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Simultaneous Determination of Moisture and Oil Content In Oilseeds by Pulsed Nuclear Magnetic Resonance

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ABSTRACT

Pulsed nuclear magnetic resonance technique using Carr-Purcell-Meiboom-Gill (CPMG) sequence has been used for simultaneous determination of moisture and oil content in rapeseed-mustard. This method involves sampling the free induction decay (FID) following 90° pulse in the CPMG sequence and resolving the trace of the amplitude of the CPMG echo signals into exponentially decaying liquid components of oilseeds. The data show that water in oilseeds generally exists in 2 phases and the relatively slow decaying component disappears around moisture content of 7% and below. The moisture and oil content have been determined by the method for 34 samples of 5 different varieties of seeds at varying moisture levels (~3% to 22%). The measured moisture and oil content have been compared with the values obtained by the oven drying method and earlier known FID method of pulsed nuclear magnetic resonance (NMR) respectively, and the agreement is fairly good for rapid estimation with standard deviation of 0.70% for oil content and 0.99% for moisture content. This is a rapid and nondestructive method for determination of both moisture and oil content without weighing and drying the seeds and also seems suitable for other matrix samples.

INTRODUCTION

Proton NMR techniques have been used extensively for determination and improvement of oil content in oilseeds (1-4). The continuous wave wide-line and pulse NMR techniques, being rapid and non-destructive methods for determination of oil content, have acquired great importance in plant breeding programs. The measurement of oil content by these methods in seeds containing moisture exceeding 5% requires drying of samples in order to eliminate interference by moisture signals. In addition, samples have to be weighed, which reduces the speed of the method (5). The free induction decay (FID) method of pulse techniques has been developed further to estimate oil content without weighing and drying the samples, but this method requires that moisture content should not change significantly from one sample to another (6). The different pulse techniques, namely Free Induction Decay (FID), spin echo (SE) and Carr-Purcell-Meiboom-Gill (CPMG) sequence (7,8), for estimation of oil content, have been compared. It is reported that relative oil content estimated by the Carr-Purcell-Meiboom-Gill (CPMG) sequence is not much influenced even at 20% moisture content (9). However, the CPMG method as performed previously required dry weight of the samples to estimate oil content. The pulsed NMR methods also have been used for moisture determination in barley, corn, wheat (10) and paddy (11) seeds.

Both oil and water content in olive husk have been determined simultaneously by solid to liquid ratio and SE decay curve of the sample (5). In the present paper, the feasibility of the CPMG method of the pulsed NMR technique for simultaneous determination of moisture and oil content in oilseeds is established. This rapid and nondestructive method does not require either weighing or drying of the sample and has significant applications in postharvest storage, germ-plasm evaluation, physiological studies, the food industry and marketing and plant-breeding programs.

EXPERIMENTAL METHODS

A pulsed NMR spectrometer operating at 20 MHz, Bruker Minispec pc 20 was used for all measurements. The 90° pulse width and the receiver dead time were about 4 μsec and 9.5 μsec (from middle of 90° pulse) respectively. The r.f. coil used provided a uniform field up to the sample height of 25 mm in a sample tube of 12 mm diameter. The sample height was restricted to 20 mm, and about 1.5 g of seeds were used for each analysis. The L/L + S EDM from Bruker was installed to operate the instrument, but the required pulse sequence was entered manually. The measured CPMG echo amplitudes were read out and recorded manually for analysis. The diode detection mode was used with 1 MHz band width and the probe temperature was around 40 C.

Seed samples of 5 rapeseed-mustard varieties—Pusa-Bold, Varuna, BS58, RLM198 and DYS-1—were hydrated by storing the seeds in a desiccator over water for about a week. The moisture content in hydrated seeds was decreased slowly by natural air drying and storing the seeds over calcium chloride in a desiccator at room temperature. By this process we were able to dry the seeds down to approximately 3% moisture content. Final drying was done in an oven at 105 C. Oil percentage in the seeds dried at 105 C was determined by the FID method of pulsed NMR technique (4).

For each sample, a CPMG pulse sequence with 180° pulse spacing of 0.1 msec was used. The signal was sampled at even echoes leading to measured data points every 0.2 msec. One hundred measurements were averaged for each sample to improve S/N ratio. The repetition time for individual pulse sequences was 2.0 sec, leading to a total measurement time of about 200 sec. In addition, the FID following the 90° pulse in CPMG sequence also was sampled just after the dead time of the receiver. The representative FID's were stored on a Nicolet Explorer III digital scope having a resolution of 12 bits at a sampling rate of 0.5 µsec/point and were plotted on an HP x-y recorder. The Bruker Minispec pc 20 spectrometer permits only 100 measured data points at a time which can be accessed manually via keyboard.

MEASUREMENT PROCEDURES

The free induction decay signal can be written as:

$$S_t = S_o^S \exp-(t/T_2^S)^2 + S_o^L \exp(-t/\overset{*}{T}{}_2^L) \qquad [1]$$

where subscripts 't' and 'o' stand for time and superscripts 'S' and 'L' indicate solid and liquid components respectively of the sample. $\overset{*}{T}{}_2^L$ is governed mainly by magnetic field inhomogeneity. The liquid signal components can be resolved by the exponential trace of echo signals using a CPMG sequence. The signal then can be described by the equation

$$S_t^L = S_t^{CPMG} = S_o^W \exp(-t/T_2^W) + S_o^O \exp(-t/T_2^O) \qquad [2]$$

where superscript 'W' stands for the water component and 'O' stands for the oil component of seeds.

The method used in the present study is based upon:

– Resolving the CPMG echoes signal into 3 exponentially decaying liquid components of oilseeds (2 due to moisture and 1 due to oil, as discussed in the following section).

– Determining the signal contributed by the solid matrix of seeds by sampling the FID following the 90° pulse in CPMG sequence at t = 10 µsec and subtracting the extrapolated liquid component signals.

– Computation of moisture % and oil % using a predetermined extrapolation coefficient (f_s) for the solid component; correction factors (α_1 and α_2) for water signals, and signal sensitivities (volt/gm) g_w for water, g_o for oil and g_s for solid components of rapeseed-mustard varieties.

RESULTS AND DISCUSSION

A recorder plot of the pulse sequence used for measurement of oil and moisture content and the signal decay is shown in Figure 1. This figure shows clearly the fast decaying signal from immobile solid constituents and slowly decaying signal from mobile liquid constituents of seeds.

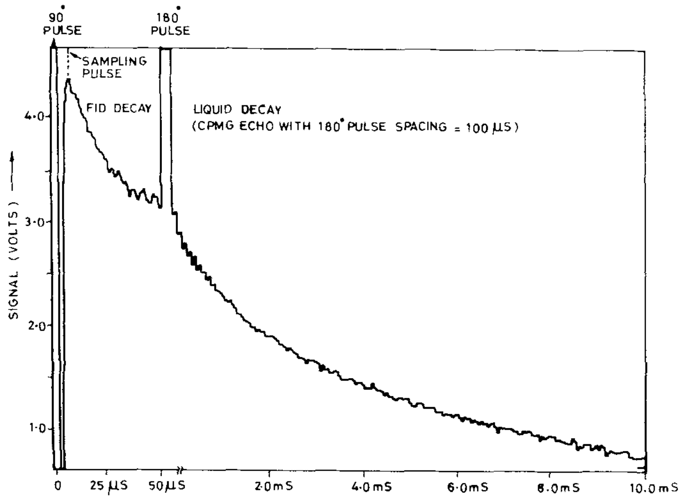

FIG. 1. Recorder plot of FID signal decay from a rapeseed-mustard sample. Only the first 180° CPMG pulse is shown, the rest being suppressed for clarity.

Determination of Liquid Signal Components

A plot of CPMG signal vs. time on a semilog scale for a variety of rapeseed-mustard at various moisture levels is shown in Figure 2. The plots for the other varieties studied were similar. From this figure, it is clear that the signal beyond t = 15 msec is almost the same (except for the first curve with 20% moisture). This is because the moisture signal decays to vanishingly small values and only the slow decaying oil signal persists beyond that time. The time decay of moisture signals obtained by subtracting out the extrapolated oil signal is given in Figure 3 on a semilog plot. It is evident from this figure that the moisture signal contains more than one exponentially decaying component, and as the moisture content decreases, a single rapidly decaying component becomes dominant. Numerical analysis reveals that these decay curves can be fitted adequately by a 2 component exponential decay. Equation 2 can then be written as

$$S_t^{CPMG} = S_o^{W_1} \exp(-t/T_{21}^W) + S_o^{W_2} \exp(-t/T_{22}^W) + S_o^O \exp(-t/T_2^O) \qquad [3]$$

where superscripts W_1 and W_2 stand for 2 water components and T_{21}^W and T_{22}^W are their respective spin-spin relaxation times. The extrapolated signal values and spin-spin relaxation times of 2 moisture components are given in Table I. The correlation coefficients (r^2) for exponential fits of the data varied from 0.991 to 0.999. The T_2 values for various components in the samples studied varied from ~0.2 to 0.7 msec for fast decaying moisture, ~1 to 6 msec for slow decaying moisture and ~90 to 105 msec for oil protons. To simplify computations to allow execution on a programmable pocket calculator, 2 points from each of the above 3 ranges were selected instead of using all the data points for evaluating the respective signals at t = 0. A comparison of the results from these 2 modes of calculation is given in Table I. It is evident that the results are comparable. Therefore, signal measurements at 6 selected points are adequate for quick determination. In the subsequent analysis, signal values at the following times were used for all computations: 0.2, 0.4, 2.4, 5.6, 15.2 and 19.2 msec.

The signal for the 20% moisture curve is higher than those due to other moisture levels even beyond t = 15 msec (Fig. 2). This may be attributed to the existence of a third moisture component which is very slowly decaying and appears at higher moisture levels. This requires that the

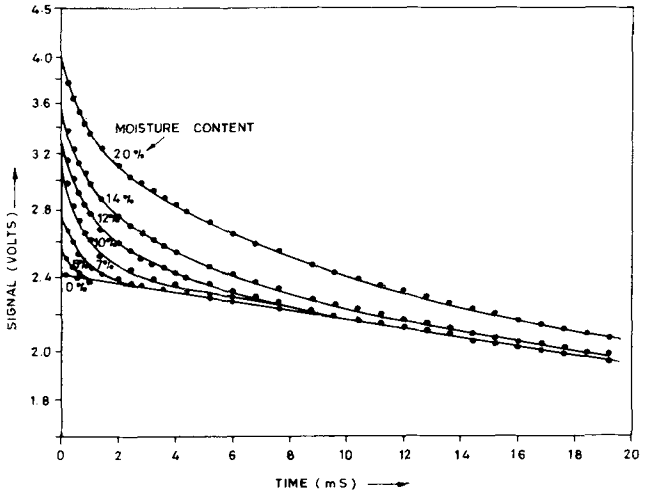

FIG. 2. Decay of CPMG echo amplitude versus time for a rapeseed-mustard sample at various moisture levels.

TABLE I

Extrapolated Signal Values and Spin-Spin Relaxation Time of Moisture Components of Rapeseed-Mustard at Various Moisture Levels

Moisture (% dry weight basis)	Fast decaying component			Slow decaying component		
	Extrapolated signal $S^{W_1}_{O}$ (volt)		T^{W}_{21} (msec)	Extrapolated signal $S^{W_2}_{O}$ (volt)		T^{W}_{22} (msec)
	Using all data points	Using 2 selected points		Using all data points	Using 2 selected points	
20.1	0.53	0.47	0.71	1.01	1.01	6.10
14.3	0.45	0.42	0.66	0.66	0.65	3.78
11.7	0.43	0.38	0.50	0.48	0.48	2.72
9.6	0.42	0.45	0.47	0.30	0.25	1.90
7.2	0.26	0.28	0.41	0.14	0.13	1.05
4.9	0.15	0.14	0.37	–	–	–
3.0	0.05	0.04	0.15	–	–	–

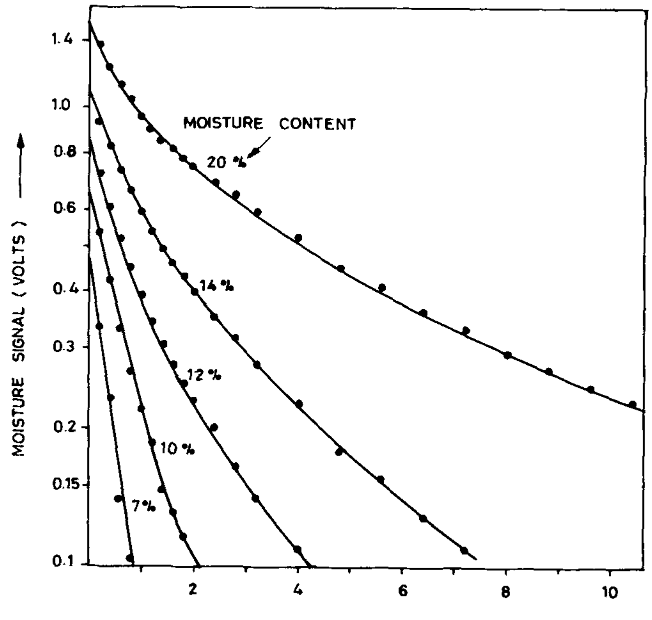

FIG. 3. Decay of moisture component signal with time for a rapeseed-mustard sample of various moisture levels.

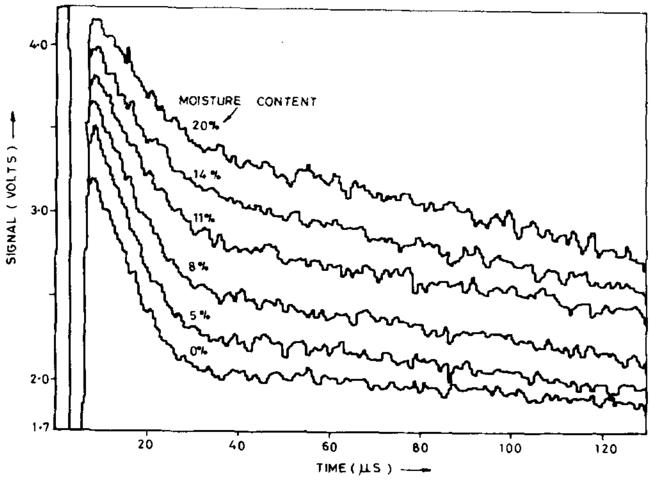

FIG. 4. Recorder plots of digitized FID signal of solid and liquid components of a rapeseed-mustard sample.

points selected for the oil component signal be moved to still higher time values so that the moisture signal decays completely. But this cannot be done because of the limitation of 100 accessible data points on our Bruker pc 20 spectrometer, and for maintaining a data point spacing of 0.2 msec to resolve the rapidly decaying water component. Because of this limitation, the oil component signals of seeds dried at 105 C were used for computing the water component signals for moisture levels around 15% and higher. (The extrapolated oil component signals at higher moisture levels were determined for a few samples by doubling the usual pulse spacing used in the present study. The values obtained were found to be coincident with those due to the dried seeds.) The data in Table I further show that the slowly decaying moisture component signal and its T_2 decrease rapidly with decreasing moisture content as compared to the fast decaying moisture component signal. The former component almost disappears at moisture levels around 7% and lower.

Computation of Solid Component Signal and Its Extrapolation Coefficient (f_S)

The recorder plots of the FID signals for a variety of rapeseed-mustard at varying moisture levels are shown in Figure 4. It is evident that the signal contributed by the solid portion of seeds decays fast to almost zero at 50 µsec. This figure further reveals that the signal due to solid matrix sampled at 10 µsec decreases with increasing moisture. Gaussian extrapolation of solid component signals at different moisture levels of the same seed sample results in varying zero time signals. The problem, however, is that the digitized FID cannot be accurately established from the available data. Since the hydrated seeds are partly in a semisolid phase, the FID cannot be expected to be a pure Gaussian. For this reason, a reliable estimate of total solid signal in the seed sample is not possible by extrapolation unless the receiver dead time is reduced substantially. An indirect method is, therefore, used to estimate the extrapolation coefficients at different moisture levels.

The digitized FID of the 105 C oven-dried seed samples was used to obtain a Gaussian fit for the solid matrix. Since our Nicolet digitizer does not have an averaging facility, a smooth curve was drawn manually through the plotted noisy FID. The signal contributed at zero time by the solid present in rapeseed-mustard is thus obtained. A knowledge of this and gravimetrically known moisture content in the hydrated seeds of the same sample permits the estimation of extrapolation coefficients (f_S) for solid component signals at various moisture levels. The signal sensitivities (volt/gm) for water (g_w) and pure mustard oil (g_o) were measured separately using the same CPMG sequence and were found to be 3.73 and 3.90 respectively. For a seed sample containing w(gm) moisture, we can write the following expression:

$$S_o^{S'} + S_o^{O} + g_w \cdot W = f_s\, S_t^S + S_o^L \qquad [4a]$$

where S' stands for the solid component of 105 C dried seeds and f_s is an extrapolation coefficient to account for the decay of the solid component at different moisture levels. S_o^L is the total extrapolated liquid signal determined by CPMG, and S_t^S is obtained by subtracting S_o^L from the total signal measured at t = 10 μsec. Equation 4a can be written as:

$$f_s = (S_o^{S'} + S_o^{O} + g_w \cdot W - S_o^L)/S_t^S \qquad [4b]$$

The computed values of f_s using Equation 4b for the same sample of a rapeseed mustard variety at various moisture levels are given in Table II and are found to be fairly constant, leading to a mean value of 1.30. The mean extrapolation coefficients (f_s) computed for different varieties of rapeseed-mustard studied are 1.22, 1.28, 1.34, 1.27 and 1.30, yielding an average value for f_s of 1.28 with a standard deviation of 0.04. The nearly constant values of f_s obtained for different varieties establish the validity of the indirect method used for its computation; the average f_s value has been used in the subsequent calculations.

Estimation of Moisture Content

The data in Table II clearly show that the solid component signal (S_t^S) measured at 10 μsec decreases with increasing moisture content. This indicates that a part of the solid component of seeds gets dissolved in the available moisture and is converted into a semisolid state. This is further confirmed by the fact that the total water component signal measured by CPMG pulse sequence is more than the expected signal calculated from known water content and signal sensitivity for water (g_w). The estimation of moisture content in the seeds requires a knowledge of the fraction of signals contributed by moisture in the observed total water signal ($S_o^{W_1} + S_o^{W_2}$). Further, the total water signal observed for low moisture levels (~5% and less) is lower than the expected water signal. This may be attributed to a small quantity of water remaining tightly bound to the solid matrix. Thus the expected water signal in the seeds may be written as:

$$S_o^W = \alpha_1 (S_o^{W_1} + S_o^{W_2}) + \alpha_2\, f_s\, S_t^S \qquad [5a]$$

where α_1 is the fraction of observed water signal contributed by moisture and α_2 is the fraction firmly held by the solid matrix which does not contribute to the water signal. Equation 5a can be rewritten as:

$$S_o^W/(f_s \cdot S_t^S) = \alpha_1 (S_o^{W_1} + S_o^{W_2})/(f_s \cdot S_t^S) + \alpha_2 \qquad [5b]$$

α_1 and α_2 then can be obtained by a linear fit of the data. Table III gives the expected water signals (signal sensitivity × water weight), total water signals observed, corrected solid component signals at different moisture levels and computed moisture % using α_1 and α_2 values from the linear fit. The correlation coefficient between the gravi-

TABLE II

Extrapolation Coefficients (f_s) for Solid Component Signals at Different Moisture Levels for a Variety of Rapeseed-Mustard (Using Equation 4b)

Moisture (% dry weight basis)	Water weight (g_m) (gravimetrically determined)	Total liquid signal (S_o^L)	FID signal at t = 10 μsec (S_t^T)	Solid component signal at t = 10 μsec (S_t^S)	Extrapolation coefficient (f_s)
20.1	0.271	3.91	4.71	0.80	1.33
14.3	0.193	3.50	4.42	0.92	1.28
11.7	0.158	3.27	4.29	1.02	1.25
9.6	0.130	3.10	4.14	1.04	1.29
7.2	0.097	2.82	3.98	1.16	1.29
4.9	0.066	2.56	3.81	1.25	1.32
3.0	0.041	2.45	3.70	1.25	1.33
MEAN	–	–	–	–	1.30

TABLE III

Expected Water Signal (signal sensitivity × water weight), Total Water Signal, Corrected Solid Component Signal at Different Moisture Levels and Computed Moisture % using α_1 and α_2 Values from Linear Fit[a]

Moisture (% dry weight basis)	Expected water signal ($S_o^W = g_w \times W$)	Total water signal observed ($S_o^{W_1} + S_o^{W_2}$)	Corrected solid component signal ($f_s \times S_t^S$)	Computed moisture % using α_1 and α_2 values from fit and using seed weight
20.1	1.01	1.48	1.04	19.5%
14.3	0.72	1.07	1.20	14.7%
11.7	0.59	0.86	1.33	12.3%
9.6	0.48	0.70	1.35	10.4%
7.2	0.36	0.41	1.51	7.1%
4.9	0.25	0.14	1.63	3.9%
3.0	0.15	0.04	1.63	2.7%

[a] A linear fit of data using equation 4b yielded α_1 = 0.613, α_2 = 0.0694 and correlation coefficient (r^2) was 0.993.

metrically determined moisture % and computed moisture % using fitted values of α_1 and α_2 is 0.988, which is fairly high. The values of α_1 and α_2 obtained from the fit of data for 5 varieties of rapeseed-mustard studied are given in Table IV. The average values of α_1 and α_2 thus obtained were used for subsequent computations.

Determination of Oil and Moisture Content Without Weighing Seeds

The discussion in the above section implies that only part of the observed water signal $(S_o^{W_1} + S_o^{W_2})$ is contributed by seed moisture. The remaining portion of the water signal then clearly corresponds to the solid component. The net observed extrapolated solid signal in the seeds may then be written as:

$$S_o^S = (1-\alpha_2)\ f_s \cdot S_t^S + (1-\alpha_1)\ (S_o^{W_1} + S_o^{W_2}) \qquad [6]$$

where S_t^S is the measured solid component signal at t = 10 μsec and other symbols already have been defined. Table V gives the extrapolated solid component signals of 105 C dried seeds of rapeseed-mustard varieties (using Gaussian extrapolation) and their corresponding T_2 values. The correlation coefficient of Gaussian fit in all samples was ~0.999, and the T_2 values also were almost the same for the varieties studied. Using the Gaussian extrapolated signal and computed weight of solid component of seed, the signal sensitivity for solid matrix (g_s) has been calculated and given in Table V along with the ratio of oil sensitivity to solid matrix sensitivity ($K_s = g_o/g_s$). The values of k_s and T_2^S measured directly for a dried defatted mustard seed sample yielded 1.702 and 18 μsec respectively, which are comparable with the computed values given in Table V. The measured ratio of signal sensitivity of oil to that of water K_W (= g_o/g_w) comes out to be 1.046, which is very close to the theoretically expected value from hydrogen percentage in the 2 components. The moisture % and oil % on initial weight basis then can be described by the following expression:

$$\text{Moisture \%} = \frac{K_W \quad S_o^W}{S_o^O + K_W \cdot S_o^W + K_s \cdot S_o^S} \times 100 \qquad [7]$$

$$\text{Oil \%} = \frac{S_o^O}{S_o^O + K_W \cdot S_o^W + K_s \cdot S_o^S} \times 100 \qquad [8]$$

The above equations can be rewritten for dry weight basis:

$$\text{Moisture \%} = \frac{K_W \cdot S_o^W}{S_o^O + K_s \cdot S_o^S} \times 100 \qquad [9]$$

$$\text{Oil \%} = \frac{S_o^O}{S_o^O + K_s \cdot S_o^S} \times 100 \qquad [10]$$

Using average values of α_1, α_2, f_s, K_s and measured value of K_w, the moisture % and oil % values have been computed on dry weight basis by using seed weight and also without using seed weight for 34 samples of 5 varieties of rapeseed-mustard and are given in Table VI. The agreement between the values is fairly good for reliable and fast simultaneous determination of oil and moisture content in seeds. The standard deviation (s.d.) between the moisture % values obtained by the oven drying method and by the simultaneous pulsed NMR method without weighing the seeds is 0.99%. The s.d. between the oil % determined by using seed weight and oil signal only and that obtained without weighing and using the simultaneous method was 0.70%. The s.d. within the method was estimated by repeatedly measuring a sealed sample of mustard seeds over the entire duration of the measurements; it came out to be 0.5%. The s.d. can be improved by using the computing parameters for the same variety instead of the average values used in the present study, but this is not practical because of the large number of seed varieties. The accuracy can be slightly improved by using more complex curve fitting to evaluate the oil and water signal in place of the rapid 6-point procedure used here. The method can be applied to other oil seeds and also to the samples of different matrices after evaluating the various parameters discussed. It is a rapid and non-destructive method for simultaneous determination of moisture and oil content without weighing and drying the seeds and has several important applications.

TABLE IV

Linear Fit Parameters (α_1 and α_2) for 5 Varieties of Rapeseed-Mustard for Estimation of Moisture Content from Observed Water Signal

Sample no.	Value of α_1	Value of α_2	Correlation coefficient (r^2)
1	0.660	0.089	0.993
2	0.575	0.082	0.995
3	0.625	0.049	0.995
4	0.602	0.090	0.997
5	0.613	0.069	0.993
Average	0.615	0.076	

TABLE V

Signal Sensitivity (g_s) for Solid Component of 105 C Dried Rapeseed-Mustard of 5 Varieties

Sample no.	Gaussian fit of solid decay[a] Extrapolated signal	Gaussian fit of solid decay[a] T_2^S	Extrapolated oil signal	Dry seed weight (gm)	Weight of solid component of seeds (gm)	Signal sensitivity for solid matrix (volt/gm)	Ratio of oil sensitivity to solid matrix sensitivity $K_s = g_o/g_s$
1	1.44	15.9	2.00	1.246	0.733	1.96	1.99
2	1.67	14.8	1.91	1.294	0.804	2.08	1.88
3	1.82	15.0	1.90	1.323	0.836	2.18	1.79
4	1.68	15.0	2.00	1.313	0.800	2.10	1.86
5	1.55	15.8	2.41	1.348	0.730	2.12	1.84
							Average = 1.87

[a]The middle of 90° pulse was taken as 0 time for the Gaussian fit; the correlation coefficient of this fit for all samples was ~0.999.

TABLE VI

Comparison of Moisture and Oil Percentage Determined by the Simultaneous Method with Moisture Determined Gravimetrically and Oil Determined using Seed Weight and Oil Signal Only

Variety name	Moisture % (oven method)	Using seed weight		Without using seed weight	
		Moisture %	Oil %	Moisture %	Oil %
Pusa Bold	25.9	24.4	41.2	23.9	40.3
	19.9	18.6	41.2	19.1	42.2
	14.4	15.5	41.2	15.9	42.4
	11.1	11.9	41.2	12.0	41.7
	7.9	7.6	41.9	7.6	41.7
	5.2	5.0	40.9	5.1	41.5
	3.1	3.4	40.9	3.5	41.7
Varuna	21.8	22.6	37.6	22.7	37.8
	16.1	17.5	37.6	17.8	38.3
	13.5	15.2	37.6	15.4	37.9
	11.4	12.0	38.8	12.1	39.0
	8.4	8.8	37.6	8.8	37.2
	5.9	6.0	38.4	6.1	39.1
	3.6	2.9	37.9	3.1	40.0
BS58	22.1	22.3	36.2	22.3	36.3
	16.2	16.9	36.2	17.1	36.8
	13.5	14.5	36.7	14.6	36.8
	11.2	12.5	36.3	12.8	37.0
	8.4	8.2	37.1	8.1	36.7
	7.5	6.1	36.3	6.2	37.0
	3.5	3.9	35.6	4.0	36.5
RLM-198	23.3	23.9	38.7	23.9	38.7
	17.4	18.6	38.7	18.8	39.1
	14.2	15.8	38.7	16.0	39.1
	11.3	12.5	38.6	12.7	39.0
	8.1	8.8	38.1	8.7	38.0
	3.3	3.2	38.6	3.2	38.7
DYS-1	20.1	19.8	45.2	20.0	45.6
	14.3	15.0	45.2	15.2	45.9
	11.7	12.6	45.9	12.7	46.2
	9.6	10.6	45.7	10.9	46.9
	7.2	7.3	45.9	7.3	45.8
	4.9	4.3	46.0	4.3	46.0
	3.0	3.1	45.8	3.1	46.3

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