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REFLECTANCE NEAR INFRARED ANALYSIS OF DAIRY PRODUCTS AND AIR-CLASSIFIED BEAN FLOUR

By

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ABSTRACT

REFLECTANCE NEAR INFRARED ANALYSIS OF
DAIRY PRODUCTS AND AIR-CLASSIFIED BEAN FLOUR

By

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Low-moisture dairy products and air-classified bean powders were analyzed by conventional methods and by near infrared reflectance (NIR). For 41 spray-dried milk samples prepared by premixing whole milk with skim milk at various proportions, the correlation coefficients (r) between the data of the two analytical procedures were 0.7889, 0.9672, 0.9892, 0.9437 and 0.9528 for moisture, protein, fat, ash and lactose contents, respectively. For six random spray-dried milk samples the corresponding r values were 0.8384, 0.9282, 0.9908, 0.9886 and 0.9882, respectively.

The r values of 31 samples prepared by mixing whole milk powder with skim milk powder were 0.8208, 0.9844, 0.9956, 0.9846 and 0.9959 for moisture, protein, fat, ash and lactose, respectively. For 6 unknown samples the r values were 0.6017, 0.9945, 0.9514, 0.8625 and 0.9628 for moisture, protein, fat, ash and lactose contents, respectively.

Eleven commercially dehydrated milk powders had r values 0.8507, 0.9878, 0.9992, 0.9949 and 0.9888 for moisture, protein, fat, ash and lactose contents, respectively.

For 40 commercial cheese powder samples the r values were 0.7494, 0.9352, 0.9803 and 0.7486 for moisture, protein, fat and ash contents, respectively,

Thirty air-classified bean flour samples were analyzed for moisture, protein, and ash contents using conventional methods and NIR. The r values were 0.8971, 0.9878 and 0.9571 for moisture, protein and ash contents, respectively. Six unknown samples had the corresponding r values: 0.7467, 0.9394 and 0.9699, respectively.

F-values obtained from the analysis of variance of conventional and NIR data for each food components were smaller than F-table values in all cases except for the moisture content of the random spray-dried milk samples. The F-values indicated that there was no significant statistical difference between NIR data and that obtained by the conventional procedures.

The NIR method is rapid (30 seconds), involves little sample preparation, and provides a direct read out. The test is not destructive and requires 8-12 g sample. Accurate calibration of the NIR instrument with a reliable reference method of analysis is necessary for consistent and meaningful data.

TO

My Mother, Noura and My Sister Hilala

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INTRODUCTION

Near infrared reflectance spectroscopy has been developed by Norris and Hart (1965), to measure the moisture content of grains and oilseeds. The technique was later expanded to measure other food constituents, such as protein, oil, starch, sugar and fiber.

The measuring system consists of interference filters to isolate selected wavebands of near infrared energy, a photosensor, a signal conditioning amplifier, and a computer for collecting and analyzing the data.

In 1971, NIR was introduced to the grain industry as a means of rapid analysis for moisture, oil and protein (Rosenthal, 1971). At least three instrument manufacturers, Neotec, Technicon and Dickey-john, made available instruments of increasing sofistication for the analysis of grains and oilseeds.

In 1978, NIR was introduced to the baking industries in the United States, as a high speed analytical technique of flour and a quality control tool.

In the last fifteen years, many workers used the NIR technique to estimate the concentrations of several constituents of agricultural products (Norris et al., 1976, Stermer et al, 1977, Watson et al., 1976, Williams and Starkey, 1980).

The work reported here describes an effort to use NIR for the determination of moisture, protein, fat, ash and lactose contents in low-

moisture dairy products and moisture, protein and ash contents in air-classified bean flours.

REVIEW OF LITERATURE

Norris and Hart (1965), investigated the water absorption bands at 0.76, 0.97, 1.18, 1.45 and 1.94 μ for the spectrophotometric measurement of water in seeds and grains. These efforts led to the development of a near infrared reflectance (NIR) technique, which originally aimed at determining the moisture content of agricultural products, but later was expanded to the measurement of protein, oil, starch and other constituents in these products.

Hymowitz et al (1974), estimated the protein and oil content in corn, soybean, and oat seeds by NIR using the grain analyzer manufactured by Dickey-john Corp. These authors also studied the effect of sample grinding time on the grain analyzer readings for protein and oil content. Their most important findings were: a) the high correlation between the NIR and Kjeldahl data for protein content, and; b) the lack of statistical significance between grinding time and protein or oil content estimates.

Pomeranz and Moore (1975), compared six methods of protein determination: Kjeldahl, biuret, dye binding, alkaline distilation, NIR by GAC-2 (Dickey-john) and NIR by GQA (Neotec). They reported a high correlation by all the six methods for protein determination in wheat.

Williams (1975) applied NIR to the analysis of several cereal grains and oil seeds including hard red spring wheat, hard and soft wheat flour, barley, oats, rapeseeds and soybeans. Protein content

was determined by Kjeldahl and by two different NIR instruments. He concluded that, the introduction of NIR instrumentation represents a rapid routine analysis of cereal grains and oil seeds for oil, protein and moisture, and that sample preparation and accurate calibration of the NIR instrument are very important.

Watson et al. (1976) evaluated the GQA-1 and grain analyzer computer (GAC) for protein determination in hard red winter wheat. They reported correlation coefficients of 0.979 with GQA and 0.982 with GAC for protein estimation between Kjeldahl and NIR. GQA was easier to calibrate and operate than the GAC.

Watson et al. (1977) developed a regression equation for protein determination by Kjeldahl and NIR using five classes of wheat. It was found that the slope of the regression equation depended on the wheat class and that the effect of wheat class on the regression equation was not related to the particle size distribution of the ground sample.

Stermer et al. (1977) attempted to estimate the moisture of whole grain corn and sorghum using the neotec GQA-41 instrument. The correlation coefficients between the NIR values and those by the oven method were 0.997 for whole corn and 0.974 for whole grain sorghum.

Rubenthaler and Bruinsma (1978) estimated the amino acid lysine in wheat samples using NIR. They used the Technicon Infraalyzer interfaced with the Hewlett-Packard 9815A programmable calculator, and found a high correlation between the lysine values predicted by NIR with those determined by amino acid analysis.

Rosenthal (1978) reported the absorption spectra of various food components with the emphasis on the best absorption wave lengths. In addition to agricultural products he noted a wide scope for the use of

NIR in industrial and clinical applications.

Several commercial models of NIR instruments are manufactured by Dickey-john, Neotec and Technicon Corporations.

The Neotec Feed Quality Analyzer model 51 provides optical data at a greater number of wave lengths and directly prints percentage of protein, oil, moisture and fiber. Dairy products have become an important part of American diet. In these products the amount of the yield is related to the total solid content of fluid milk. Hence the need for rapid and accurate measurement of fluid milk constituents is essential.

Hooton (1978) emphasized the versatility of NIR in several business areas as receiving, manufacturing, shipping, laboratory and inventory. On the other hand, he pointed out several industrial areas as grain handling, milling, mixed feeds, dry corn milling, and residual oil in corn germ cake, where NIR rapid analyses are very important.

Norris (1978) showed that NIR measurements are not limited by the instrument noise alone. Sample preparation is the greatest source of variability and grinding is important. Williams and Thompson (1978), investigated the effect of granularity of the hard red spring wheat using NIR analysis for protein and moisture content and found several factors effecting granularity, such as genetic constitution, chromosome number, wheat type, variety, soil, grinder and grinding conditions as well as sample size. The researchers recommended the following steps to achieve accurate results: 1) Use of high speed hammer mill for sample preparation. 2) Optimum mean particle size for NIR analysis of hard red spring wheat is between 180-220 μ, 3) At least fifty samples to be used for calibration. 4) A wide range of the content of food constituent is necessary for calibration of the instrument.

Williams et al. (1978) applied the NIR technique for protein and moisture testing in pulse breeding programs to improve both yield and quality of pulses in the dry and tropic areas. Pulses were obtained from different research institutes all over the world and were analyzed for protein content by Kjeldahl and were subsequently analyzed by NIR using the Neotec Quality Grain Analyzer model 31. They found correlation coefficients from 0.89 to 0.96 between Kjeldahl and NIR.

Miller et al. (1978) investigating the protein content in hard red winter wheat samples found excellent reproducibility in the values obtained by NIR.

Williams (1979) investigating the possibility of screening wheat for protein content and hardness, used two sets of wheat samples which varied in Kjeldahl protein content and hardness. Hardness was assessed by the particle size index (PSI) test. All wheats were ground with a Burr mill and an Impeller-type mill, and passed through a 1.0 mm screen. Neotec GQA-31 was calibrated against protein content and PSI with the Burr mill and for protein with the Impeller-milled samples. He reported that protein content was predictable to within 0.31% in the Impeller-ground wheat samples and within to 0.70% in the Burr milled samples, and the PSI was predicted to within less than two units. He further analyzed five classes of hard wheat by one calibration and another five soft wheat classes by different calibration and showed that analysis of hard wheat was more accurate than that of the soft wheat.

Birth (1979) reviewed the measurement of food quality by radiometric methods. He concluded that the evaluation of any new method relays on statistical analysis and found that spectral reflectance or transmittance data generated by computation of derivative spectra can

be analyzed by multiple linear regression to develop the best equation for food quality prediction.

Giangiacomo et al. (1981) employed the NIR technique to measure the concentration of fructose, glucose and sucrose in model systems. Subsequently, measurements were made to estimate the same sugars in dried apple tissues. The correlation coefficients of the actual concentrations versus the predicted values were 0.995, 0.994 and 0.986 for fructose, glucose and sucrose, respectively in the model systems comprising 20 samples. But when they tried to use the prediction equations to estimate the sugars in the dried apple samples, the corresponding correlation coefficients were 0.70, 0.55 and 0.90 for fructose, glucose and sucrose, respectively.

Fernandez (1981) using the GQA-41 estimated the nitrogen content of several dried tissues of bean plants (seed, pod wall, leaf blade, petiole, steam and root) with a correlation coefficient ranging from 0.873 to 0.973, between Kjeldahl and NIR.

Shenk et al. (1981) using the spectro-computer Neotec 6100 evaluated the acid fiber, neutral fiber, lignin, cellulose, Ca, P and K in forage. In addition, 90 samples of Canadian wheat were also evaluated for their protein content. They concluded that this instrument provides an acceptable means of evaluating forages and grains. The accuracy of NIR depends on the successful completion of the following:

1. Selection of a representative set of samples from the population.
2. Accurate laboratory analysis of the quality parameters of interest.
3. Accurate NIR data.

4. Appropriate transformations of the NIR data for each quality parameter to be predicted.
5. Appropriate wavelengths for the whole population.

Chief sources of error in near infrared reflectance testing include:

1. Selection of calibration samples.
2. Accuracy of standard chemical analysis used in calibration or monitoring.
3. Particle size and particle size distribution.
4. Homogeneity of ground sample.
5. Moisture status of samples.
6. Sample storage.
7. Uneven or inconsistent loading of cell.

METHODS AND MATERIALS

Spray-dried milks, milk powders prepared by dry-mixing, cheese powders, commercially dried milk and air-classified bean flours were used in this research. All samples were analyzed in duplicates. The data are reported on dry weight basis for air-classified bean powder and on "as is" basis for dairy products.

A. Sample Preparation.

Spray-dried milk

Forty one 3-liter samples were prepared by mixing 0+3000, 75 + 2925, 150 + 2850.....and 3000 ml of whole pasteurized milk (3.06% fat) + 0 ml of skim milk (0.11% fat). The mixtures were stirred, spray-dried and samples of the powders were collected in glass jars, packed in two-layer polyethylene bags, sealed and stored at room temperature until the time of analysis. A spray drier manufactured by Swanson Evaporator Company, Harvey, IL, was used. The inlet air temperature was 350°F, and that of outlet air temperature was 150°F. The atomizer air pressure was 35 PSI and similar air pressure was exerted on the feed tank. The purpose of mixing two kinds of milk was to establish a wide range of fat concentrations which is necessary for calibrating the grain quality analyzer (GQA-41). The forty one spray-dried samples were used for calibrating the instrument. Some other samples were prepared in a similar way and later were used as unknowns.

Dry-mixed milk samples

Thirty one samples were prepared by mixing 0, 1, 2, 3....and 30 g of commercially spray-dried whole milk (Valley Lea Dairies, Inc., South Bend, Indiana) with 30, 29, 28, 27,.....and 0 g non-fat dry milk (Golden Guernsey Dairy, Sparta, WI). These samples were stored in glass jars until the proximate analysis were performed. The purpose of mixing the two kinds of dry milk powder was to obtain samples with a wide range of concentrations for calibrating the GQA-41 instrument. Some unknown samples were prepared in a similar way.

Cheese powder

Forty samples of cheese powder were provided by Commercial Creamery Co., Spokane, WA. These cheese powders were representative of the powders sold in the industry for snack seasoning. All samples were used to calibrate the GQA-41 instrument.

Air-classified bean flour

Thirty air-classified bean flour samples were provided by Dr. M. Uebersax (Food Science Department) which were used to calibrate the instrument. Several other samples were used as unknowns. Also three commercial samples were analyzed as unknowns. The samples were kept in polyethylene bags until they were analyzed.

Commercial dry milk

Eleven commercially dried skim and whole milk were provided by the Michigan State Department of Agriculture, Laboratory Division. Two milk samples were bought from the market in E. Lansing area and were used as unknowns.

All samples except the bean flour were ground for two minutes in a Mitey-mill, a high speed rotating blade type. The powders were passed

through a 100 mesh sieve to enforce a uniformed particle size, which was later packed and used for calibrating the GQA-41 instrument and proximate analysis.

B. Sample analysis

Moisture and ash contents of all samples were determined by the oven method and dry ashing respectively according to AOAC (1975). Lactose content of the milk powders were calculated by difference.

Nitrogen determination by the micro-Kjeldahl method

Approximately 50 mg of each sample were digested for one hour in duplicate according to AOAC (1975). Sulfuric acid of 1.84 specific gravity was used for digestion. Potassium sulfate and mercuric oxide were added as catalysts. After cooling the flasks, the sides were rinsed with deionized water and digestion continued for another hour.

The digests were transferred into the distillation apparatus by using approximately 10 ml deionized water. The diagested mixture was neutralized with 15 ml of 50% NaOH solution, containing 5% sodium thiosulfate. The liberated ammonia was steam-distilled into 5 ml of 5% boric acid solution, containing 4 drops of methyl red-methylene blue indicator (2 parts of 0.2% methyl red in alcohol with one part of 0.2% methylene blue in alcohol). The distillation was continued until the volume in the receiving flask reached 25 ml. The ammonium borate complex was titrated with 0.02 N HCl which had been accurately standardized against tris-hydroxy amino methane (THAM) as a primary standard. Nitrogen was calculated from the following formula:

$$N\% = \frac{(\text{ml HCl-ml blank})\ (\text{normality of HCl})\ (14.007)}{\text{mg of sample}} \times 100$$

Protein content was calculated as follows:

% Protein = %N x 6.25 for bean flour.

% Protein - %N x 6.38 for dairy products.

Fat determination

Approximately 2 g of the dairy product samples were transferred carefully to a Mojonnier flask and analyzed according to Mojonnier (1925). The sample was mixed with 8.5 ml deionized water. Two ml of ammonium hydroxide was added to neutralize the acidity, followed by adding 10 ml of ethyl alcohol. The fat was extracted with 25 ml anhydrous diethyl-ether, flasks were stoppered with a rubber stopper and shaken vigorously for 2 minutes. About 25 ml of petroleum ether was added to the mixture, followed by shaking for 2 minutes, and the flasks were set aside to assure complete separation of the two layers. The upper layer was carefully poured into previously pre-weighed can, second extraction was performed by adding 5 ml ethyl alcohol, 25 ml ethyl ether and 25 ml petroleum ether to the flask containing the sample mixture. The upper layer was combined with the previous extract.

The fat extractant was evaporated by placing the can on a steam bath. Further, the cans were dried in the vaccum oven at 100°C for 20 minutes, followed by cooling for 5 minutes in a desiccator. The cans were re-weighed and the fat content was calculated.

Instrumentation

The Neotec Grain Quality Analyzer model 41 (GQA-41) interfaced with a teletype computer that could be connected with the main computer at Michigan State University was used in this research.

The GQA-41 is built around a Carry model 14 prism-grating infrared monochromator. The sample is packed into the sample cell and a quartz

glass window covers the smooth surface of the sample. A spring-loaded pressure plate is used to hold the sample smoothly against the glass window.

The sample is illuminated through the window and the diffused reflected light is collected with four lead sulfide cells placed at equal distances around the sample. A solid teflon plate is used as a reflectance reference. The signal from the detectors is fed to the computer after it has been amplified with a logarithmic response amplifier which is digitized.

The sample is scanned in the useful range of the infrared, 1.2 - 2.5 μ at a scanning speed of 10 mm per second and the reflectance values are recorded as $dR/_R$, where dR is the differential coefficient of a line tangential to the absorption curve peak, R is the absolute reflectance. A change in a food constituent concentrations results in a change in the corresponding dR. This mathematical model is referred to as $dR/_R$.

C. Instrument Calibration

The GQA-41 (Figure 18) was calibrated for moisture, protein, fat, ash and lactose (by difference) content of the dairy products under study. Similarly, the instrument was calibrated for moisture, protein, and ash content of the air-classified bean flour. The calibration was performed as follows:

1. The GQA-41 was connected to the computer to collect and analyze the reflectance measurements.
2. Three samples of one product were selected: among those to be used for calibration of the instrument one had the highest content of the food constituent of interest (moisture for instance), the second sample had the lowest content, the

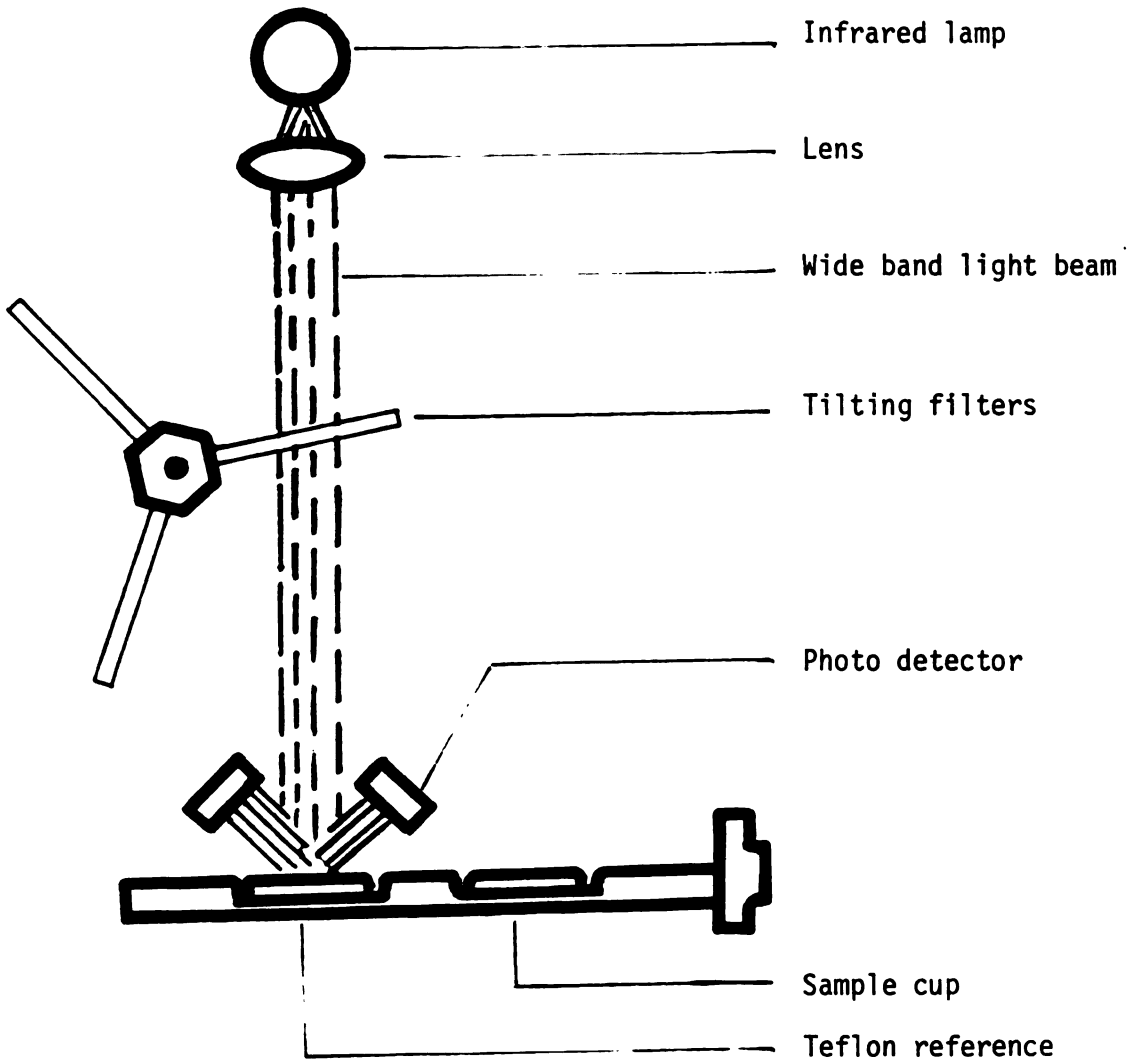

Figure 18. Near infrared reflectance instrument used in this research.

third was randomly selected to be in between the two extremes. Samples were placed in the sample cell, introduced to the radiation beam one after another, the reflectance values were amplified, digitized and recorded. Four reflectance values were obtained at each pulse point, where the maximum response of the system takes place at a certain wave length.

3. The 300 reflectance values for each sample were fed to the computer along with the corresponding analytical values for each food constituent. The computer selected four wave lengths (pulse points) which best correlated with the analytical values for each food component.

4. All of the calibration samples were now read by the GQA-41 computer at the four selected pulse points for each food constituent. The computer performed a polynomial regression analysis and provided the regression coefficients, K values, for the following prediction equation:

$$\% \text{ Food Constituent} = K_0+K_1(\frac{dR}{R_1})+K_2(\frac{dR}{R_2})+K_3(\frac{dR}{R_3})+K_4(\frac{dR}{R_4})$$

Where: K_0 is the intercept of the multilinear regression line. K_1, K_2, K_3 and K_4 are the regression coefficients at four different wavelengths dR is the differential coefficient. R is the absolute reflectance value at a particular wavelength.

5. Adjustment of K_0

The intercept K_0 was adjusted by inserting the pulse points and K values of the particular food component into the GQA-41 computer. All samples were measured again and a reading was recorded as % food constituent. The difference between the mean analytical values and the mean regression values was calculated and was substrated or added to the K_0 value. The new calculated K_0 was considered the most suitable value to analyze the product for that food constituent. The new calculated K_0 along with the rest of K values and pulse points were inserted into the GQA-41 computer for routine analysis.

D. Statistical analysis

The data obtained by conventional analytical procedures and those estimated by NIR were subjected to correlation analysis and analysis of variance according to Cochran (1957). The 95% confidence limit intervals were calculated according to Snedecor (1955).

RESULTS AND DISCUSSION

The tables 1, 2, 3 and 4, show the moisture, protein, fat and ash contents of the spray-dried milk, dry-mixed milk, cheese powder, and air-classified bean flour respectively by the conventional and NIR procedures.

All food constituents were reported on "as is" except for the air-classified bean flour which was calculated on a dry basis. The lactose content in dairy products was calculated by difference. The analytical values were used to calibrate the GQA-41 and later used to estimate the NIR values, which are shown in the Tables 1, 2, 3, and 4. The best four pulse points which were selected by the regression analysis are shown in Table 5. The P_0 is constant and is 80 according to Neotec GQA-41 manual. The P_1, P_2, P_3 and P_4 are shown for each food component in all the products.

The pulse points were selected from the relationship between the reflectance of a particular food constituent and the change in the wave length at the NIR region.

To estimate the percentage food constituent content in a sample, the pulse points (λ_S) and the K values (Table 6) of that particular food component must be fed back to the computer of the GQA-41. The sample is introduced in the sample cup to the NIR light, the instrument will measure the reflectance and the computer will solve the prediction equation for % food constituent, and the result is displayed or recorded. The means and ranges of the food constituent contents analyzed by both

Table 1. Analysis of spray dried milk by conventional and NIR procedures.

	Conventional					NIR				
Sample Number	% H_2O	% Protein	% Fat	% Ash	% Lactose (By difference)	% H_2O	% Protein	% Fat	% Ash	% Lactose
1	3.81	33.54	1.01	7.33	54.31	3.83	32.13	0.75	7.32	54.34
2	2.82	32.39	2.24	7.36	55.19	3.20	32.14	2.55	7.28	54.44
3	5.12	31.03	5.81	7.04	51.00	4.60	30.24	5.29	7.08	52.39
4	5.26	28.89	10.77	6.79	48.29	5.29	28.86	8.60	6.99	49.93
5	4.61	29.31	9.85	6.51	49.72	3.33	29.48	9.92	6.80	50.63
6	1.98	28.99	12.69	6.71	49.63	2.50	28.50	13.67	6.66	47.69
7	2.93	28.70	14.82	6.38	47.17	3.02	27.95	15.17	6.52	47.05
8	3.85	28.01	13.54	6.46	48.14	3.07	28.42	14.09	6.65	47.50
9	2.37	28.56	12.76	6.79	49.52	2.27	28.90	12.08	6.68	47.74
10	4.47	28.53	11.18	6.78	49.04	4.21	28.83	11.64	6.73	49.06
11	4.99	27.61	12.53	6.49		6.50	28.15	14.56	6.35	
12	2.59	29.35	9.53	7.09	51.44	3.57	29.26	10.84	6.79	50.07
13	3.19	28.73	10.98	6.98	50.43	4.04	29.13	11.23	6.81	49.55
14	3.12	28.93	10.75	7.01	50.19	3.23	29.18	12.41	6.69	48.90
15	3.15	28.86	11.15	6.87	49.97	2.18	28.70	13.57	6.66	48.57
16	3.13	28.97	11.68	6.74	49.48	4.06	29.09	12.07	6.75	49.57
17	2.55	28.94	12.34	6.79	49.38	2.99	29.18	11.38	6.60	50.14
18	2.04	27.56	14.72	6.64	49.04	2.00	27.96	14.95	6.56	47.05
19	1.22	27.57	15.71	6.71	49.32	2.35	27.87	15.50	6.50	46.62
20	3.52	26.94	15.04	6.68	47.82	3.40	27.73	14.70	6.53	47.10
21	0.64	28.26	15.45	6.68	48.97	2.69	28.21	16.40	6.48	47.97
22	1.55	27.67	16.97	6.60	47.21	2.45	27.26	17.46	6.46	46.55
23	2.07	27.47	16.07	6.74	47.65	1.44	27.45	15.85	6.47	46.31
24	1.75	26.72	22.41	6.25	42.87	2.11	26.07	21.15	6.19	43.38
25	2.77	25.76	21.49	6.16	43.32	1.70	26.27	21.47	6.20	44.58
26	3.36	26.05	19.83	6.19	44.57	1.87	26.10	20.82	6.11	46.75
27	2.19	25.95	20.37	6.35	45.14	2.66	26.06	18.52	6.20	45.52
28	1.89	26.91	21.12	6.26	43.82	1.64	26.28	19.78	6.17	45.09
29	1.47	26.09	21.41	6.21	44.82	1.69	26.07	20.50	6.07	44.72
30	1.73	25.52	21.96	6.09	44.70	1.36	25.93	22.66	6.05	42.99
31	3.07	25.51	21.81	6.03	43.58	2.40	25.49	20.86	6.06	44.27
32	1.38	26.61	23.32	6.03	42.66	1.45	25.43	22.17	6.06	44.26
33	3.34	24.54	21.29	5.97	44.86	3.78	24.58	21.33	6.05	44.02
34	2.77	25.37	23.03	5.92	42.91	2.27	25.07	22.04	6.04	43.98
35	1.76	25.75	24.39	5.87	42.23	1.52	25.16	24.24	6.12	42.08
36	1.18	24.77	25.07	5.81	43.17	1.21	24.90	23.17	6.04	42.25
37	1.36	25.80	26.03	4.80	41.01	1.08	24.65	25.34	5.97	41.26
38	1.51	24.56	25.37	5.80	42.76	2.42	24.83	24.39	5.90	41.91
39	1.81	24.91	25.59	5.75	41.94	1.58	24.49	25.15	5.88	41.75
40	1.53	25.02	25.35	5.80	42.30	2.34	24.21	24.63	6.90	41.77
41	2.35	23.91	26.49	5.72	41.53	1.79	24.73	25.76	5.78	41.41

Table 2. Analysis of dry-mixed milk powders by conventional and NIR procedures.

	Conventional					NIR				
Sample Number	% H_2O	% Protein	% Fat	% Ash	% Lactose (By difference)	% H_2O	% Protein	% Fat	% Ash	% Lactose
1	3.11	33.51	1.03	7.80	54.55	3.16	33.29	0.55	7.59	54.16
2	3.09	32.93	1.83	7.94	54.21	3.07	32.84	2.80	7.57	54.32
3	3.11	33.15	2.96	7.83	52.95	3.08	32.52	2.44	7.50	53.24
4		33.02	3.42	7.78	52.73		32.26	3.82	7.45	52.82
5	3.09	32.73	4.56	7.89	51.73	2.88	31.91	4.98	7.46	52.34
6	2.97	32.23	5.47	7.65	51.69	2.85	31.36	4.49	7.41	51.98
7	3.19	31.23	6.23	7.63	51.72	2.99	31.19	6.23	7.42	51.36
8	3.12	31.75	6.74	7.38	51.01	2.95	31.11	7.49	7.46	51.18
9	3.01	30.71	7.68	7.53	51.07	2.99	30.79	7.35	7.41	50.82
10	2.85	30.75	8.85	7.46	50.09	2.93	30.80	8.29	7.40	50.41
11	2.65	30.46	9.53	7.40	49.96	2.93	30.29	9.61	7.37	50.00
12	2.61	30.08		7.32	49.70	2.69	29.96		7.21	49.47
13	2.89	29.81	11.19	7.12	48.99	2.69	29.87	9.59	7.22	49.10
14	2.78	28.91	11.96	7.06	49.29	2.82	29.27	11.07	7.26	48.68
15	2.66	28.77	12.43	7.08	49.06	2.82	28.99	12.93	7.19	47.92
16	2.61	28.79	13.69	6.97	47.94	2.80	29.13	12.73	7.18	47.70
17	2.38	28.75	14.42	6.85	47.60	2.68	28.40	14.29	7.11	47.42
18	2.34	27.94	15.39	6.78	47.55	2.65	28.34	14.07	7.03	46.78
19	2.68	28.17	16.07	6.77	46.41	2.61	27.29	16.49	7.05	46.38
20	2.46	27.94	17.11	6.76	45.73	2.53	27.22	17.46	7.01	45.75
21	2.31	27.85	17.78	6.66	45.40	2.49	27.31	18.42	6.98	45.32
22	2.22	27.82	18.99	6.62	44.35	2.49	26.38	19.68	6.89	44.59
23	2.88	26.30	19.23	6.25	45.34	2.69	26.23	17.72	6.85	45.25
24	2.84	25.68	20.34	6.23	44.91	2.75	25.81	19.87	6.83	44.65
25	2.74	25.51	20.87	6.17	44.71	2.74	25.50	20.72	6.75	44.24
26	2.56	25.47	22.07	6.17	43.73	2.63	25.56	21.48	6.80	43.77
27	2.67	24.99	22.94	6.16	43.24	2.57	25.02	22.86	6.77	43.16
28	2.51	24.16	24.22	6.03	42.08	2.56	24.19	23.43	6.76	42.48
29	2.65	24.92	24.75	6.05	41.63	2.57	23.97	25.00	6.74	41.87
30	2.63	24.83	25.83	5.98	41.18	2.51	23.68	26.89	6.66	41.18
31	2.52	25.27	26.49		40.04	2.71	25.59	25.31		40.04

Table 3. Analysis of cheese powders by conventional and NIR procedures.

Sample number	Conventional				NIR			
	% H_2O	% Protein	% Fat	% Ash	% H_2O	% Protein	% Fat	% Ash
1	3.64	18.12	30.23	9.79	3.67	17.98	28.08	8.93
2	1.54	13.60	70.60	2.85	2.58	13.03	70.94	3.42
3	3.63	21.53	40.37	9.52	3.91	21.55	40.28	7.66
4	3.32	13.96	22.36		4.26	17.41	26.59	
5	4.34	20.49	39.69	7.88	4.20	19.29	40.45	8.06
6	3.91	18.02	31.43	9.51	4.00	17.25	30.49	8.96
7	3.81	16.55	40.09	7.93	3.60	17.07	40.42	7.73
8	6.01	24.88	48.91	8.13	3.50	23.01	46.86	7.96
9	4.74	19.66	37.59	8.07	4.34	17.69	34.93	8.61
10	4.51	18.44	37.44	7.42	3.81	18.11	36.64	8.09
11	4.79	19.13	33.49	8.16	4.56	17.31	31.80	9.02
12	5.02	17.35	30.23	9.48	4.95	17.62	31.49	9.19
13	4.93	18.69	32.89	8.03	4.73	17.29	31.75	9.12
14	5.03	22.22	38.91	7.87	4.85	20.32	37.49	8.57
15	2.90	29.37	53.76		3.28	28.25	55.03	
16	3.21	15.92	38.25	7.38	4.08	14.06	40.22	8.08
17	3.80	17.93	32.06	8.11	4.01	17.73	31.28	8.81
18	3.95	19.88	39.51	8.07	4.88	20.36	41.65	8.44
19	4.17	22.18	38.52	8.64	3.94	21.58	39.43	7.70
20	4.13	19.65	39.48	7.11	4.80	19.07	40.33	8.37
21	4.44	15.96	32.47	8.56	4.24	16.26	32.57	8.79
22	4.05	17.53	34.59	8.65	3.87	17.53	34.57	8.26
23	4.06	16.64	34.13	7.79	3.87	16.66	41.95	7.73
24	4.01	21.99	40.74	8.46	3.70	21.63	42.20	7.49
25	5.71	19.53	49.53	8.41	5.61	18.74	53.80	7.45
26	3.75	22.45	34.19	8.94	3.94	21.03	33.57	8.59
27	4.56	18.24	32.68	8.47	4.11	16.84	32.72	8.61
28	4.65	20.86	38.21	7.81	4.19	20.46	38.91	8.19
29	3.31	20.77	38.56	9.22	3.86	21.16	39.76	7.68
30	3.91	17.01	28.86	9.76	4.37	17.50	30.27	9.12
31	3.95	17.81	38.02	7.70	3.93	15.55	39.06	8.02
32	3.56	18.64	33.56	7.88	3.80	19.21	33.78	8.32
33	5.21	17.45	30.22	8.93	4.12	17.58	28.04	8.85
34	4.62	16.89	41.14	9.08	4.84	16.02	44.54	7.87
35	3.77	21.56	37.84	9.13	3.52	21.16	38.34	7.66
36	4.79	21.07	37.77	7.89	4.48	19.79	38.28	8.40
37	4.23	16.72	31.68	8.12	3.61	17.97	30.47	8.40
38	4.26	21.30	38.49	7.89	3.61	20.33	41.38	7.63
39	4.14	17.30	29.54	8.69	3.49	17.30	31.20	8.42
40	1.98	13.69	73.86	3.52	2.25	12.46	75.09	2.69

Table 4. Analysis of air-classified bean flour by conventional and NIR procedures.

Bean type / Flour Class	Conventional			NIR		
	% H_2O	% Protein	% Ash	% H_2O	% Protein	% Ash
Black Beans						
Granular	10.65	25.75	4.16	10.10	23.29	4.27
Fine I	9.44	35.27	5.20	8.74	35.91	5.30
Coarse I	9.63	23.17	3.84	8.77	23.47	4.15
Fine II	8.85	37.09	5.62	8.27	37.56	5.71
Coarse II	9.85	20.52	3.53	9.32	18.71	3.45
Fine High Protein	9.36	36.55	5.36	8.65	35.45	5.39
Pinto Beans						
Granular	8.35	26.80	4.28	8.79	27.62	4.73
Fine I	8.33	39.96	6.14	7.81	39.85	5.91
Coarse I	8.64	20.94	3.34	8.28	18.85	3.64
Fine II	8.35	34.10	5.12	8.27	34.75	5.51
Coarse II	8.47	18.64	2.91	8.13	15.57	3.16
Fine (I + II)	8.40	38.28	6.04	8.11	38.21	5.83
Navy Beans						
Granular	8.88	23.11	3.84	8.77	25.62	4.55
Fine I	7.87	39.14	5.72	8.05	40.78	5.95
Coarse I	8.80	20.29	3.22	8.07	19.59	3.62
Fine II	7.76	39.78	5.57	7.33	41.31	6.03
Coarse II	8.21	17.16	2.76	7.66	16.02	3.25
Fine (I + II)	7.66	40.41	5.63	8.04	40.24	5.98
Roasted Navy Beans						
Granular	7.13	27.12	3.70	7.86	28.93	4.56
Fine I	7.49	39.87	5.46	7.40	43.18	5.91
Coarse I	7.46	20.47	3.23	7.08	19.38	3.35
Fine I	7.02	40.40	5.30	7.19	42.18	5.97
Coarse II	6.92	17.33	2.83	7.06	15.91	2.99
Fine (I + II)	7.04	42.36	5.40	7.69	41.39	5.94
Tempered Roasted Navy Beans						
Granular	7.92	28.11	3.91	7.78	28.98	4.49
Fine I	7.25	44.95	5.34	7.54	43.89	6.00
Coarse I	7.78	21.21	3.67	7.11	20.37	3.40
Fine II	7.16	42.69	5.15	7.60	41.64	5.96
Coarse II	7.33	17.81	3.42	7.54	17.81	3.14
Fine (I + II)	7.11	47.03	5.19	7.19	43.27	6.01

Table 5. The best four reflectance pulse points selected by regression analysis for dehydrated milk, cheese and bean flour constituents.

Product	Pulse points				
Food Component	P_o*	P_1	P_2	P_3	P_4
Wet-mixed milk					
Moisture	80	265	611	903	963
Protein	80	594	635	927	960
Fat	80	601	635	914	967
Ash	80	605	629	909	965
Lactose	80	275	914	938	965
Dry-mixed milk					
Moisture	80	306	628	912	968
Protein	80	274	306	612	906
Fat	80	566	609	906	961
Ash	80	245	267	926	977
Lactose	80	311	611	906	959
Cheese-powder					
Moisture	80	233	271	303	596
Protein	80	233	307	596	962
Fat	80	243	258	927	979
Ash	80	245	267	926	977
Air-classified bean flour					
Moisture	80	270	567	599	912
Protein	80	273	312	599	899
Ash	80	604	626	934	962

*P_o - Number of revolutions of the filters per minute.

Table 6. Multilinear regression coefficients (K values) used for NIR analysis of milk powder, cheese and bean flour.

Product					
Food Component	K_0	K_1	K_2	K_3	K_4
Wet-mixed milk					
Moisture	4.3	300.7	-1161.5	402.5	2430.3
Protein	28.3	890.6	-944.2	-159.4	-1324.5
Fat	15.8	-2512	1524.9	242	-3264.3
Ash	6.1	117.4	180.2	-125.1	194
Lactose	49.4	4627.9	2304.3	-1015.4	1615.6
Dry-mixed milk					
Moisture	3.9	352.5	-1072.3	-334.6	-70.1
Protein	4.7	394.7	365	266.5	-2407.8
Fat	30.9	-279.5	5410.8	4550	532.3
Ash	3.0	18	339.9	-294	1.7
Lactose	43.4	-529.3	1130.1	-661.3	-1182.3
Cheese powder					
Moisture	2.1	107.9	147.2	-539.1	-28.5
Protein	32.8	354.3	2106.2	246.1	-1461.6
Fat	2.7	128.9	-1296.9	86.4	-2134.7
Ash	6.7	71.2	147.1	-40.3	92.3
Air-classified bean flour					
Moisture	7.9	-150.1	-148.8	-157.4	-1430.4
Protein	28.4	1506.1	-1824.1	5629.4	1693.3
Ash	7.1	-279.3	319.3	-56.3	2.6

conventional and NIR methods are shown in Table 7. A wide range is necessary for accurate calibration of GQA-41 instrument. The relationships between moisture content obtained by the oven method and those predicted by the NIR of the spray-dried milk, dry-mixed milk, cheese powder, and air-classified bean flour are shown in Figures 1, 6, 11 and 15.

The correlation coefficients (r) for the moisture content of spray-dried milk, dry-mixed milk, cheese powder, and air-classified bean flour were 0.7889, 0.8208, 0.7494 and 0.8971, respectively. Hymowitz et al. (1974), working with corn, soybeans and oats reported r values for moisture as 0.839, 0.944 and 0.621, respectively.

The narrow range of moisture content of the products, might have contributed to the relatively low moisture correlations compared to those obtained for protein, fat and ash. The regression lines between Kjeldahl and NIR data for the protein content of the same four products are shown in Figures 2, 7, 12 and 16. The correlation coefficients were 0.9672, 0.9844, 0.9352 and 0.9571 for spray-dried milk, dry-mixed milk, cheese powder and bean flour, respectively.

The fat content determined by Mojonnier and NIR methods were also highly correlated. The corresponding r values were 0.9892, 0.9954 and 0.9803 for spray-dried, dry-mixed milks, and cheese powder, respectively. The regression lines are shown in Figures 3, 8 and 13.

The Figures 4, 9, 14 and 17 show the relationships between % ash determined by dry ashing and NIR. The correlation coefficients obtained were: 0.9437 for spray-dried milk, 0.9846 for dry-mixed milk, 0.7486 for cheese powder and 0.9571 for bean flour. The low correlation coefficient 0.7486 for the ash in cheese powder may be due to the uneven

Table 7. Mean and range of the content in several constituents of foods analyzed by conventional methods and subsequently subjected to NIR analysis.

Food Constituents	Mean	Range
	Spray dried milk	
Moisture	2.64	0.64 - 4.99
Protein	27.43	23.91 - 33.54
Fat	16.68	1.01 - 26.49
Ash	6.51	5.72 - 7.36
Lactose	46.55	41.01 - 55.19
	Dry-mixed milk	
Moisture	2.75	2.22 - 3.11
Protein	28.89	24.83 - 33.51
Fat	13.69	1.03 - 26.49
Ash	6.94	5.98 - 7.94
Lactose	47.76	40.04 - 54.55
	Cheese powder	
Moisture	4.03	1.45 - 6.01
Protein	19.03	13.69 - 24.88
Fat	38.30	22.36 - 73.86
Ash	8.13	2.85 - 9.79
	Air-classified bean flour	
Moisture	8.17	6.92 - 10.65
Protein	30.88	17.16 - 47.03
Ash	4.51	2.76 - 6.03

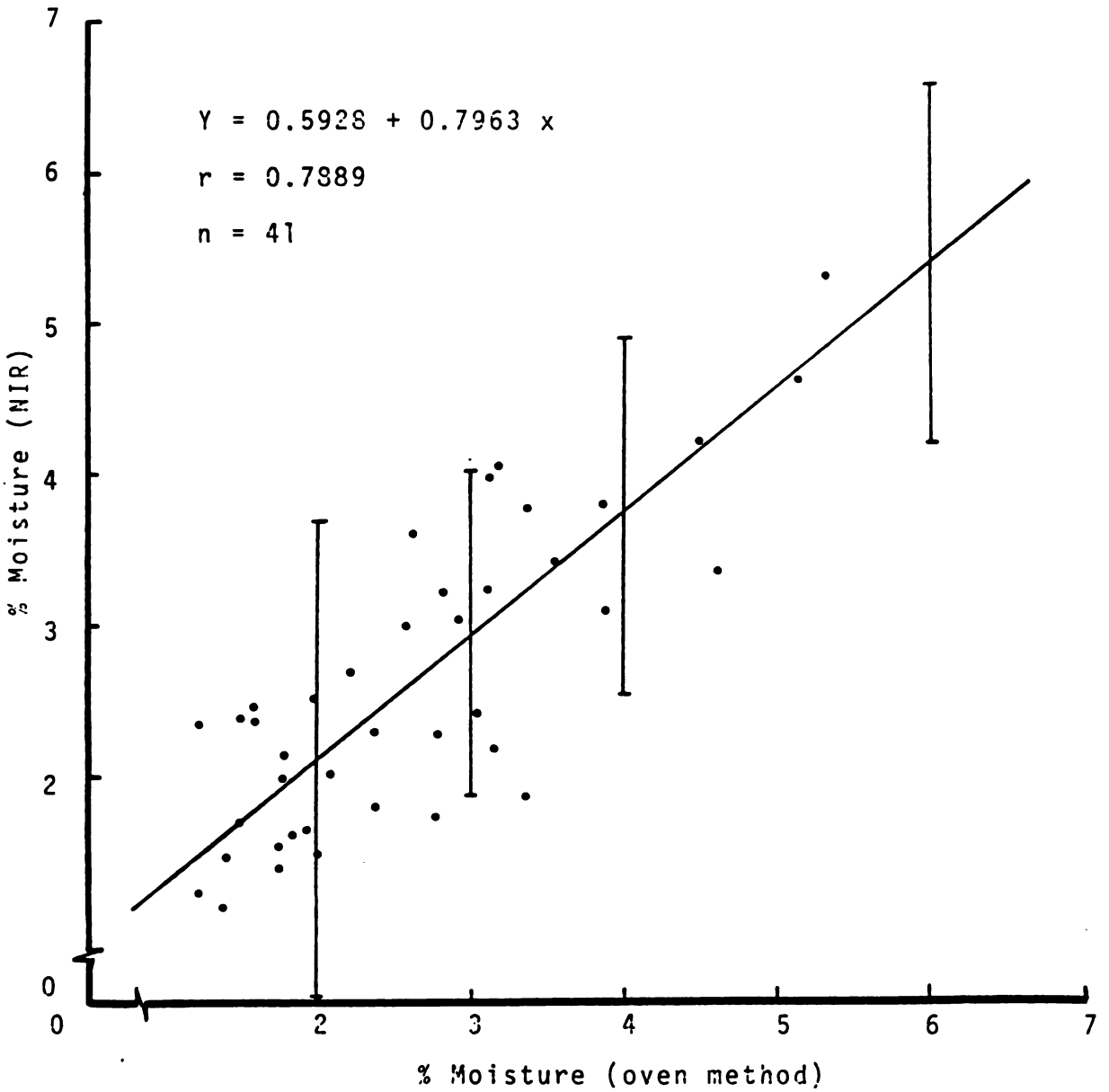

Figure 1. Relationship between % moisture determined by the oven method and by the NIR method in spray dried milk. Bars show the 95% confidence limits of the linear regression.

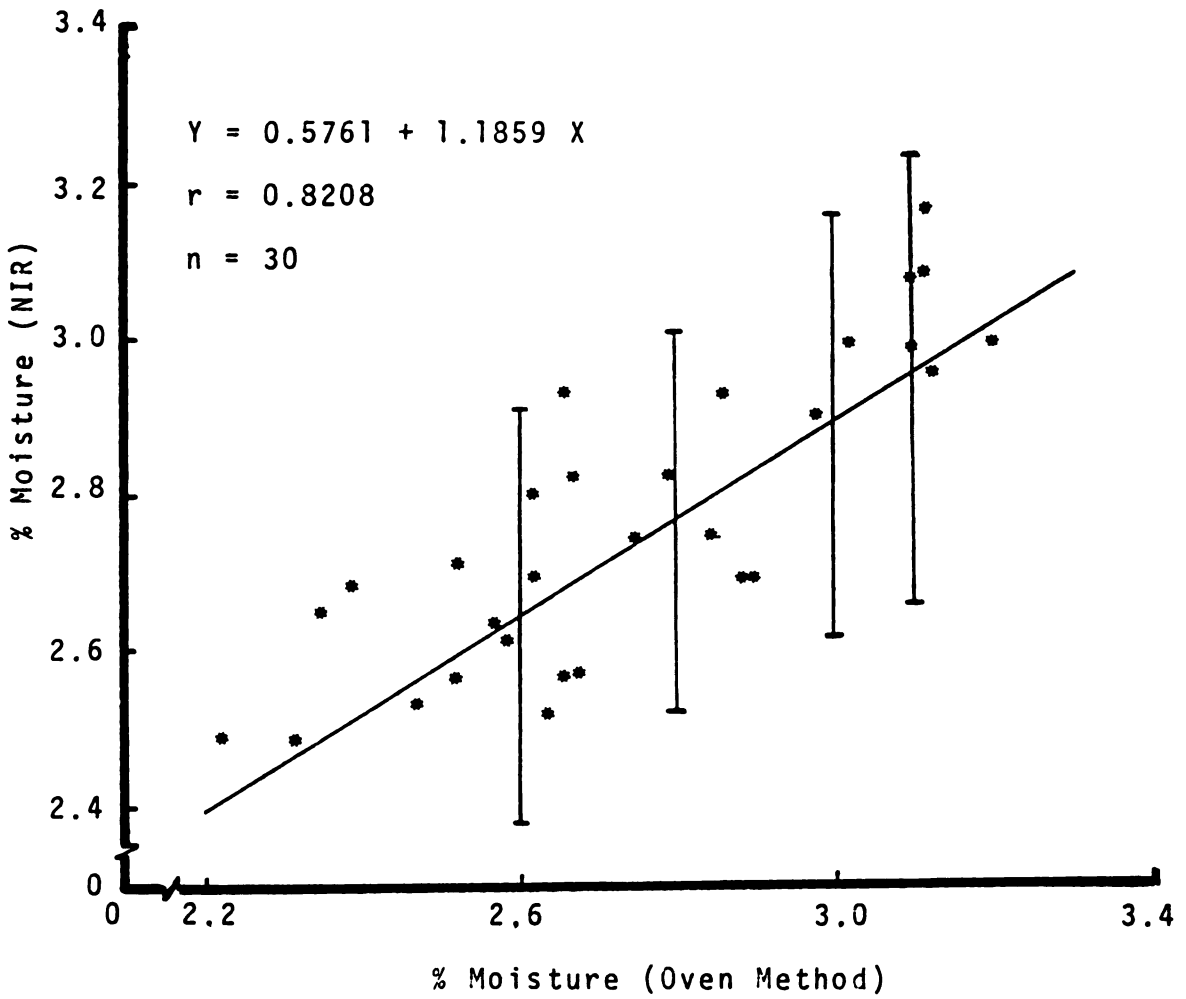

Figure 6. Relationship between % moisture determined by the oven method and by the NIR method in dry-mixed milk. Bars show the 95% confidence limits of the linear regression.

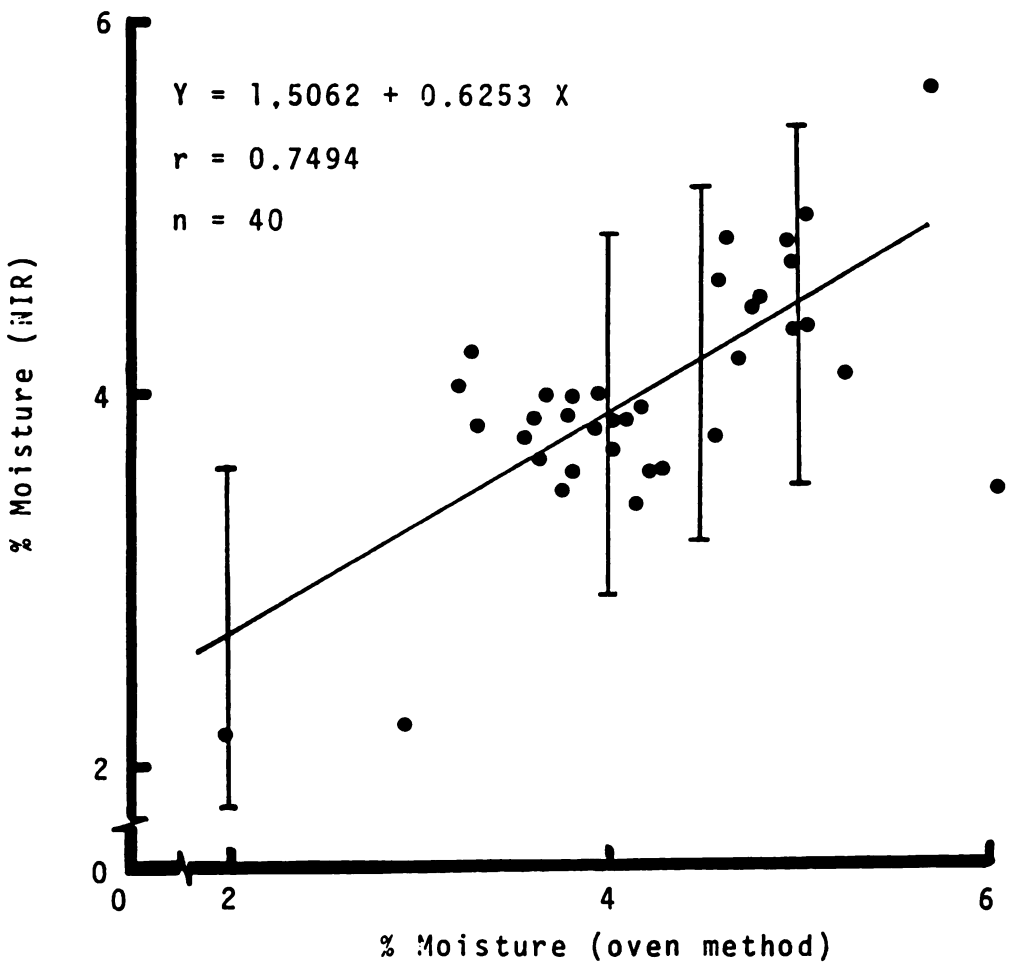

Figure 11. Relationship between % moisture determined by the oven method and by the NIR method in cheese powder. Bars show the 95% confidence limits of the linear regression.

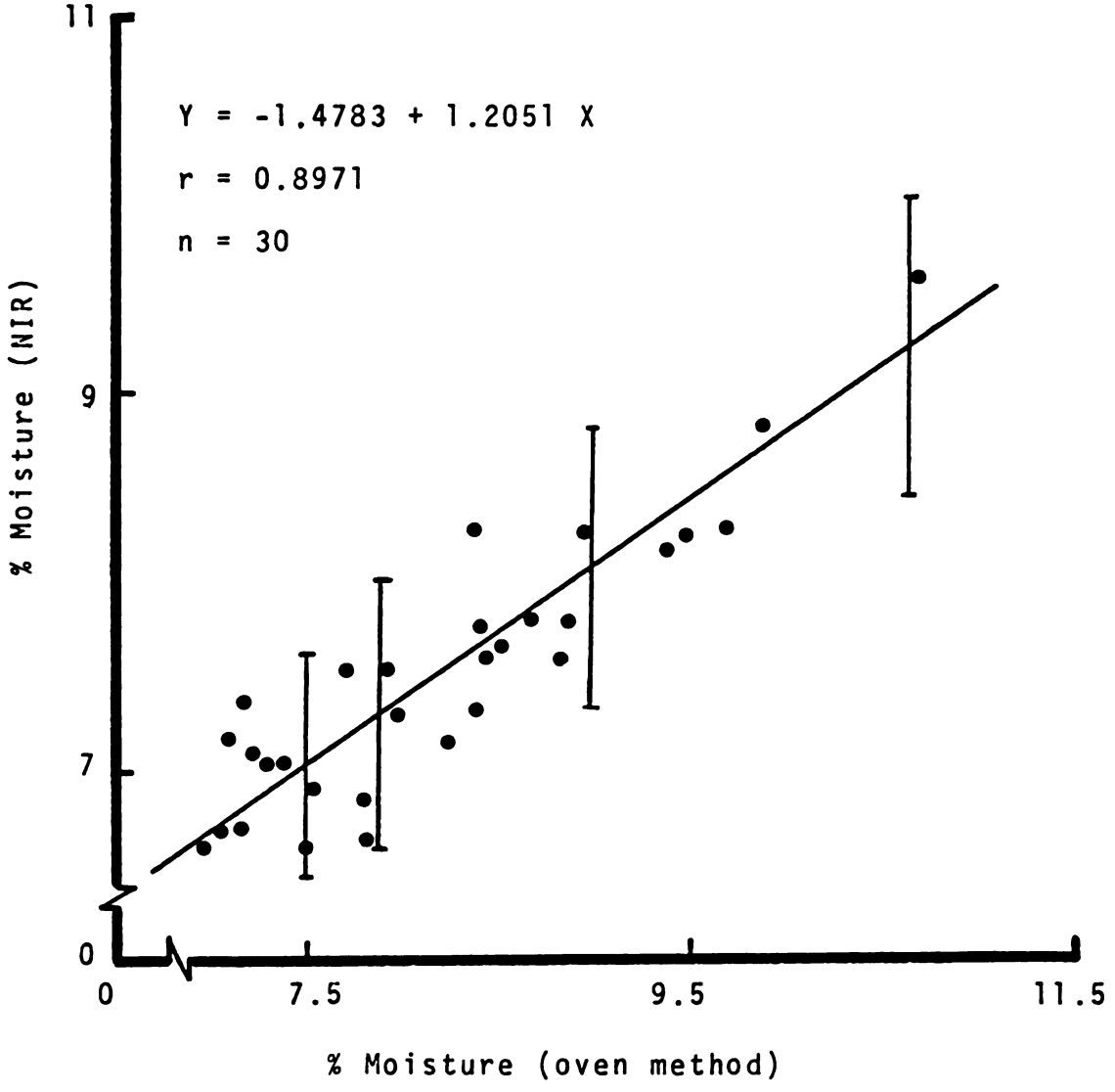

Figure 15. Relationship between % moisture determined by the oven method and by the NIR in air-classified bean powder. Bars show the 95% confidence limits of the linear regression.

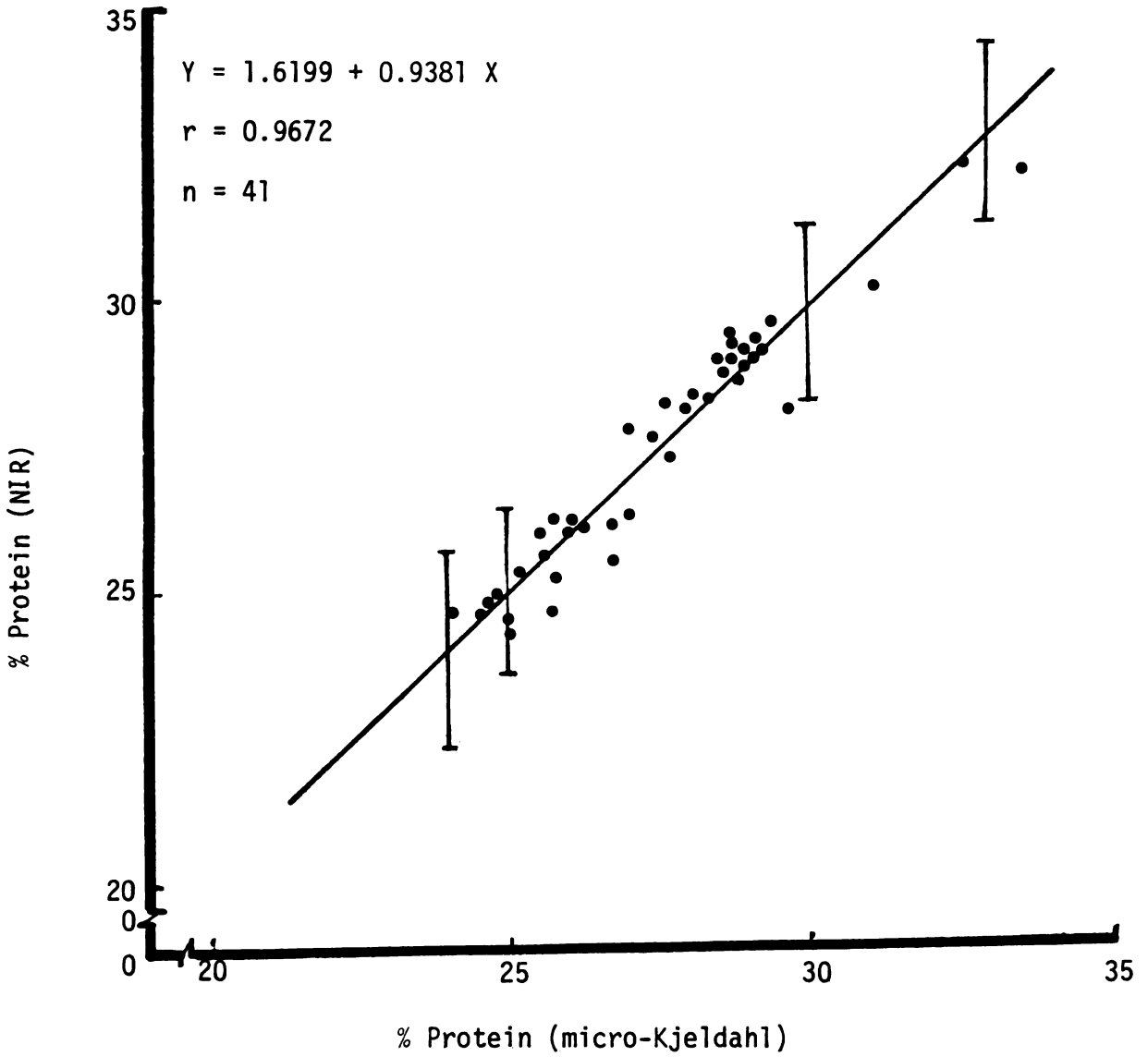

Figure 2. Relationship between % protein determined by micro-Kjeldahl and by the NIR methods in spray dried milk. Bars show the 95% confidence limits of the linear regression.

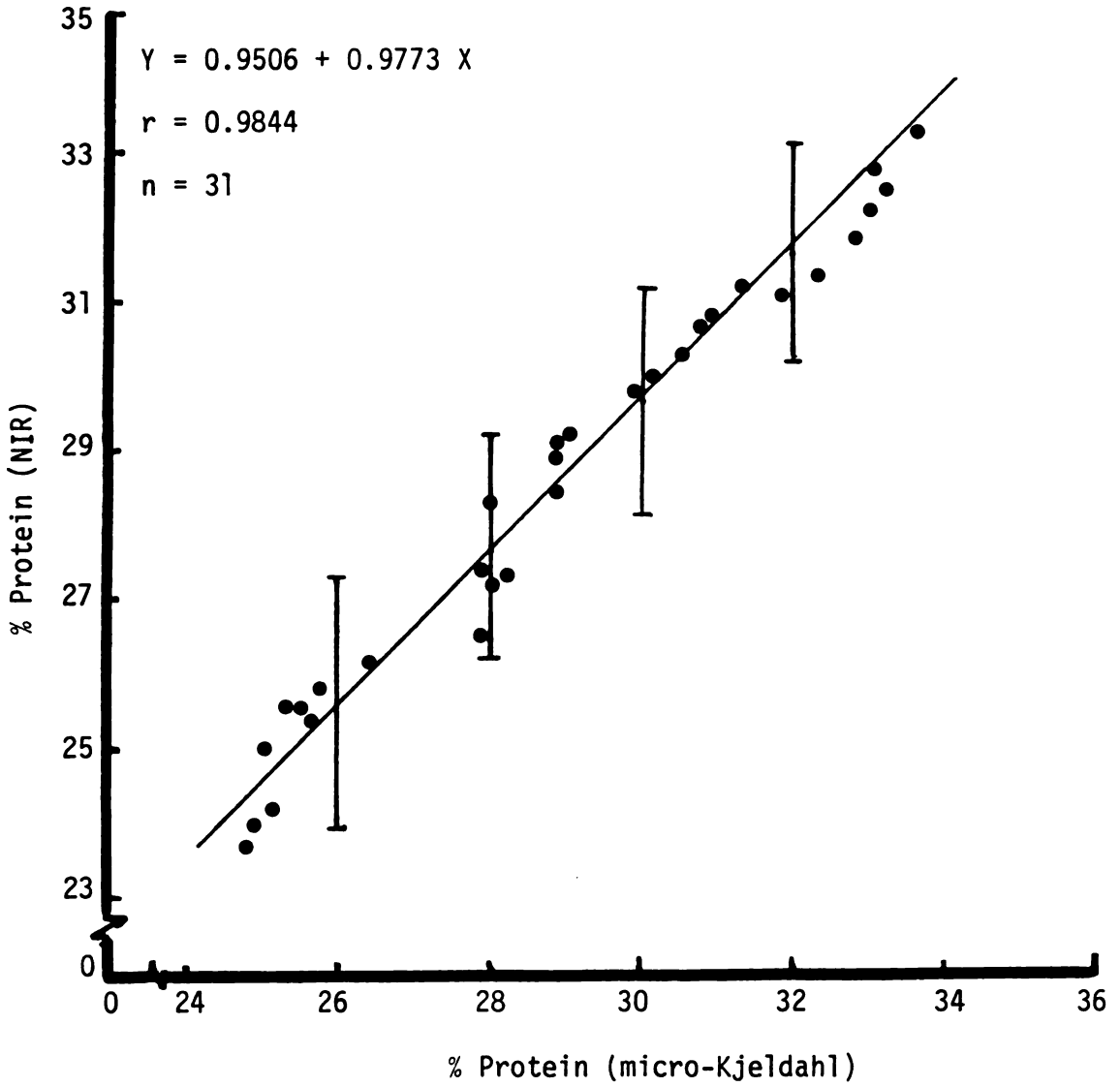

Figure 7. Relationship between % proteins determined by micro-Kjeldahl method and by the NIR method in dry-mixed milk. Bars show the 95% confidence limits of the linear regression.

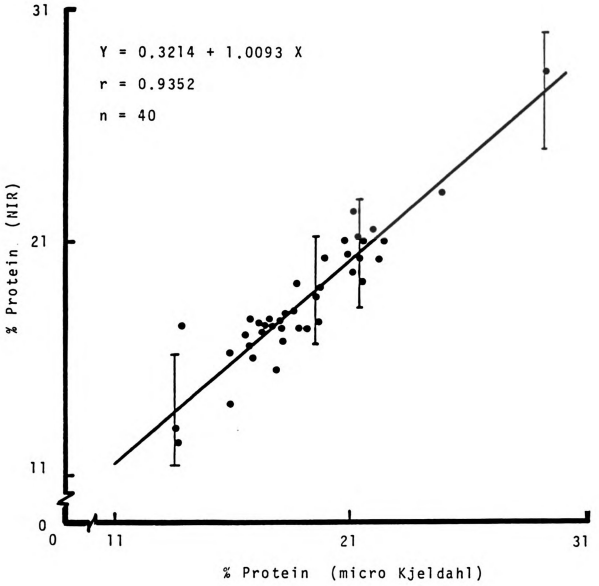

Figure 12. Relationship between % proteins determined by micro Kjeldahl method and by the NIR method in cheese powder. Bars show the 95% confidence limits of the linear regression.

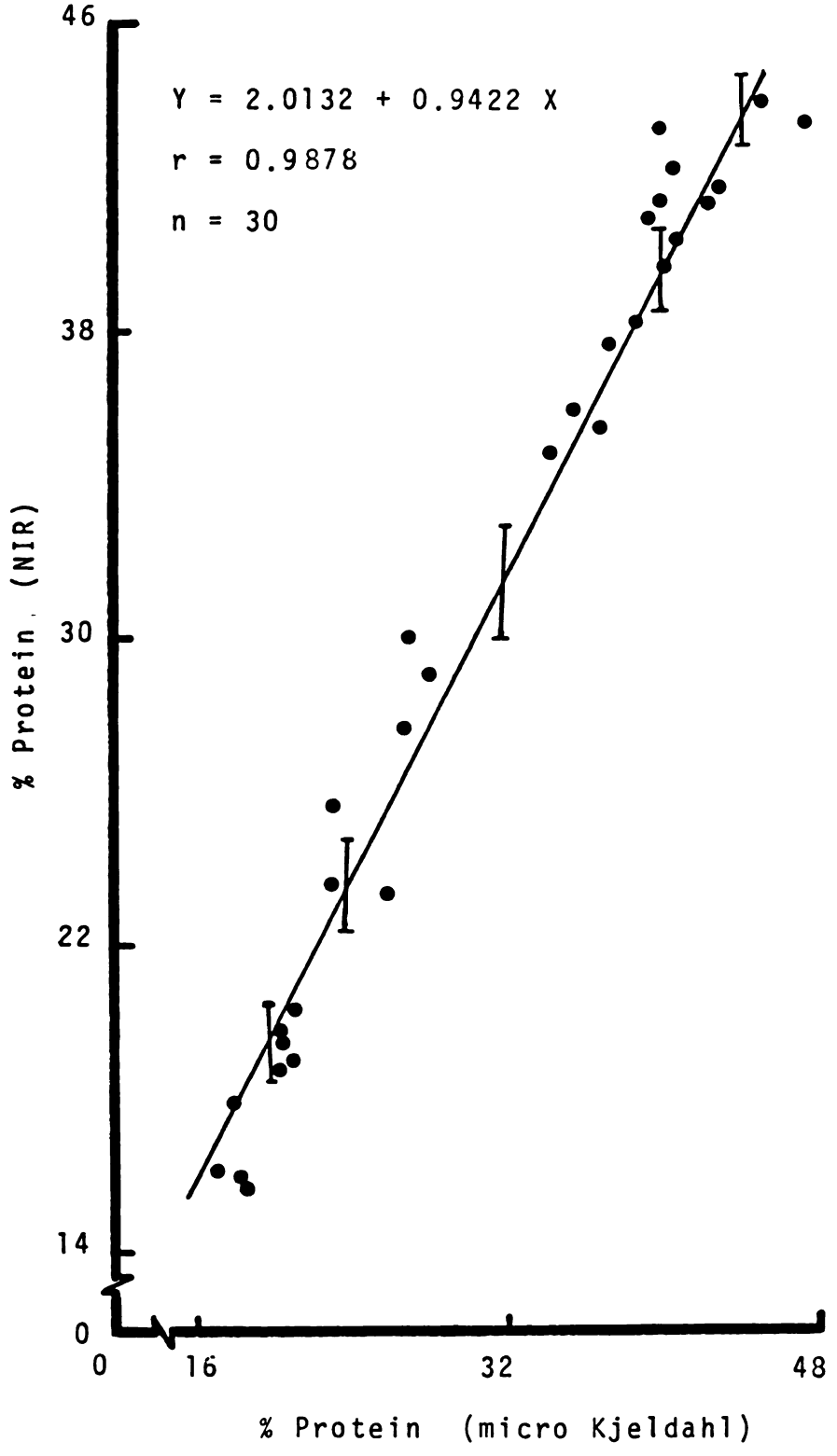

Figure 16. Relationship between % proteins determined by micro Kjeldahl and by the NIR in air-classified bean powder. Bars show the 95% confidence limits of the linear regression.

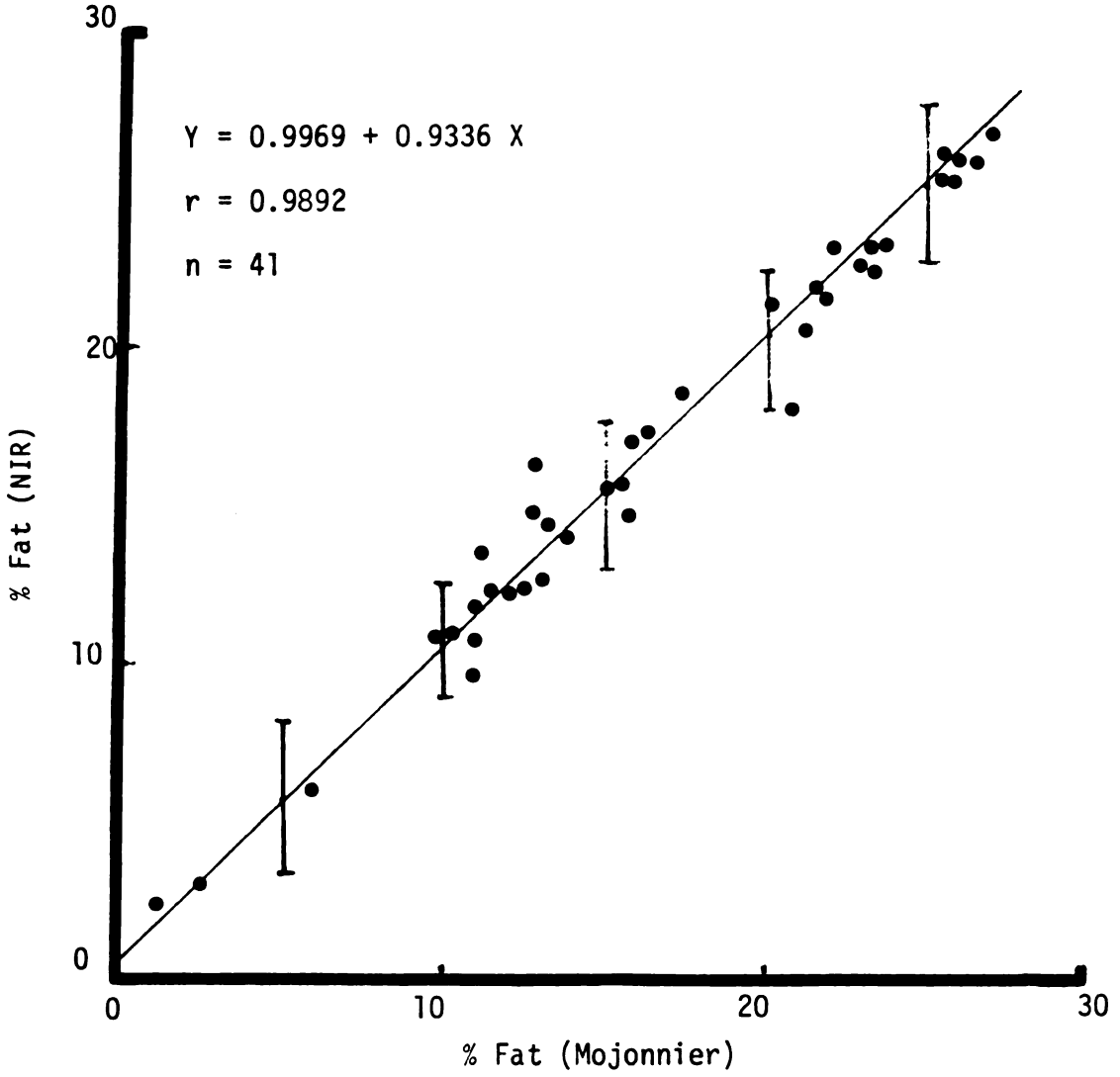

Figure 3. Relationship between % fat determined by the Mojonnier and by the NIR methods in spray dried milk. Bars show the 95% confidence limits of the linear regression.

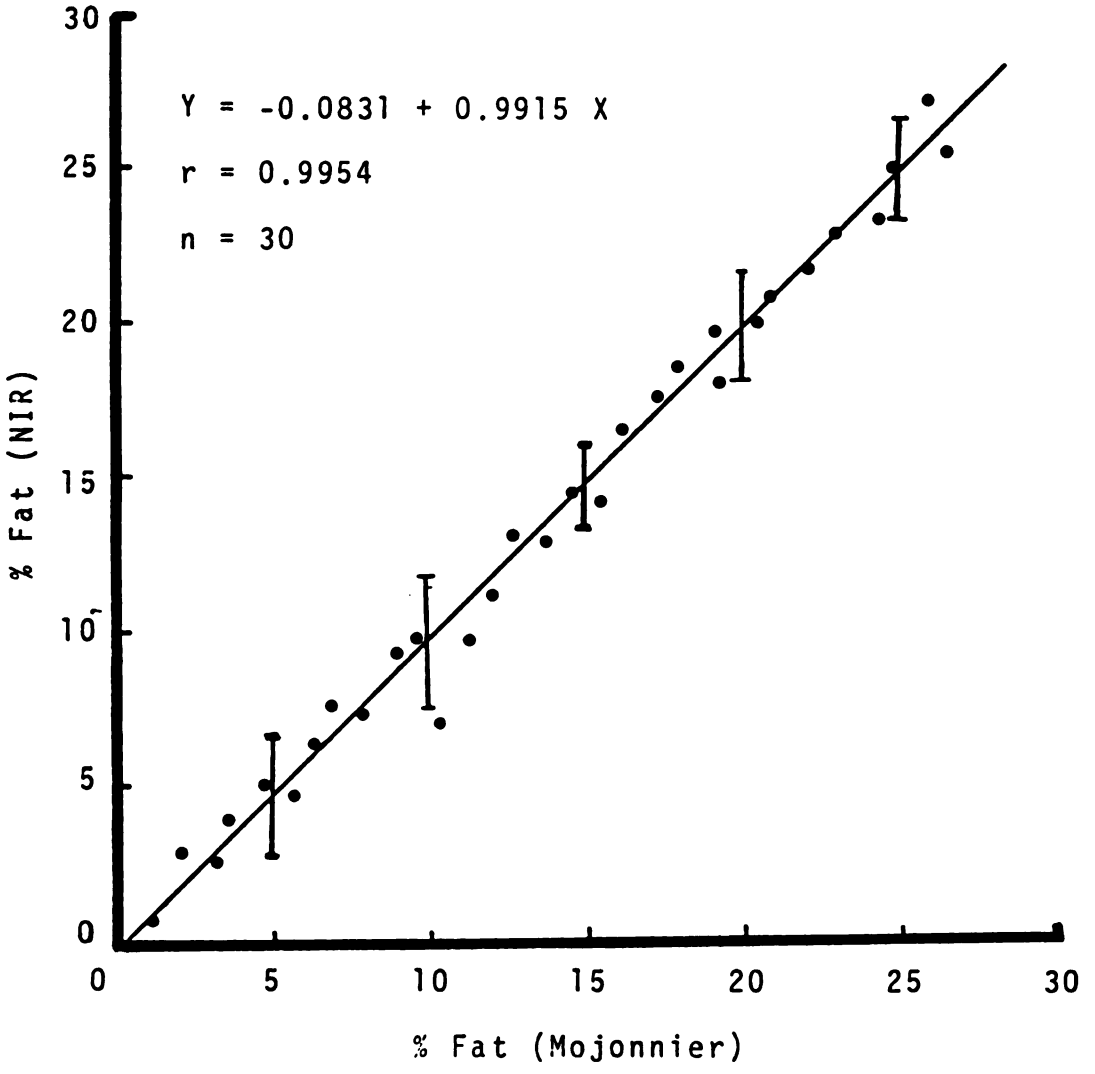

Figure 8. Relationship between % fat determined by Mojonnier method and by the NIR method in dry-mixed milk. Bars show the 95% confidence limits of the linear regression.

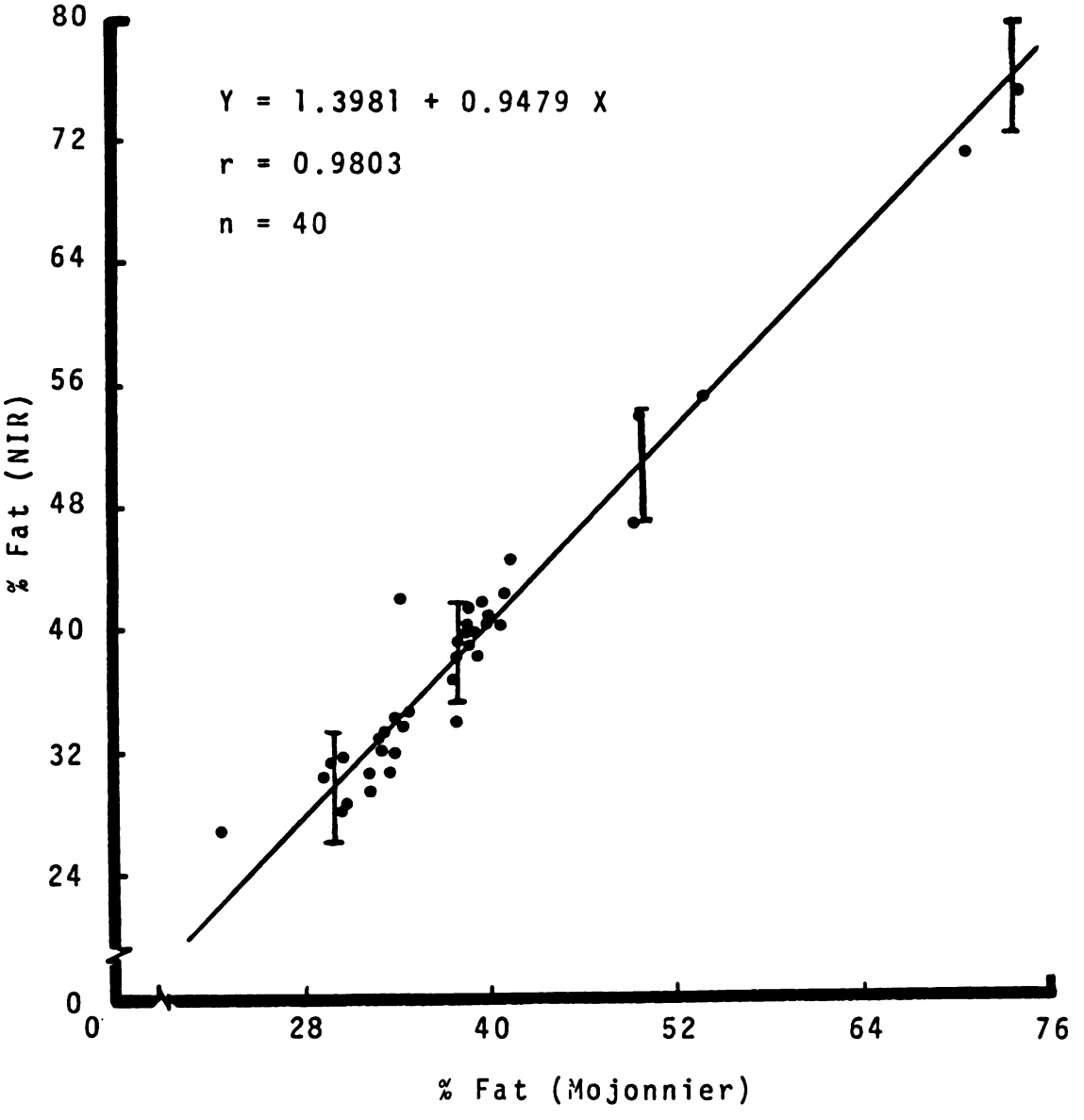

Figure 13. Relationship between % fat determined by Mojonnier method and by the NIR method in cheese powder. Bars show the 95% confidence limits of the linear regression.

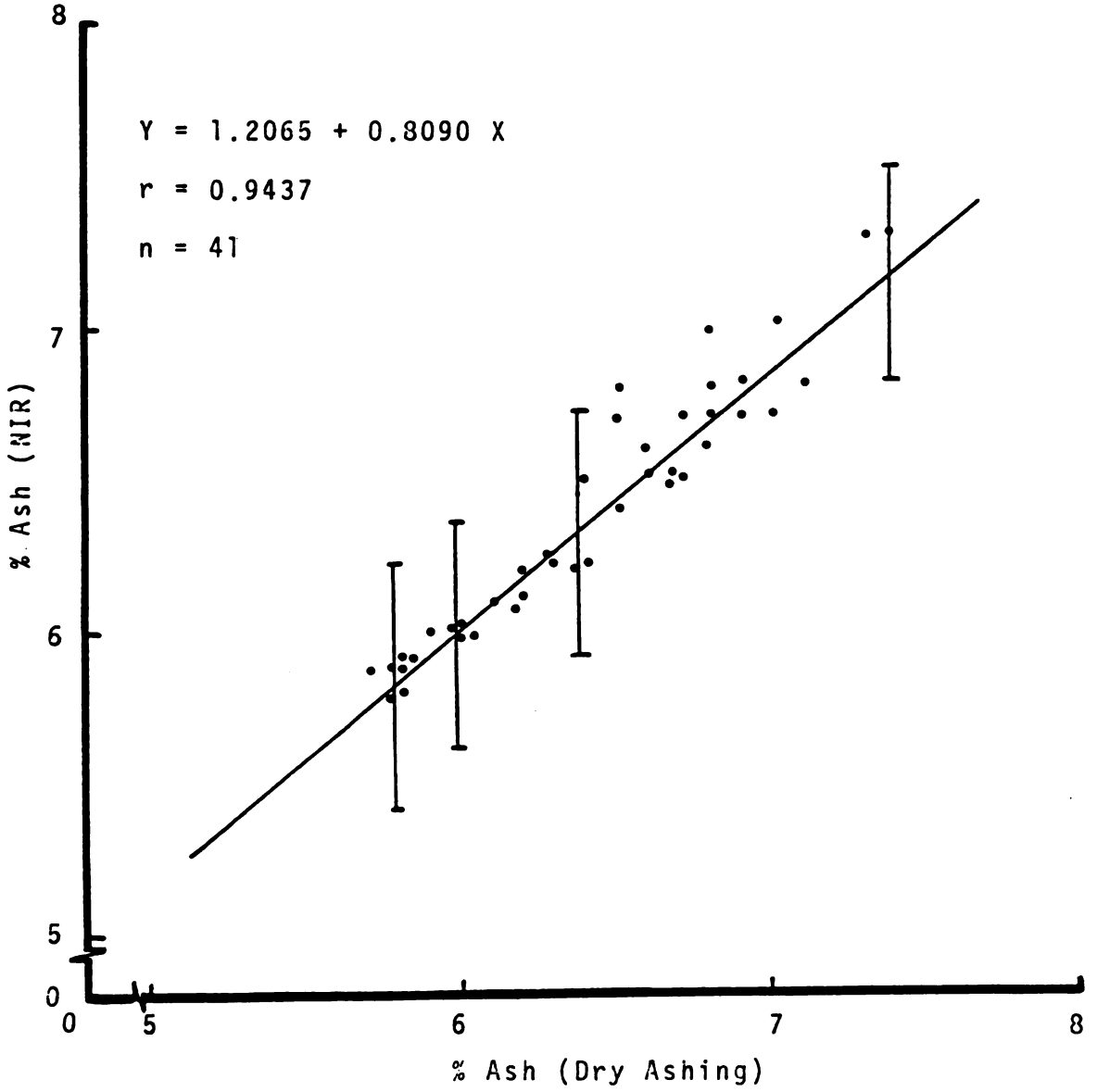

Figure 4. Relationship between % ash determined by dry ashing and by the NIR methods in spray dried milk. Bars show the 95% confidence limits of the linear regression.

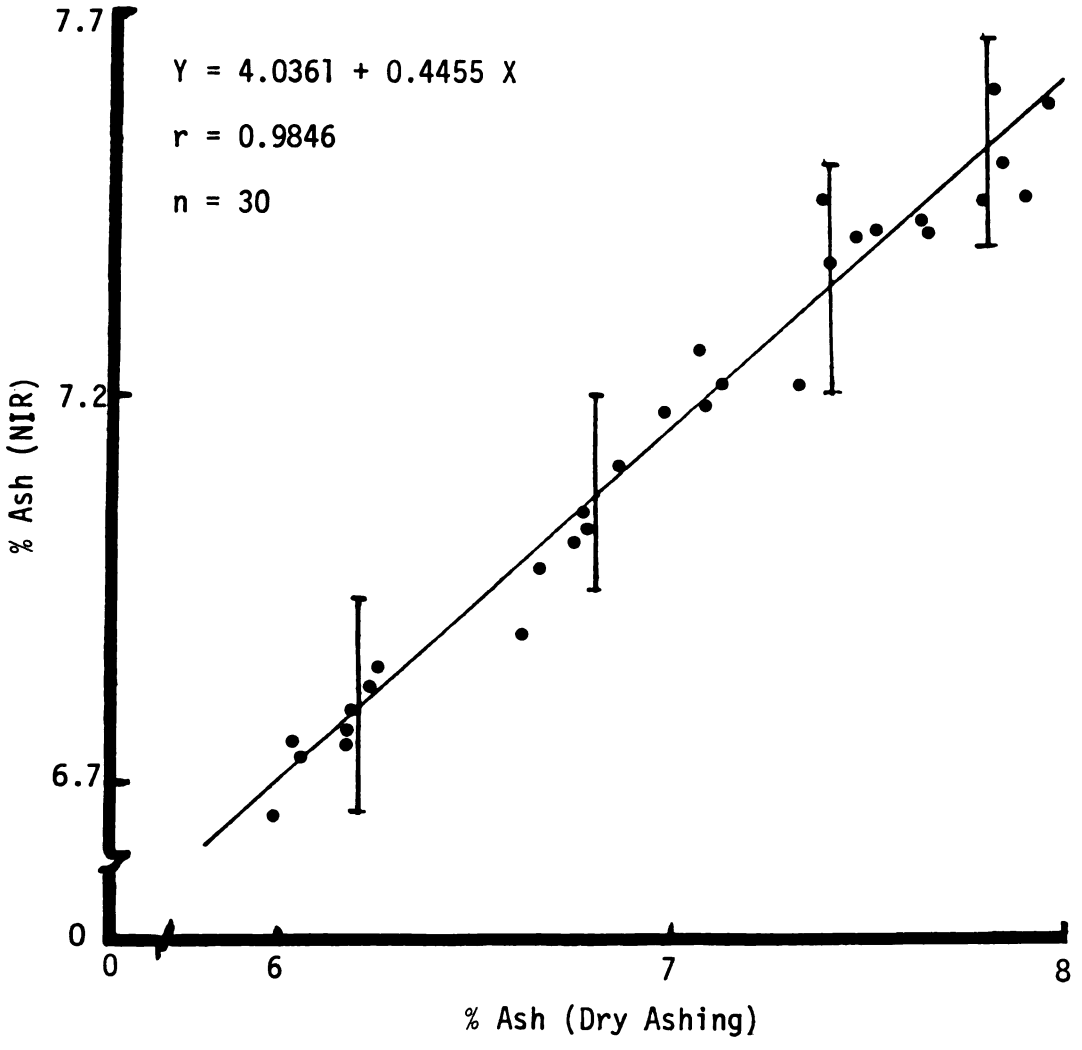

Figure 9. Relationship between % ash determined by dry ashing method and by the NIR method in dry-mixed milk. Bars show the 95% confidence limits of the linear regression.

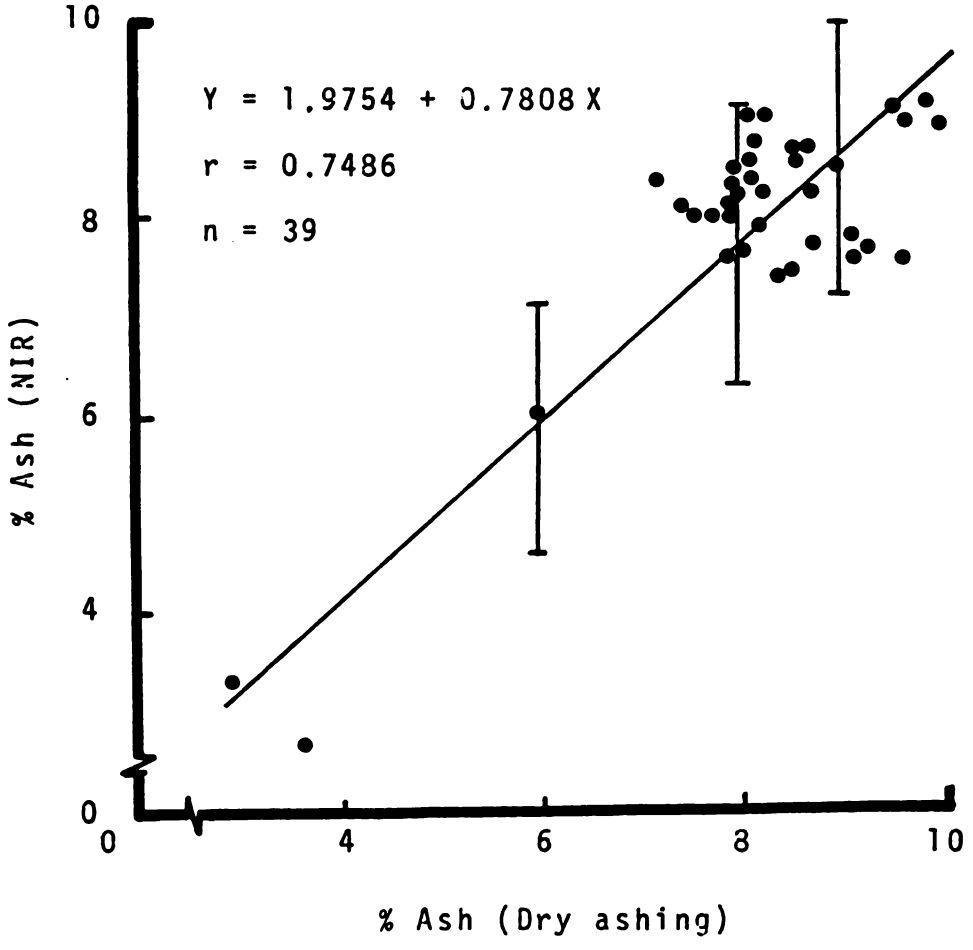

Figure 14. Relationship between % ash obtained by dry ashing method and by the NIR in cheese powder. Bars show the 95% confidence limits of the linear regression.

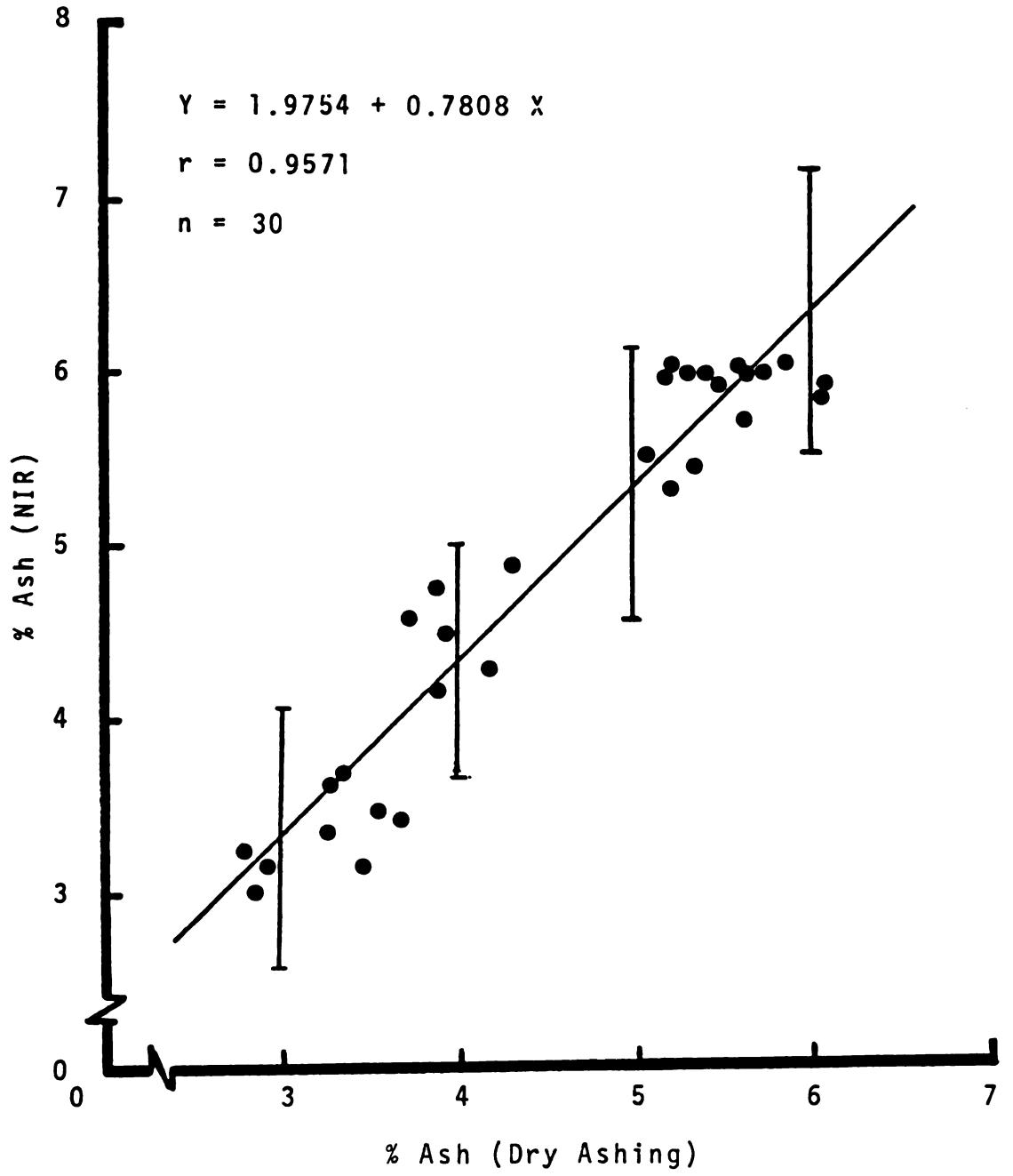

Figure 17. Relationship between % ash determined by the dry ashing and by the NIR methods in air-classified bean powder. Bars show the 95% confidence limits of the linear regression.

distribution of values, the vast majority of which were clustered in the 8% to 10% range.

The relationships between % lactose obtained by difference and those estimated by NIR instrument are shown in Figures 5 and 10. The r values were 0.9528 and 0.9959 for spray-dried and dry-mixed milk, respectively. The unknown samples of spray-dried milk, dry-mixed milk and bean flour were prepared in the same way as the calibrating samples and were subjected to NIR analysis using the pulse points and K values shown in Tables 5 and 6. The samples were later analyzed by the conventional methods. Table 25 shows the analysis by conventional and NIR methods.

The correlation coefficients are shown in Table 26, the moisture had lower r value in comparison to protein, fat, ash and lactose. The r values were 0.8384, 0.6017, and 0.7467 for spray-dried milk, dry-mixed milk and bean flour, respectively. The r values of the other food constituents were higher and ranged from 0.8625 to 0.9908.

The analysis of variance for the calibrating samples are shown in Tables 8 to 24. The F-value was calculated for each food component (moisture, protein, fat, ash and lactose). It was found that all F-values were smaller than F-tables at 10% probability level. It shows that there was no significant statistical difference at 10% probability level between the data obtained by conventional methods and those predicted by NIR, and also demonstrates the capability of NIR technology in predicting moisture, protein, fat, ash and lactose contents of the dairy products and bean flour.

The analysis of variance of the unknown dairy products and bean flour samples are shown in Tables 27 to 39. The F-value of all food constituents except moisture of spray-dried milk provide another

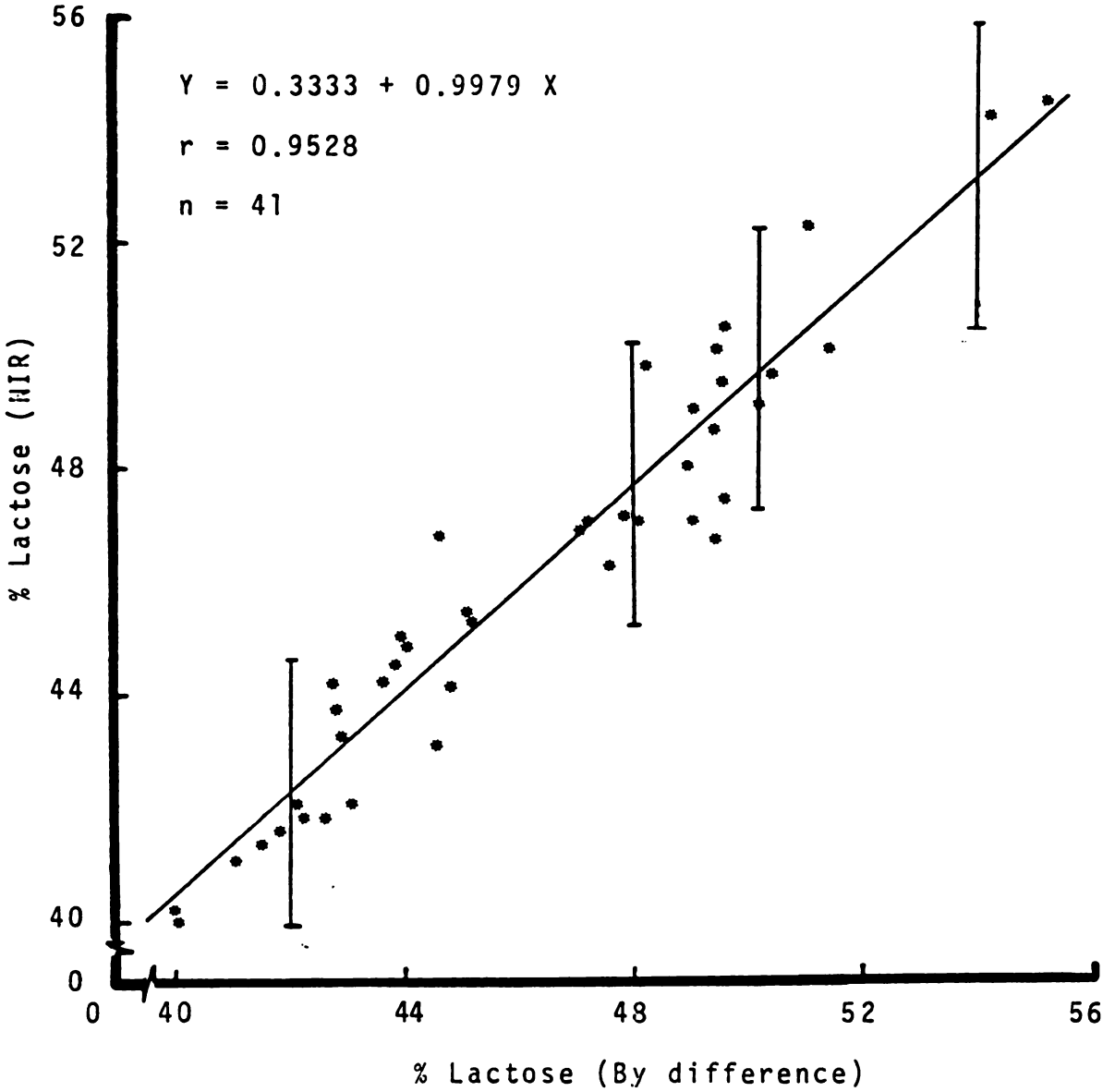

Figure 5. Relationship between % lactose calculated by difference and predicted by NIR method in spray dried milk. Bars show the 95% confidence of the linear regression.

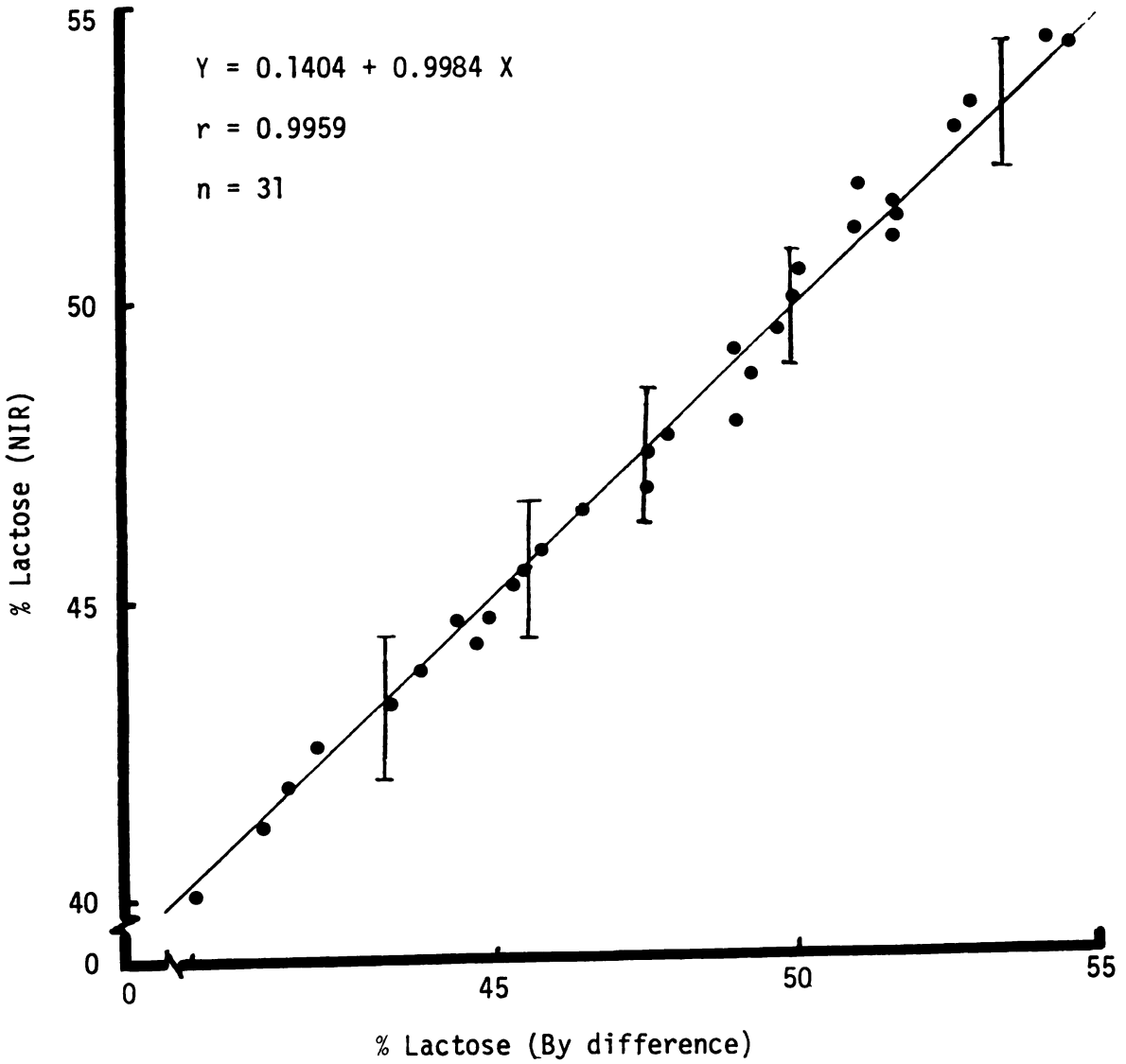

Figure 10. Relationsihp between % lactose calculated by difference and predicted by the NIR in dry-mixed milk. Bars show the 95% confidence limits of the linear regression.

Table 25. Analysis of spray dried milk, dry-mixed milk and air-classified bean flour unknown samples by conventional and NIR methods.

Sample Number	Conventional					NIR				
	Spray dried milk									
	% H_2O	% Protein	% Fat	% Ash	% Lactose (by difference)	% H_2O	% Protein	% Fat	% Ash	% Lactose
1	4.69	34.45	1.11	7.85	52.38	3.35	33.65	1.46	7.35	56.99
2	4.98	32.81	17.03	7.82	54.46	3.93	33.61	14.58	7.37	56.81
3	4.68	33.14	19.20	7.89	53.96	2.66	33.57	16.99	7.32	57.16
4	4.59	28.18	19.70	6.53	44.40	2.60	27.80	18.47	6.69	49.36
5	4.08	27.92	-	6.39	42.88	7.63	27.45	-	6.52	48.57
6	4.66	27.88	-	6.29	42.86	2.73	30.18	-	6.35	47.31
	Dry-mixed milk									
1	4.45	32.12	2.81	7.78	52.84	3.10	33.39	2.46	7.63	53.40
2	4.49	32.01	3.63	7.72	52.14	3.01	33.20	3.54	7.65	53.07
3	4.21	31.39	5.34	7.50	51.56	2.94	32.22	5.67	7.58	52.79
4	3.77	31.33	7.53	7.34	50.03	2.84	31.72	7.85	7.59	51.76
5	3.77	30.90	11.49	7.05	48.80	1.17	30.99	10.28	7.49	49.62
6	3.78	30.58	9.64	7.20	-	2.49	30.34	11.92	7.43	-
	Air-classified bean flour									
1	9.12	27.78	-	4.21		9.12	24.89	-	4.21	
2	8.33	42.80	-	6.20		9.04	37.19	-	5.79	
3	8.54	31.91	-	4.89		8.97	32.42	-	4.93	
4	8.40	22.69	-	3.41		8.49	21.99	-	3.65	
5	8.10	28.32	-	4.09		8.07	28.18	-	4.49	
6	-	32.02	-	4.29		-	32.58	-	4.69	

Table 26. Number of samples, correlation coefficient (r), slopes and Y-intercepts of the linear regression of unknown samples analyzed by conventional and NIR methods.

Food constituents	n	r	slope	y-Intercept
		Spray dried milk		
Moisture	6	0.8384	3.1079	-8.8589
Protein	6	0.9282	0.9023	3.3147
Fat	4	0.9908	0.8996	0.2969
Ash	6	0.9886	0.5767	2.8222
Lactose (by difference)	6	0.9882	0.8269	12.6024
		Dry-mixed milk		
Moisture	6	0.6017	1.2248	-2.4784
Protein	6	0.9945	1.9893	-30.4649
Fat	6	0.9514	1.0393	-0.0521
Ash	6	0.8625	0.2538	5.6759
Lactose (by difference)	5	0.9628	0.8975	6.2889
		Air-classified bean flour		
Moisture	5	0.7467	0.7071	2.5689
Protein	6	0.9394	0.7789	5.4563
Ash	6	0.9699	0.7346	1.3100

Table 8. Analysis of variance of moisture content of spray dried milk analyzed by conventional and NIR methods.

Source of variance	Degree of freedom	Sum of squares	Mean square	F-statistic	F-tabulated α=0.10
Treatment	1	0.1018	0.1018	0.0754	2.75
Experimental error	80	0.0169	1.3506		
Total	81	0.0179	1.4524		

Table 9. Analysis of variance of protein content of spray dried milk.

Source of variance	Degree of freedom	Sum of squares	Mean square	F-statistic	F-tabulated α=0.10
Treatment	1	0.1598	0.1598	0.0368	2.75
Experimental error	80	0.0543	4.3415		
Total	81	0.0556	4.5013		

Table 10. Analysis of variance of fat content of spray dried milk.

Source of variance	Degree or freedom	Sum of squares	Mean square	F-statistic	F-tabulated α = 0.10
Treatment	1	0.3367	0.3367	0.0080	2.75
Experimental error	80	0.5249	41.9921		
Total	81	0.5226	42.3288		

Table 11. Analysis of variance of ash content in spray dried milk.

Source of variance	Degree of freedom	Sum of squares	Mean square	F-statistic	F-tabulated α=0.10
Treatment	1	0.1980	0.1980	0.8536	2.75
Experimental error	80	0.0029	0.2320		
Total	81	0.0053	0,4300		

Table 12. Analysis of variance of lactose content of spray dried milk.

Source of variance	Degree of freedom	Sum of squares	Mean square	F-statistic	F-tabulated α=0.10
Treatment	1	1.1139	1.1139	0.0879	2.75
Experimental error	78	0.1624	12.6674		
Total	79	0.1745	13.7813		

Table 13. Analysis of variance of moisture content of dry-mixed milk analyzed by conventional and NIR methods.

Source of variance	Degree of freedom	Sum of squares	Mean square	F-statistic	F-tabulated α=0.10
Treatment	1	0.0025	0.0025	0.0382	2.79
Experimental error	60	0.0011	0.0675		
Total	61	0.0012	0.0700		

Table 14. Analysis of variance of protein content of dry-mixed milk analyzed by conventional and NIR methods.

Source of variation	Degree of freedom	Sum of squares	Mean square	F-statistic	F-tabulated α=0.10
Treatment	1	1.4130	1.4130	0.1730	2.79
Experimental error	60	0.1361	8.1635		
Total	61	0.1569	9.5765		

Table 15. Analysis of variance of fat content of dry-mixed milk analyzed by conventional and NIR methods.

Source of variation	Degree of freedom	Sum of squares	Mean square	F-statistic	F-tabulated α=0.10
Treatment	1	1.4342	1.4342	0.0242	2.79
Experimental error	60	0.9872	59.2342		
Total	61	0.9946	60.6684		

Table 16. Analysis of variance of ash content of dry-mixed milk analyzed by conventional and NIR methods.

Source of variation	Degree of freedom	Sum of squares	Mean square	F-statistic	F-tabulated α=0.10
Treatment	1	0.5748	0.5748	2.1180	2.79
Experimental error	60	0.0045	0.2714		
Total	61	0.0139	0.8462		

Table 17. Analysis of variance of lactose content of dry-mixed milk analyzed by conventional and NIR methods.

Source of variance	Degree of freedom	Sum of squares	Mean square	F-statistic	F-tabulated α=0.10
Treatment	1	0.7436	0.7436	0.0441	2.79
Experimental error	60	0.2807	16.8429		
Total	61	0.2883	17.5865		

Table 18. Analysis of variance of moisture content of cheese powder analyzed by conventional and NIR methods.

Source of variance	Degree of freedom	Sum of squares	Mean square	F-statistic	F-tabulated α=0.10
Treatment	1	0.1110	0.1110	0.2005	2.75
Experimental error	78	0.0071	0.5536		
Total	79	0.0084	0.6646		

Table 19. Analysis of variance of protein content of cheese powder analyzed by conventional and NIR methods.

Source of variance	Degree of freedom	Sum of squares	Mean square	F-statistic	T-tabulated α=0.10
Treatment	1	4.8609	4.8609	0.5683	2.75
Experimental error	78	0.1097	8.5529		
Total	79	0.1698	13.4138		

Table 20. Analysis of variance of fat content of cheese powder analyzed by conventional and NIR methods.

Source of variance	Degree of freedom	Sum of squares	Mean square	F-statistic	F-tabulated α=0.10
Treatment	1	7.9632	7.9632	0.0800	2.75
Experimental error	78	1.2751	99.4540		
Total	79	1.3597	107.4172		

Table 21. Analysis of variance of ash content of cheese powder analyzed by contentional and NIR methods.

Source of variance	Degree of freedom	Sum of squares	Mean square	F-statistic	F-tabulated α=0.10
Treatment	1	0.2063	0.2063	0.1170	2.75
Experimental error	74	0.0238	1.7629		
Total	75	0.0263	1.9692		

Table 22. Analysis of variance of moisture content of air-classified bean flour analyzed by conventional and NIR methods.

Source of variance	Degree of freedom	Sum of squares	Mean square	F-statistic	F-tabulated α=0.10
Treatment	1	0.4018	0.4018	0.5639	2.84
Experimental error	58	0.0123	0.7125		
Total	59	0.0189	1.1143		

Table 23. Analysis of variance of protein content of air-classified bean flour analyzed by conventional and NIR methods.

Source of variance	Degree of freedom	Sum of squares	Mean square	F-statistic	F-tabulated α=0.10
Treatment	1	0.7216	0.7216	0.0072	2.84
Experimental error	58	1.7262	100.1174		
Total	59	1.7091	100.8390		

Table 24. Analysis of variance of ash content of air-classified bean flour analyzed by contentional and NIR methods.

Source of variance	Degree of freedom	Sum of squares	Mean square	F-statistic	T-tabulated α=0.10
Treatment	1	1.4322	1.4322	1.1724	2.84
Experimental error	58	0.0211	1.2215		
Total	59	0.0451	2.6537		

Table 27. Analysis of variance of moisture content of spray-dried milk unknown samples analyzed by contentional and NIR methods.

Source of variance	Degree of freedom	Sum of squares	Mean square	F-statistic	F-tabulated α=0.05
Treatment	1	3.3920	3.3920	7.9045	4.96*
Experimental error	10	0.0429	0.4291		
Total	11	0.3474	3.8211		

*Significant at 5% probability level.

Table 28. Analysis of variance of protein content of spray dried milk unknown samples analyzed by conventional and NIRS methods.

Source of variance	Degree of freedom	Sum of squares	Mean square	F-statistic	F-tabulated α=0.10
Treatment	1	0.2945	0.2945	0.0325	3.29
Experimental error	10	0.9044	9.0437		
Total	11	0.8489	9.3382		

Table 29. Analysis of variance of fat content of spray dried milk unknown samples analyzed by conventional and NIR methods.

Source of variance	Degree of freedom	Sum of squares	Mean square	F-statistic	F-tabulated α=0.10
Treatment	1	3.8364	3.8364	0.0553	3.29
Experimental error	10	11.5566	69.3398.		
Total	11	10.4537	73.1762		

Table 30. Analysis of variance of ash content of spray dried milk unknown samples analyzed by conventional and NIR methods.

Source of variance	Degree of freedom	Sum of squares	Mean square	F-statistic	F-tabulated α=0.10
Treatment	1	0.1140	0.1140	0.2671	3.29
Experimental error	10	0.0427	0.4269		
Total	11	0.0492	0.5409		

Table 31. Analysis of variance of lactose content of spray dried milk unknown samples calculated by difference and predicted by NIR method.

Source of variance	Degree of freedom	Sum of squares	Mean square	F-statistic	F-tabulated α=0.10
Treatment	1	53.1723	53.1723	1.9472	3.29
Experimental error	10	2.7304	27.3043		
Total	11	7.3161	80.4766		

Table 32. Analysis of variance of moisture content of dry-mixed milk unknown samples analyzed by conventional and NIR methods.

Source of variance	Degree of freedom	Sum of squares	Mean square	F-statistic	F-tabulated α=0.10
Treatment	1	2.7840	2.7840	1.4676	3.29
Experimental error	10	0.1897	1.8969		
Total	11	0.4256	4.6809		

Table 33. Analysis of variance of protein content of dry-mixed milk unknown samples analyzed by conventional and NIR methods.

Source of variance	Degree of freedom	Sum of squares	Mean square	F-statistic	F-tabulated α=0.10
Treatment	1	1.0384	1.0384	1.1425	3.29
Experimental error	10	0.0909	0.9088		
Total	11	0.1770	1.9472		

Table 34. Analysis of variance of fat content of dry-mixed milk unknown samples analyzed by conventional and NIR methods.

Source of variance	Degree of freedom	Sum of squares	Mean square	F-statistic	F-tabulated α=0.10
Treatment	1	0.1365	0.1365	0.0106	3.29
Experimental error	10	1.2870	12.8699		
Total	11	1.1824	13.0064		

Table 35. Analysis of variance of ash content of dry-mixed milk unknown samples analyzed by conventional and NIR methods.

Source of variance	Degree of freedom	Sum of squares	Mean square	F-statistic	F-tabulated α=0.10
Treatment	1	0.0507	0.0507	1.1192	3.29
Experimental error	10	0.0045	0.0452		
Total	11	0.0087	0.0959		

Table 36. Analysis of variance of lactose content of dry-mixed milk unknown samples calculated by difference and predicted by NIR method.

Source of variance	Degree of freedom	Sum of squares	Mean square	F-statistic	F-tabulated α=0.10
Treatment	1	2.7667	2.7667	1.0988	3.46
Experimental error	8	0.3147	2.5178		
Total	9	0.5872	5.2845		

Table 37. Analysis of variance of moisture content of air-classified bean flour unknown samples analyzed by conventional and NIR methods.

Source of variance	Degree of freedom	Sum of squares	Mean square	F-statistic	F-tabulated α=0.10
Treatment	1	0.1440	0.1440	0.8326	3.46
Experimental error	8	0.0216	0.1729		
Total	9	0.0352	0.3169		

Table 38. Analysis of variance of protein content of air-classified bean flour unknown samples analyzed by conventional and NIR methods.

Source of variation	Degree of freedom	Sum of squares	Mean square	F-statistic	F-tabulated α=0.10
Treatment	1	5.6994	5.6994	0.1483	3.29
Experimental error	10	3.8421	38.4210		
Total	11	4.0110	44.1204		

Table 39. Analysis of variance of ash content of air-classified bean flour unknown samples analyzed by conventional and NIR methods.

Source of variance	Degree of freedom	Sum of squares	Mean square	F-statistic	F-tabulated α=0.10
Treatment	1	0.0374	0.0374	0.0525	3.29
Experimental error	10	0.0712	0.7120		
Total	11	0.0681	0.7494		

evidence that both NIR and conventional procedures are identical and there is no significant statistical difference at the 10% probability level. The F-value for moisture in the spray-dried milk (Table 27) was significant at the 5% probability level, and might be due to the changes in moisture content from the time of calibration of GQA-41. To test the capability of GQA-41 prediction, some commercial bean flours were tested for their moisture, protein and ash contents. The samples were subjected to NIR analysis using the corresponding pulse points and K values (Tables 5 and 6), and later subjected to conventional analysis. The discrepancies were small.

Table 40 shows the analysis of commercial bean flour by conventional and NIR methods. The commercial bean flour samples were ground for 2 minutes using the Dickey-john grain grinder to see any differences due to grinding. The samples were subjected to NIR analysis and the data are shown in Table 40. The extra 2 minutes of grinding did not improve the predictability.

Eleven commercially dried milk powder samples were obtained from the Michigan State Department of Agriculture in E. Lansing. Their moisture, protein, fat, ash and lactose content was estimated by NIR using the pulse points and K values of the spray-dried milk samples, and were later analyzed by the conventional methods. It was found that the discrepancies between the NIR and conventional values were very large.

The pulse points and K values of the dry-mixed milk (Tables 5 and 6) were used, but the differences between the estimated and analytical values were large. This may be due to the different physical characteristics of the commodities resulting in different reflectance spectra.

Table 40. Analysis of commercial navy bean air-classified flour by conventional and NIR methods before and after grinding for 2 minutes.

Bean flour fraction	Before grinding			After grinding		
Food Constituent	Conventional	NIR	Difference	Conventional	NIR	Difference
Navy bean Fraction II						
$\%H_2O$	8.21	6.97	+1.24	8.21	6.35	+1.86
%Protein	16.23	16.39	-0.16	16.23	16.60	-0.37
%Ash	2.74	2.96	-0.22	2.74	2.93	-0.19
Navy bean Fraction I + II						
$\%H_2O$	7.77	8.09	-0.32	7.77	8.08	-0.31
%Protein	41.11	40.30	+0.81	41.11	40.81	+0.30
%Ash	5.65	5.99	-0.34	5.65	6.02	-0.37
Navy bean Fraction II						
$\%H_2O$	8.27	7.11	+1.16	8.27	6.84	+1.43
%Protein	16.15	16.60	-0.45	16.15	16.02	+0.13
%Ash	2.73	2.96	-0.23	2.73	3.16	-0.43

Milk, in industry is never dried with its original moisture content, but evaporated to concentrations of 45-52% total solids prior to spray drying, and hot-air temperature up to 750°F (400°C) may be used for drying with secondary cool air introduced lower in the drying chamber. The temperature of 480°F (250°C) appears to be the maximum without the cool air system. Skim milk powder is dusty, and to overcome this problem, the powder is agglomerated thereby improving its wetability and dispersibility characteristics. When the eleven commercial milk powders were used for calibration of the GQA-41, pulse points and K-values were obtained which were different from those of the spray-dried and dry-mixed samples prepared in the laboratory. This indicates that the spectral characteristics of the commercial and laboratory samples were different and explains the failure of predicting the composition of the former using the calibration data of the latter. However, when the calibration data of the eleven commercial samples (Tables 42 and 43) were used to predict the composition of two unknown commercial samples (Table 41), the agreement between analytical and predicted values for protein, fat, ash and lactose were very good for the 13 commercial samples of Table 41. The correlation coefficients between the analytical and NIR data were 0.8507, 0.9878, 0.992, 0.9949 and 0.9888 for moisture, protein, fat, ash and lactose contents, respectively (Table 44).

The analysis of variance for each food constituent of the commercially dried milk are shown in Tables 45 to 49. F-values for moisture, protein, fat, ash and lactose were not statistically significant at 10% probability level.

Table 41. Analysis of commercially dried milk samples by conventional and NIR methods.

Sample Number	Sample Identification	Conventional					NIR				
		% H_2O	% Protein	% Fat	% Ash	% Lactose (by difference)	% H_2O	% Protein	% Fat	% Ash	% Lactose
1	IGA Tablerite	4.15	32.05	1.06	7.76	54.98	4.18	32.47	0.57	7.77	54.24
2	IGA Tablerite	4.27	32.89	0.97	7.67	54.20	3.97	32.46	0.62	7.80	55.20
3	Kroger's	3.89	31.92	1.04	7.61	55.54	4.01	31.58	0.85	7.69	56.35
4	A&P	3.84	33.37	1.11	7.89	53.79	3.63	33.77	1.35	7.84	51.95
5	Carnation	2.60	31.79	1.14	7.55	56.92	2.42	31.19	0.97	7.66	56.20
6	Verndale Products	3.48	25.23	26.26	5.98	39.05	3.65	25.48	27.48	5.92	35.32
7	" "	3.82	25.88	26.73	5.97	37.60	3.90	25.51	26.30	5.95	37.75
8	" "	3.73	25.58	26.65	6.07	37.97	3.57	24.69	27.11	5.93	38.32
9	" "	3.69	25.48	26.72	6.11	38.00	4.37	24.09	25.91	6.01	37.85
10	" "	3.67	25.14	26.51	6.04	38.64	3.64	25.35	26.33	5.99	38.51
11	" "	3.61	25.22	26.53	5.91	38.73	3.51	25.04	26.51	6.08	38.96
12*	A&P Carnation	3.74	31.56	1.19	7.81	55.70	2.86	31.27	1.07	7.62	55.65
13*	ShopRite nonfat dry milk	4.10	33.48	1.14	7.76	53.52	2.06	33.18	0.74	7.95	53.74

*Unknown samples.

Table 42. The best four reflectance pulse points selected by regression analysis for commercially dried milk.

Food Constituent	P_0	P_1	P_2	P_3	P_4
% Moisture	80	288	611	903	962
% Protein	80	264	286	601	961
% Fat	80	614	915	954	978
% Ash	80	248	309	620	973
% Lactose	80	597	624	906	964

Table 43. Multilinear regression coefficients (K values) used for NIR analysis of commercially dried milk.

Food Constituent	K_0	K_1	K_2	K_3	K_4
% Moisture	4.7	673.0	1762.7	429.2	-171.5
% Protein	26.5	1311.3	2113.4	-2168.1	-245.8
% Fat	0.70	-343.4	266.4	365.9	-536.1
% Ash	7.3	13.4	-163.2	-163.2	105.5
% Lactose	75.6	-2648.7	-2124.8	-741.8	1224.2

Table 44. Number of samples (n), correlation coefficients (r), slope, y-intercepts and, the equation of the linear regression between the conventional and NIR values of commercially dried milk.

Food Constituent	n	r	Slope	y-intercept	Equation
Moisture	11	0.8507	1.0021	0.0013	Y= 0.0013 + 1.0021X
Protein	11	0.9878	1.0249	-0.9085	Y=-0.9085 + 1.0249X
Fat	11	0.9992	1.0088	-0.1976	Y=-0.1976 + 1.0088X
Ash	11	0.9949	1.0436	-0.2883	Y=-0.2883 + 1.0436X
Lactose	11	0.9888	1.0119	-0.9787	Y=-0.9787 + 1.0019X

Table 45. Analysis of variance of moisture content of commercially dried milk analyzed by conventional and NIR methods.

Source of variance	Degree of freedom	Sum of squares	Mean of squares	F-statistic	F-Tabulated α=0.10
Treatment	1	0.2002	0.2002	0.4021	2.97
Experimental error	20	9.9563	0.4978		
Total	21	10.1565	0.6980		

Table 46. Analysis of variance of protein content of commercially dried milk analyzed by conventional and NIR methods.

Source of variance	Degree of freedom	Sum of squares	Mean of squares	F-statistic	F=tabulated α=0.10
Treatment	1	0.3876	0.3876	0.0272	2.97
Experimental error	20	284.8026	14.2401		
Total	21	285.1903	14.6277		

Table 47. Analysis of variance of fat content of commercially dried milk analyzed by conventional and NIR methods.

Source of variance	Degree of freedom	Sum of squares	Mean of squares	F-statistic	F-tabulated α=0.10
Treatment	1	0.0236	0.0236	0.0001	2.97
Experimental error	20	3582.227	179.1113		
Total	21	3582.2506	179.1349		

Table 48. Analysis of variance of ash content of commercially dried milk analyzed by conventional and NIR methods.

Source of variance	Degree of freedom	Sum of squares	Mean of squares	F-statistic	F-tabulated α=0.10
Treatment	1	0.0001	0.0001	0.00012	2.97
Experimental error	20	16.4244	0.8212		
Total	21	16.4245			

Table 49. Analysis of variance of lactose content determined by difference and NIR method.

Source of variance	Degree of freedom	Sum of squares	Mean of squares	F-statistic	F-tabulated α=0.10
Treatment	1	1.0341	1.0341	0.0131	2.97
Experimental error	20	1582.8499	79.1424		
Total	21	1583.8790	80.1765		

SUMMARY

Forty one spray-dried milk samples prepared in the laboratory were analyzed for moisture, protein, fat, ash and lactose contents by conventional methods and by NIR.

The Neotec GQA-41 instrument was used for the NIR method. The optimum pulse points and the regression coefficients (K-values) of each constituent were obtained and used to calibrate the instrument. The correlation coefficients (r values) between analytical and NIR values of the samples were 0.7889, 0.9672, 0.9892, 0.9437 and 0.9528 for moisture, protein, fat, ash and lactose contents, respectively. Some unknown samples prepared in a similar way were subjected to analysis by the two procedures. The r values for moisture, protein, fat, ash and lactose determined by conventional methods and estimated by NIR were 0.8384, 0.9282, 0.9908, 0.9886 and 0.9882, respectively.

Thirty one milk powders prepared by mixing whole milk powder and skim milk powder were analyzed by both procedures. With pulse points and K values characteristic for these powders the following r values were obtained: 0.8208, 0.9044, 0.9954, 0.9846 and 0.9959 for moisture, protein, fat, ash and lactose, respectively. Some unknown samples prepared in the laboratory by dry-mixing were analyzed by both techniques and the r values were 0.6017, 0.9945, 0.9514, 0.8625 and 0.9628 for moisture, protein, fat, ash and lactose, respectively. The pulse points and K values of the dry-mixed powders were different from those of the

spray-dried samples,

Forty cheese powder samples obtained from the industry were analyzed by both procedures. The r values were 0.7494, 0.9352, 0.9803 and 0.7486 for moisture, protein, fat and ash, respectively.

Thirty samples of air-classified bean flour were analyzed by both methods. The correlation coefficient values were 0.8971, 0.9878 and 0.9571 for moisture, protein and ash content, respectively. The r values for six unknown bean flours were 0.7467, 0.9394, and 0.9699 for moisture, protein and ash, respectively. The predictability of the NIR method for three commercial bean flours was very good.

Eleven commercially dried milk samples were analyzed by both techniques. The reflectance characteristics were different from those obtained from the milk powders prepared in the laboratory, apparently due to different drying treatments. However, when the instrument was calibrated with eleven commercial milk powders, the r values were 0.8507, 0.9878, 0.9878, 0.992 and 0.9949 for moisture, protein, fat, ash and lactose, respectively.

Two additional commercial dry milk samples were analyzed by NIR and conventional procedures and showed satisfactory agreement between analytical and predicted values.

Analysis of variance was performed for all food constituents in the dairy and bean samples. The calculated F-values were smaller than F-table in all products, (except for moisture in unknown spray-dried milk), indicating no significant difference between the conventional and NIR methods.

This work indicates that the NIR technology holds considerable promise for the analysis of dairy and bean powders by a quick, non-destructive, and non-polluting method.

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