

# Measuring High Moisture Content of Cereal Grains by Pulsed Nuclear Magnetic Resonance<sup>1</sup>

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ABSTRACT

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Moisture in barley, corn, grain sorghum, and wheat was determined for two crop years by methods based on oven air-drying and pulsed nuclear magnetic resonance (PNMR) spectroscopy. The PNMR method was

satisfactory for determining from 15 to 40% moisture. Calibration requirements for moisture determinations by PNMR in naturally high moisture grain differed from those for conditioned high moisture grain.

The significance and measurement of moisture in cereal grains have been reviewed by Hunt and Pixton (1974). The review implies that, because no perfect method exists for measuring moisture, compromises must be made, depending on purpose of the test, conditions of the test (including speed and sample size), and type of information sought (precision and accuracy).

Current moisture measurements in the cereal grains trade are based on capacitance, which varies with the moisture contents, but the measurements are not accurate above approximately 30% moisture. The inaccuracy is partly due to the fact that the grain is often mechanically damaged. Grains (particularly corn) harvested with a moisture content greater than 28% are susceptible to mechanical damage, and such damage causes capacitance meters to give readings that are too high.<sup>4</sup>

A method to measure moisture (particularly in corn) must be unaffected by kernel damage and be fast and accurate on grain containing up to 40% moisture. Pulsed nuclear magnetic resonance (PNMR) spectroscopy has been applied successfully to the measurement of moisture in many food products (Brosio et al 1978; Hester and Quine 1976, 1977). Measurements are not affected by particle size, optical properties, or sample homogeneity, and they can be made rapidly. When PNMR spectroscopy is used, sample weighing is unnecessary if the sample packing does not vary significantly from that used in calibration. For each material to be tested, however, PNMR signals must be correlated with moisture values determined by a standard method, such as the air-oven method, and the calibration needs to be checked periodically.

We attempted to determine the relationship between PNMR values and percent moisture determined by an air-oven method for barley, corn, grain sorghum, and wheat.

## MATERIALS AND METHODS

### Natural High Moisture Grain

Corn and grain sorghum from the 1977 crop and barley, corn, grain sorghum, and wheat from the 1978 crop were hand-harvested at Manhattan, KS. Their moisture contents ranged from 14 to 40%.

### Rewetted Grain

The moisture content of mature grain from the 1976 crop was adjusted by two rewetting methods (soak and equilibration) to values up to 45%. By the soak method, samples of barley, corn,

grain sorghum, and wheat were soaked for 48 hr in distilled water. The excess water was drained, and water adhering to the grain was removed with paper toweling. Portions of the grain were spread on paper and air-dried with a fan to the desired moisture contents. By the equilibration method, 100-g samples of grain were placed in sealed glass jars with amounts of distilled water calculated to

TABLE I  
Heating Time and Temperature for Determining Moisture Contents of Whole Cereal Grains

Grain	Oven Temp. ( $\pm 1^\circ\text{C}$ )	Heating Time (hr)
Wheat <sup>a,b</sup>	130	19
Corn <sup>a</sup>	103	72
Grain sorghum <sup>a,b</sup>	130	18
Barley <sup>a,b</sup>	130	20

<sup>a</sup>From ASAE (1978).

<sup>b</sup>From Hart et al (1959).

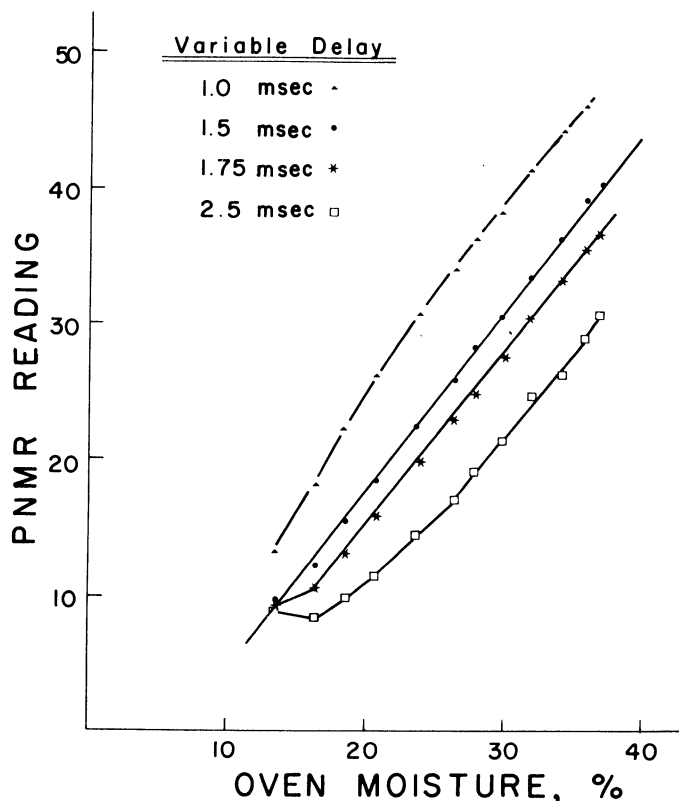


Fig. 1. Effect of variable delay time on pulsed nuclear magnetic resonance (PNMR) spectroscopy response to moisture in grain sorghum.

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provide the desired moisture contents. The samples were shaken periodically for 48 hr.

Moisture content of the whole grain was determined by a single-stage-heating, air-oven method (Hart et al 1959, ASAE 1978) as indicated in Table I.

The Praxis PR-103, PNMR spectrometer (Praxis Corp., San Antonio, TX 78251) was used with the following operating parameters: response, fast; variable delay (msec), determined for each type of grain; display, echo/ratio; program counter, 3; clock, 0.5 sec; function, 90–180°; control, automatic. Screw-cap (25-mm diameter × 95-mm high) glass tubes were filled with unweighed whole grain and placed on a Maxi Mix vibrator (Thermolyne Sybron Corp.) to pack the sample. Because the PNMR readout is the ratio of two intensities (echo and ratio), any minor packing effect is cancelled; each intensity would be affected in the same direction by a packing difference.

The PNMR reading was taken immediately after the sample was placed between the poles of the magnet. No temperature conditioning was used since Hester and Quine (1977) found that PNMR results were affected little by changes in temperature from 15 to 35°C. Our results also were not affected significantly by temperature, for the same reasons that minor differences in packing had no effect.

## RESULTS

### Tests with Rewetted Grain

For the initial PNMR measurements of moisture we used samples of grain that had been rewetted by the equilibration method. For each cereal, we had to determine the variable time delay that resulted in PNMR readings linearly related to oven moisture. Figure 1 shows that a variable delay setting of 1.5 msec was required for rewetted grain sorghum. Rewetted barley, corn, and wheat required variable delay settings of 1.25, 1.00, and 1.75 msec, respectively.

Figure 2 shows that the optimum calibration curve for the moisture content of wheat measured by PNMR depended on how the moisture content was attained. The calibration curves for the two wetting methods differed from each other and also from the

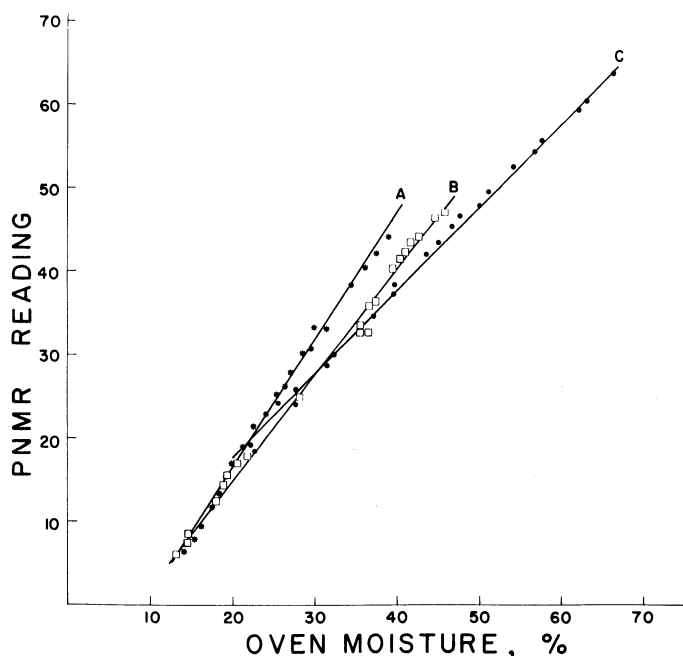


Fig. 2. Relation between pulsed nuclear magnetic resonance (PNMR) spectroscopy readings and moisture contents of wheat determined by an air-oven method. A, samples equilibrated (tempered) with different amounts of water, optimum variable delay time 1.5 msec; B, samples soaked in water and dried for various times, optimum variable delay time 1.5 msec; C, freshly harvested wheat, variable delay time 2.0 msec.

calibration curve for wheat of naturally high moisture. This is to be expected because the PNMR signals depend on the force with which water is bound to other components of the grain. Hence, PNMR readings depend on the grain and its history.

### Studies with Freshly Harvested Grain

Calibration curve C in Fig. 2 shows that PNMR readings were linearly related to moisture content between 21 and 66% moisture in freshly harvested wheat. If a linear relation for the range between 15 and 40% moisture is desired, an instrumental variable time delay setting of 2.5 msec is required (Fig. 3) instead of 2.0 msec for moisture contents of 21–66% (Fig. 2). Similar calibration curves were obtained for freshly harvested barley, corn, and grain sorghum (Fig. 3). The correlation coefficients for corn and grain sorghum of 0.992 and 0.993 (20 and 18 degrees of freedom, respectively) correspond with values of 0.966 and 0.819 (12 and 16 degrees of freedom, respectively) reported by Stermer et al (1977). They measured moisture contents as high as 47.4% in corn and 42.4% in grain sorghum, using a model 919 Motomco moisture meter.

The moisture contents of freshly harvested corn measured by the air-oven method and PNMR are recorded in Tables II and III. Calibration curve C (Fig. 2) was used for the moisture contents measured by PNMR. Correlation coefficients for the data were highly significant ( $P < 0.01$ ).

A *t*-test analysis of the moisture data (30–40%) for the corn varieties in Table II showed that the results from PNMR readings are higher than those from air-oven analysis ( $t = 3.46$  under the hypothesis that the difference = 0). This suggests that the

TABLE II  
Moisture Content<sup>a</sup> (%) of Several Varieties of Freshly Harvested High-Moisture Corn

Moisture Measuring Method	Samples						
	1	2	3	4	5	6	7
Air-oven <sup>b</sup>	38.5	34.4	38.4	36.7	31.3	36.7	33.8
PNMR <sup>c</sup>	38.4	35.4	40.0	38.5	32.3	37.0	34.2

<sup>a</sup>Standard deviation = 0.74,  $r = 0.97$  (5df),  $P < 0.01$ .

<sup>b</sup>From ASAE (1978).

<sup>c</sup>Pulsed nuclear magnetic resonance spectroscopy. From calibration curve C (Fig. 2).

TABLE III  
Moisture Content<sup>a</sup> (%) of a Single Variety of Corn Freshly Harvested Over a Period of Time

Moisture Measuring Method	Samples									
	1	2	3	4	5	6	7	8	9	10
Air-oven <sup>b</sup>	23.0	21.1	19.2	18.8	16.6	16.4	15.2	14.8	14.7	13.8
PNMR <sup>c</sup>	22.2	20.5	19.9	19.0	15.0	14.9	16.1	14.1	13.1	13.4

<sup>a</sup>Standard deviation = 0.91,  $r = 0.96$  (8df),  $P < 0.01$ .

<sup>b</sup>From ASAE (1978).

<sup>c</sup>Pulsed nuclear magnetic resonance spectroscopy. From calibration curve C (Fig. 2).

TABLE IV  
Lipid Content of Cereal Grains

Grain	Lipid Content Range (%)
Barley <sup>a</sup>	3.3–4.6
Corn <sup>b</sup>	2.2–7.0
Grain sorghum <sup>b</sup>	1.4–5.8
Wheat <sup>c</sup>	2.1–3.3

<sup>a</sup>From Morrison (1978).

<sup>b</sup>From Rooney (1978).

<sup>c</sup>From Pomeranz et al (1966).

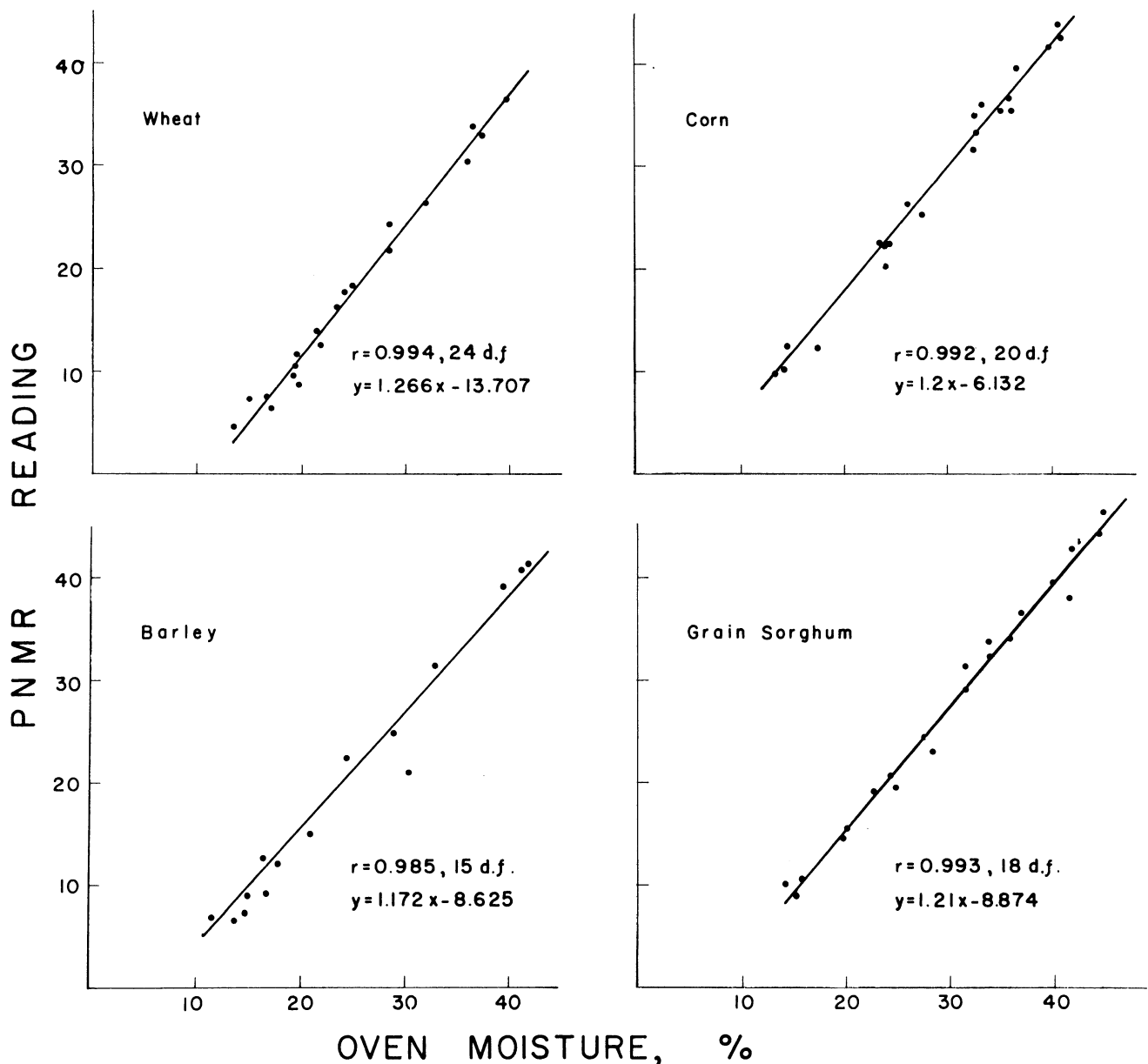


Fig. 3. Relation between pulsed nuclear magnetic resonance (PNMR) spectroscopy signal and moisture content of freshly harvested barley, corn, grain sorghum, and wheat determined by an air-oven method. A variable time delay of 2.5 msec was used for wheat and 2.0 msec for barley, corn, and grain sorghum.

calibration curve should perhaps be based on several samples, much like a calibration curve for analysis of protein by near infrared analysis (Norris 1978). For the moisture data (13–23% in Table III for a single variety of corn harvested over a period of time, the *t* test shows no difference in results from the two methods of analysis ( $t = 1.88$  under the hypothesis that the difference = 0).

#### DISCUSSION

The PNMR technique worked well for moisture levels exceeding 15%. Only a small (20-g) sample can be analyzed by this technique, however, and the instrument is relatively expensive. Also the free oil content of grain (Table IV) affects the free water determination. In practice, however, variations in oil content of commercial grain are much smaller than indicated in Table IV (Morrison 1978). In tests of new selections made by plant breeders or of cultivars that vary considerably in oil content, measurement of both oil and water may be necessary. This would unduly complicate the testing of grain in marketing channels. Such measurement is not likely to be necessary for commercial samples, however, because they are generally composites of several varieties from several locations.

The PNMR parameters used in this study can not be used when the moisture content is less than about 7.5% (data not shown in Fig. 2), presumably, because much of that moisture is bound (Miller and Kaslow 1963).

The usefulness of the PNMR method is in determining moisture accurately, rapidly, simply, routinely, and nondestructively over a wide range in small samples of barley, corn, grain sorghum, and wheat for use in marketing channels or plant breeding programs. Mechanical damage to grain does not affect the results.

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