and 3.0% shortening (fb); and low levels were 10.0% flour protein, 3.0% sugar (fb) and 2.0% shortening (fb). Moisture in bread was varied by baking at 70 and 65% absorption with 12–16% protein flour and at 60 and 55% absorption with 8–11% protein flour.

For air-dried calibrations, whole wheat bread was baked by the straight-dough method from mixtures of whole wheat and Waldron white flour without adding wheat gluten and starch to vary the protein content.

NIR Calibration

Bread slices and disks cut from bread slices were stored in sealed plastic bags to prevent moisture loss. Also, crumb disks were cut and were placed in or removed from the NIR instrument cell within a large plastic bag containing a wet sponge. In preliminary work, we found that bread crumb lost 0.22% moisture per minute when left out in the laboratory.

After the baked bread was cooled, the loaf was sliced with a commercial slicer into alternating 12- and 13-mm thick slices. Then three slices from near the middle of the loaf, from near one-fourth in from the end of the loaf, and from near the end were immediately placed in sealed bags for the random block experimental design. For the factorial experimental design slices from near the middle of the loaf and from near the end of the loaf were placed in sealed bags. With a special cutter (3.6 cm in diameter) made by sharpening the end of a sink drain pipe (solder joint end), a disk large enough to fill the viewing field of the InfraAlyzer cell was cut from the middle of the bread slice and placed in the NIR instrument cell. The sample cell was inserted into an InfraAlyzer 400R instrument (Technicon, Terrytown, NY), and reflectance values obtained were transferred to a printer in early work and in later work to a diskette in an Apple II-6502 computer (Apple Computer Inc., Cupertino, CA) containing a Data Capture 4.0 communication card (Southeastern Software, New Orleans, LA). The data were later transferred to the North Dakota State University campus computer for regression analysis.

In one baking experiment slices from loaves of whole wheat bread were air dried at room temperature, and then they were ground on a Wiley mill through a 20-mesh sieve. Three replicate sets of reflectance values from each loaf were measured on the ground bread.

After transferring sets of light reflectance energies at 18 wavelengths to the North Dakota State University campus computer, constituent values were added through a computer terminal, and the data were analyzed by programs of the Statistical Analysis System (SAS Institute Inc., Cary, NC). To obtain calibration equations, laboratory values (protein, moisture, sugar, and lipids) were subjected to regression analysis on the reflectance energies to obtain the regression constant for the smallest and best fitted equation of the following form:

$$\text{Laboratory value} = K_0 + K_1 L_1 + K_2 L_2 \cdots K_n L_n,$$

where K_n = constant from regression for each wavelength, L_n = $\log I$ over R (relative reflectance of sample compared to a standard) for each wavelength, and K_0 = intercept constant from regression.

Wavelength combinations with the highest correlations and the least number of filters were first selected by the R^2 procedure. The linear regression procedure was run with the best wavelength combinations and residuals were calculated. In a few cases one outlier (sample with residual more than three times the standard deviation) was eliminated, and the R^2 procedure was repeated and followed by regression analysis with the best filter combination to obtain final regression equations. The criteria for selection of final calibration equations were high correlation, low mean square error, and high Student's t test for all filter coefficients.

To test calibrations, NIR values for constituents were

determined on bread samples not used for the calibration. The standard deviation of prediction between the NIR and laboratory analyses was calculated by the following equation:

$$\text{Standard deviation of prediction (SDP)} = d^2/n,$$

where d = the individual difference between NIR and laboratory results on n number of samples.

Laboratory Analysis on Bread Crumb

The bread crumb disks used for NIR measurements for moisture were analyzed directly for moisture by heating at 130°C for 90 min in a forced-air oven by a method similar to AACC method 44-15A (AACC 1983). These dried cuts were analyzed for crude fat by AACC method 30-20 (AACC 1983). The crumb disks used for NIR measurements of protein were analyzed directly for Kjeldahl protein by AACC method 46-11 (AACC 1983). To prevent foaming during digestion, 1 ml of diluted antifoam (Sigma Chemical Co., Antifoam A, diluted 1:4 with water) and low heat were applied initially.

Bread crumb disks used for NIR measurements of total sugar were analyzed after freeze-drying. The extraction procedure described by Ponte et al (1969) was used with a modification reported by MacArthur and D'Appolonia (1979). Sugars were extracted twice from the sample using the ternary solvent system, and the combined extracts were dried on a rotary evaporator. The phenol sulfuric acid colorimetric method described by Dubois et al (1956) was used to determine the amount of sugar present in the extract. Air-dried ground bread was analyzed for lipid, protein, and moisture by AACC methods 30-20, 46-11, and 44-15A, respectively.

Constituent distribution in 3.6-cm center disks of crumb and slices throughout the loaf was investigated in two loaves of white bread, each sliced on a commercial slicer into 18 slices numbered from 1 at one end to 18 at the other. The center disks and slices were air dried 25 hr and the weight loss was recorded. After grinding in a Wiley mill through a 20-mesh sieve, moisture, protein, and lipid were determined in ground samples by AACC methods 44-15A, 46-11, and 30-20, respectively (AACC 1983). Constituent contents in undried slices and center disks were calculated from the results.

TABLE II
Moisture, Protein, and Lipid Distribution in White Bread^a

Slice No. ^b	Percent Moisture		Percent, Dry Moisture Basis			
			Protein		Lipid	
	Slice	Cut Disk	Slice	Cut Disk	Slice	Cut Disk
1	21.3		14.4		5.5	
2	33.0 ^c	38.2	14.4 ^c	14.4	5.5 ^c	5.4
3	34.3		14.4		5.4	
4	34.6		14.4		5.4	
5	34.8 ^c	40.8	14.5 ^c	14.5	5.4 ^c	5.3
6	34.1		14.4		5.5	
7	34.4		14.4		5.4	
8	34.5 ^c	42.5	14.5 ^c	14.4	5.4 ^c	5.4
9	34.6		14.4		5.4	
10	33.8		14.3		5.3	
11	34.5		14.4		5.3	
12	34.4		14.4		5.3	
13	34.4		14.4		5.4	
14	34.7		14.5		5.4	
15	34.1		14.4		5.4	
16	34.3		14.4		5.4	
17	33.3		14.4		5.4	
18	18.7		14.4		5.8	

^aAll results are averaged from analyses of duplicate loaves.

^bNumbered from 1 at one end of the loaf to 18 at the other.

^cAnalysis on material remaining after removing the 3.6-cm disk from the center.

TABLE III
White Bread Baking Experiments and Range and Mean of Moisture
and Protein in Crumb Disks Cut from Bread

Baking Experiment	Flour	Baking Method ^a	Percent Moisture			Percent Protein (as is basis)		
			<i>n</i>	Range	Mean	<i>n</i>	Range	Mean
A	Composite	Straight	50	40.6–45.6	43.5	50	6.34–8.97	7.68
B	Composite	Straight	50	39.5–47.1	43.8
C	Waldron	Straight	50	39.8–46.5	43.2	50	5.85–9.59	7.91
D	Composite	Sponge	50	35.3–44.8	41.2	50	6.04–9.46	7.95
E	Composite	No-time	50	40.6–45.6	43.5	50	5.61–9.66	7.78

^a Straight-dough, sponge-dough, and no-time-dough baking procedures.

TABLE IV
Filters and Statistics on Some Water Calibrations
for White Bread

Baking Experiment	Filters, nm			Statistics of Calibration Equation			
				<i>R</i>	SD(%)	C.V.(%)	
A	2,230	2,139	1,982	0.92	0.50	1.1	
A ^a	2,230	2,100	1,940	0.90	0.54	1.2	
B	1,722	2,100	1,445	0.95	0.57	1.3	
B ^a	2,230	2,100	1,940	0.90	0.83	1.9	
C	2,230	2,139	1,940	0.96	0.59	1.4	
C ^a	2,230	2,100	1,940	0.93	0.72	1.7	
D	2,230	2,139	1,982	0.91	0.75	1.8	
D ^a	2,230	2,100	1,940	0.84	0.90	2.3	
E	2,230	2,139	1,940	0.96	0.65	1.6	
E ^a	2,230	2,100	1,940	0.92	0.90	2.2	
Combined	2,230	2,139	1,734	1,445	0.95	0.67	1.6
Combined ^a	2,230	2,100	1,940		0.84	1.17	2.7

^a Filters used by Osborne et al (1984) for water in English white bread crumb.

TABLE V
Testing of Moisture and Protein Calibrations for White Bread

Constituent and Baking Experiment for Calibration	Baking Experiment for Testing	Statistics of Testing Results				Near-Infrared Bias (%)	
		<i>n</i>	<i>R</i>	SDP (%)	C.V. (%)		
Water	A ^a	B	45	0.91	0.75	1.7	+0.58
		C	45	0.92	1.0	2.4	+1.50
	D ^b	A	45	0.92	0.94	2.2	+1.10
		E	45	0.62	1.0	2.4	+0.49
	Combined ^c	A	45	0.93	0.45	1.0	+0.24
		B	45	0.95	0.62	1.4	+0.13
		C	45	0.96	0.62	1.4	-0.06
		D	45	0.96	0.71	1.6	-0.14
		E	45	0.96	0.71	1.6	-0.14
	Protein	A ^d	C	45	0.99	0.30	3.8
D			45	0.97	0.28	3.5	+0.28
E			45	0.99	0.22	2.8	+0.12
Combined ^e		A	45	0.99	0.14	1.8	+0.02
		C	45	0.99	0.24	3.0	-0.02
		D	45	0.96	0.29	3.6	-0.01
		E	45	0.99	0.19	2.4	+0.03

^a Calibration with 2,348, 1,722, 2,190, 1,982, 2,100, and 1,680 nm wavelengths.

^b Calibration with 2,310, 2,270, 2,230, 2,190, 2,139, and 1,445 nm wavelengths.

^c Calibration with 2,230, 1,722, 2,139, 1,940, and 1,445 nm wavelengths.

^d Calibration with 1,722, 2,139, 2,180, 1,778, 2,100, and 1,940 nm wavelengths.

^e Calibration with 1,722, 2,139, 2,180, and 1,759 nm wavelengths.

RESULTS AND DISCUSSION

Constituent Distribution Throughout the Bread Loaf

Constituent distribution was investigated to determine how NIR analysis on crumb disks could be used for determining the constituent content of the entire loaf. The moisture content of the two end slices (1 and 18) was considerably lower than the others, whereas the other slices were fairly constant in moisture (Table II). Because NIR measurements were made on 3.6-cm circular disks from the centers of slices, the moisture content of the center disks was investigated. Three circular disks cut from the center of slices 2, 5, and 8 were considerably higher in moisture than the other slices or the remaining material in a slice, and the cuts varied some between slices.

This uneven distribution, with highest moisture in the center of a loaf, is well illustrated in a paper published by Pence et al (1956). Protein, lipids, or other constituents on an "as is" moisture basis would vary because of the uneven moisture distribution. However, on a dry weight moisture basis, protein and lipids were evenly distributed among slices and between the center cuts of the slice and the material remaining after removing the cut (Table II). Thus, one should be able to determine each constituent in a loaf of bread on a dry weight basis by NIR analysis for moisture and the constituent. Preliminary work (not given) suggests the moisture content of the entire loaf might be calculated from crumb disk moisture by using a regression equation derived from a series of samples of whole loaf and crumb disk moistures.

Calibrations for Crumb Moisture

The baking experiments and range of moisture in crumb disks from slices used for calibrations are given in Table III. Baking experiments A and B were the same but baking was on different days.

Wavelengths and statistics of some water calibrations are given in Table IV. Water filters selected in our study were apparently the 1,445, 1,940 and 1,982 nm ones detecting absorption by water peaks near 1,445 and 1,940 nm. With only three filters, excellent calibrations (low coefficients of variation) were obtained, but a little better calibration was obtained by increasing the number of filters to six (data not shown). The method of baking did not significantly affect the apparent accuracy of the calibration, and the same baking experiment on different days (experiments A and B) gave good calibrations each day but with somewhat different filters. Using the three basic filters at 2,230, 2,100 and 1,940 nm that were used by Osborne et al (1984) the calibrations were not quite as good. Osborne et al reported a standard deviation of 0.46% for moisture, using the average of six replicate readings on a loaf to derive their calibration. They did not report the use of precautions to prevent moisture loss from slices or disks cut from slices during NIR measurements and laboratory analyses.

Calibrations for baking experiments A and D and combined data were tested on bread baked in other experiments (Table V). As expected, the coefficient of variation was usually higher than for calibration data but was still low enough to indicate a good analytical procedure. The bias was also sufficiently low considering the mean percent moisture was around 40.0%. The results also indicated a calibration on bread baked by one baking procedure can be used on bread baked by another procedure. For example,

the calibration of the sponge-dough procedure (bake D) was used to predict moisture in bread baked by the straight-dough procedure (bake A) and the no-time-dough procedure (bake E). Osborne et al (1984) reported an SDP of 0.51% for moisture in English white bread using the average of six replicate NIR moisture analyses on each loaf.

Calibration for Protein

The baking experiments, filters, and statistics for some bread protein calibrations are given in Tables III and VI, respectively. The best three filter protein calibrations selected in our study all contained the protein filter at 2,180 nm plus two reference filters at 2,139 and 1,778 nm. The fairly low coefficient of variation observed for our calibration indicates a good analytical procedure. Osborne et al (1984) reported a standard deviation of 0.09% using four basic filters at 2,180, 2,100, 1,940, and 1,680 nm for crumb protein. Satisfactory calibrations were obtained on our data with these same filters.

Results of testing of protein calibrations from baking experiment A and combined experiments are given in Table V. Satisfactorily predicted proteins were obtained on bread from all of the other baking experiments. Again, the method of baking had little effect on predicting protein.

Lipid Calibration

Lipid calibrations on white bread were also investigated. The number of samples and range and mean in lipid content for each baking experiment are given in Table VII. Lipid calibrations in Table VIII with our selected filters had high correlation coefficients and fairly low standard deviations and coefficients of variation. The coefficient of variation is somewhat higher than for moisture and protein but still should be satisfactory.

The major characteristic absorption bands of fat are caused by long fatty acid chains that give rise to a preponderance of CH₂ absorption broad bands at 1,200 nm (CH₂ stretch second

overtone), 1,734 nm (CH₂ stretch first overtone, asymmetry), 1,765 nm (CH₂ stretch first overtone, symmetry), and 2,130 and 2,345 nm (CH₂ stretch/CH₂ bend combinations; Osborne et al 1983b). In our calibrations, lipid absorption by these bands may have occurred

TABLE VI
Filters and Statistics on Some Protein Calibrations for White Bread

Baking Experiment	Filters, nm				Statistics of Calibration Equation		
					R	SD (%)	C.V.(%)
A	2,139	2,180	1,778		0.98	0.18	2.4
A ^a	2,180	2,100	1,940	1,680	0.99	0.14	1.8
C	2,139	2,180	1,778		0.98	0.23	2.9
C ^a	2,180	2,100	1,940	1,680	0.98	0.24	3.0
D	2,139	2,180	1,778		0.96	0.32	4.0
D ^a	2,180	2,100	1,940	1,680	0.99	0.19	2.4
E	2,139	2,180	1,778		0.99	0.21	2.6
E ^a	2,180	2,100	1,940	1,680	0.99	0.19	2.4
Combined	1,722	2,139	2,180	1,759	0.98	0.23	2.9
Combined ^a	2,180	2,100	1,940	1,680	0.93	0.398	5.0

^aWavelength used by Osborne et al (1984) for protein in crumb of English white bread.

TABLE VII
White Bread Baking Experiments and Range and Mean of Crude Fat and Sugar in Crumb Disks Cut from Bread

Baking Experiment	Constituent	n	Percent	
			Range	Mean
A	Fat	50	1.5-4.4	3.12
C	Fat	49	2.1-4.2	3.15
A	Sugar	50	0.9-5.6	2.76
C	Sugar	49	1.3-4.1	2.52

TABLE VIII
Filters and Statistics on Some Lipid and Sugar Calibrations for White Bread

Constituent and Baking Experiment	Filters, nm				Statistics of Calibration Equation			
					R	SD (%)	C.V. (%)	
Lipid								
A	1,722	1,778	1,680		0.97	0.19	5.9	
A ^a	2,310	2,100			0.89	0.37	11.5	
C	2,348	1,722	2,190	1,759	0.98	0.17	5.4	
C ^a	2,310	2,100			0.84	0.41	13.0	
Combined	2,310	1,722	2,139	1,778	0.95	0.24	7.4	
Combined ^a	2,310	2,100			0.84	0.42	13.3	
Sugar								
A	2,270	2,230	1,722	2,139	1,778	0.96	0.40	13.4
C	2,310	1,722	2,139	2,100	1,759	0.96	0.29	11.4
Combined	2,310	1,722	2,139	2,180	1,759	0.96	0.34	12.3

^aWavelengths used by Osborne et al (1984) for lipid in crumb of English white bread.

TABLE IX
Testing of Lipid and Sugar Calibrations for White Bread

Constituent	Baking Experiment for Calibration	Baking Experiment for Testing	Statistics of Testing Results				Near-Infrared Bias (%)
			n	R	SDP (%)	C.V. (%)	
Lipid ^a	A	C	49	0.91	0.21	6.7	+0.240
	Combined	A	50	0.97	0.18	5.8	+0.025
	Combined	C	49	0.98	0.14	4.4	-0.001
Sugar ^b	A	C	49	0.91	0.38	15.1	+0.040
	Combined	A	50	0.93	0.35	12.7	+0.060
	Combined	C	49	0.93	0.33	13.1	-0.070

^aTested with calibrations containing filters at 2,336, 2,310, 1,722, 2,190, 1,778, and 1,445 nm for baking experiment A and filters at 2,310, 1,722, 2,190, 1,778, 1,940, and 1,445 nm for combined data.

^bTested with calibrations containing filters at 2,270, 2,230, 1,722, 2,139, 1,778, and 1,940 for baking experiment A and filters at 2,310, 1,722, 2,139, 2,180, 1,759, and 1,445 for combined data.

with filter at wavelengths of 2,348, 2,139, 1,722, 1,778, and 1,759 nm.

Osborne et al (1984) reported a standard deviation of 0.16 for a lipid calibration equation for English white bread using the basic filters at 2,310 and 2,100 nm. Using these filters, calibration equations derived from our data had coefficients of variation of 11–13%, considerably less accurate calibration results (Table VIII).

Using a six-filter calibration from bread of baking experiment A, the lipid content in bread from baking experiment C was predicted with good results (Table IX). The standard deviation of prediction between laboratory results and NIR measurements was 0.21%, which was not as good as the 0.14% in bread reported by Osborne et al (1984). Using the combined data calibration with six wavelengths, the lipid content in bread from both experiments was predicted with good results. The standard deviation and coefficient of variation were somewhat higher than for laboratory analysis; they were calculated to be 0.078 and 2.5%, respectively, after adjusting laboratory lipid content from duplicate analyses on loaves with eight levels of lipid to 40% moisture.

TABLE X
Range and Mean of Moisture, Protein, and Lipid
in Air-Dried Whole Wheat Bread^a

Measurement	Sample <i>n</i>	Range (%)	Mean (%)
Moisture	18	8.7–12.2	10.10
Protein (as is)	18	12.7–16.0	13.76
Lipid (as is)	18	4.2– 7.5	5.81

^aThree replicate measurements on 18 loaves.

TABLE XI
Filters and Statistics on Some Calibrations
on Air-Dried Whole Wheat Bread

Constituent	Filters nm	Statistics of Calibration Equation		
		<i>R</i>	SD (%)	C.V. (%)
Moisture	2,336 1,982	0.98	0.20	2.0
	2,336 2,310 2,270 1,982	0.99	0.12	1.1
Protein	2,190 2,180 1,680	0.73	0.44	3.2
	2,230 2,190 2,180 1,778 1,680	0.78	0.41	3.0
Lipid	2,336 2,348 2,310	0.98	0.23	4.0
	2,336 1,722 2,139 1,940 1,680	0.98	0.21	3.6

TABLE XII
Prediction of Crumb Moisture, Protein, and Lipid
by Osborne et al (1984) Calibrations

Constituent Predicted in Baking Experiment	Statistics of Testing Results			Intercept ^d Constant
	<i>n</i>	SDP (%)	C.V. (%)	
Moisture ^a				
A	50	0.69	1.6	18.9
B	50	0.89	2.0	17.8
C	50	1.20	2.8	19.1
D	50	1.00	2.4	17.5
E	50	1.05	2.4	17.3
Protein ^b				
A	50	0.21	2.7	5.41
C	50	0.29	3.7	6.08
D	49	0.41	5.2	5.30
A,C,D Combined	149	0.47	6.0	5.60
Lipid ^c				
A	50	0.70	22.4	-0.79
C	50	0.43	13.6	-2.79

^aPercent moisture = (252.4)(log 1/R 2,230 nm) + (-286.5)(log 1/R 2,100 nm) + (67.24)(log 1/R 1,940 nm) + 16.13.

^bPercent protein = (317.1)(log 1/R 2,180 nm) + (-304.7)(log 1/R 2,100 nm) + (30.10)(log 1/R 1,940 nm) + (-57.02)(log 1/R 1,680 nm) + 5.79.

^cPercent lipid = (169.2)(log 1/R 2,310 nm) + (-168.9)(log 1/R 2,100 nm) - 2.46.

^dUsed in the above calibration equations.

Sugar Calibration

The range and mean sugar content in bread used for developing sugar calibrations are given in Table VII and some of the best calibrations and their statistics in Table VIII. Five filters were required for the best calibrations for total sugar. The standard deviation and coefficient of variation were a little high for a good analytical procedure. The statistically indicated accuracy of calibrations from combining data was intermediate between those of the other two experiments. The sugar content in bread from the Waldron flour (baking experiment B) was predicted satisfactorily (Table IX) with the calibration from bread of composite flour (baking experiment A). Using a calibration for combining data, the sugar content in bread of both baking experiments was also predicted satisfactorily. The standard deviation and coefficient of variation were only a little higher than for laboratory analysis. At a calculated 40% moisture basis they were determined to be 0.33 and 11.4% respectively, from duplicate results on loaves containing eight levels of sugar.

Apart from a band at 1,200 nm caused by the CH₂ stretch, bands at 1,445, 1,500, 1,580, and 2,082 nm resulted from O-H overtone and combinations; only the filter at 1,445 nm would have absorption by these bands. Based on second derivative reflectance values, McClure et al (1977) selected filters at 1,458, 1,476, 1,565, 1,592, 1,790, 1,914, 2,080, and 2,178 nm for predicting total reducing sugar in tobacco, whereas Giangiacomo et al (1982) selected 1,385, 1,433, 1,469, 1,688, 1,682, and 1,820 nm as the first two wavelengths in three-term normalized second derivative prediction equations for fructose, glucose, and sucrose in model mixtures of apple sugars.

Near-Infrared Calibrations for Air-Dried Bread

One set of calibrations was obtained on air-dried whole wheat bread baked by the straight-dough method from mixtures of whole wheat and Waldron white flour (with no addition of wheat gluten and starch to vary the protein content). The range and mean of moisture, protein, and lipid in the air-dried bread are shown in Table X and filters and statistics on some calibrations in Table XI. The coefficients of variation for the moisture protein and lipid calibrations in dried bread was near those for fresh sliced white bread (Tables IV, VI, and VIII); they indicated good analytical procedures. The low correlations observed for protein calibrations were probably caused by the low range of protein in the calibration samples when gluten and starch were not used to vary the protein.

These results confirmed the recent conclusion of Osborne et al (1984) that drying and grinding of the bread before NIR analysis does not significantly increase the accuracy of the NIR analytical method. There is, however, much more work when using a drying step before NIR analysis.

Transferability of Bread Calibrations

Calibration equations from Osborne et al (1984) were used to calculate constituent content in our bread baked by laboratory straight-dough, sponge-dough, and no-time-dough procedures. As seen in Table XII, moisture was accurately predicted in bread baked by all three procedures and protein in bread baked by straight-dough and sponge-dough procedures. Lipid was predicted with less accuracy in bread baked by the straight-dough method. Except for the lipid prediction in baking experiment A, the coefficients of variation were about the same as those of our calibrations derived with the filters used by Osborne. Calibrations of Osborne et al were developed on fresh crumb of bread baked by a typical United Kingdom commercial process from grists varying from 100% soft English to 100% Canadian Western red spring wheats. Calibrations appear to be readily transferable between bread baked by widely different procedures and flour. In practical use, the variation in intercept constant (causing bias) with different baking experiments can, however, reduce the accuracy unless this constant is appropriately adjusted from laboratory analysis.

LITERATURE CITED

AMERICAN ASSOCIATION OF CEREAL CHEMISTS. 1983. Approved

- Methods of the AACC. Method 30-20, revised October 1981; and Method 46-11, revised October 1982. The Association: St. Paul, MN.
- DUBOIS, M., GILLES, K. A., HAMILTON, J. A., REBERS, P. A., and SMITH, F. 1956. Colorimetric method for determination of sugars and related substances. *Anal. Chem.* 28:350.
- GIANGIACOMO, R., MAGEE, J. B., BIRTH, G. S., and DULL, G. G. 1981. Predicting concentration of individual sugars in dry mixtures by near-infrared reflectance spectroscopy. *J. Food Sci.* 46:531.
- MacARTHUR, L. A., and D'APPOLONIA, B. L. 1979. Comparison of oat and wheat carbohydrates. I. Sugars. *Cereal Chem.* 56:455.
- McCLURE, W. F., NORRIS, K. H., and WEEK, W. W. 1977. Rapid spectrophotometric analysis of the chemical composition of tobacco. Part I. Total reducing sugar. *Beitrage zur Tabakforschung* 9:13.
- OSBORNE, B. G., BARRETT, G. M., CAUVAIN, S. P., and FEARN, T. 1984. The determination of protein, fat, and moisture in bread by near infrared reflectance spectroscopy. *J. Sci. Food Agric.* 35:940.
- OSBORNE, B. G., FEARN, T., MILLER, A. R., and DOUGLAS, S. 1983a. Application of near infrared spectroscopy to the compositional analysis of biscuits and biscuit doughs. *J. Sci. Food Agric.* 35:99.
- OSBORNE, B. G., FEARN, T., and RANDALL, P. G. 1983b. Measurement of fat and sucrose in dry cake mixes by near infrared reflectance spectroscopy. *J. Food Technol.* 18:651.
- PENCE, J. W., STRANDRIDGE, N. N., MECHAM, D. K., LUBISICH, T. M., and OLCOTT, H. S. 1956. Moisture distribution in fresh, frozen, and frozen-defrosted bread. *Food Technol.* 10:76.
- PONTE, J. G., DeSTEFANIS, V. A., and TITCOMB, S. T. 1969. Application of thin-layer chromatography to sugar analysis in cereal based products. *Abstr. Cereal Sci. Today* 14:102.

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