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MOISTURE DETERMINATION

Determination of Water by Nuclear Magnetic Absorption in Potato and Apple Tissue

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The measurement of nuclear magnetic resonance absorption of hydrogen nuclei in water provides a rapid method for the determination of the water content of potato tissue without reference to other standard moisture procedures. In the case of apple tissue, the same procedure can be used, provided correction is made for soluble solids. Using the method previously applied to starch suspensions, the results obtained on water content of potatoes by the nuclear magnetic resonance absorption method agree with results obtained by the vacuum oven method; for apples, the results are too high because of the contribution to the nuclear magnetic absorption of hydrogen nuclei in the soluble solids. In these cases moisture can be determined if the contributing soluble constituents are known, or by means of a calibration curve.

A METHOD for the quantitative measurement of hydrogen in liquids and suspensions by nuclear magnetic resonance absorption was presented in a previous paper (*3*). When applied to aqueous suspensions of starch, it was shown that the method measured only the hydrogen in the aqueous phase and could be used to determine the amount of water in starch suspensions to within about 2%. The results obtained for starch suspensions indicated that the same method might be useful for the measurement of the water content of fruit and vegetable tissues or similar biological materials on an absolute basis. In the present paper these possibilities are investigated for two types of tissue, potato and apple. The essential features of the method are given in the previous paper (*3*).

Experimental

The methods of measurement and apparatus are the same as those used in the previous study (*3*). Potato and apple were chosen because they are typical of many others for which a rapid method of moisture measurement would be of value; moreover, they are sufficiently large to provide test specimens in the form of single cylindrical pieces about 2 cm. in diameter, 3.24 cm. in length for potato, and 1.93 cm. in length for apple.

The cylindrical specimens of tissue were obtained by means of a cork-boring machine. The cylinders of tissue were inserted into a jig consisting of a close-fitting glass cylinder. The ends of the tissue were cut off flush with the ends of the glass cylinder.

The maximum variation in the volume of the specimens was estimated to be about 2%. This variation was due primarily to variations in diameter. The volume of the cylindrical glass jig is assumed to be the maximum volume of the test specimen.

If the magnetic fields employed in the magnetic absorption measurement were uniform, no error would result from the fact that the actual volume of the test specimen is smaller than the assumed volume—i.e., the volume of the glass jig. For example, if the volume of the jig is assumed to be V_a, and the specimen volume is $V_0 < V_a$, then the observed signal is

$$(D_{\max_0})_0 = (D_{\max_0})_a \times (V_0/V_a) \quad (1)$$

and the apparent bulk density is

$$\rho_0 = \rho_a(V_0/V_a) \quad (2)$$

Now the water content (w.c.) (per cent by weight), is expressed as

$$\text{water content} = [(D_{\max_0})_0/(D_{\max_0})_{\text{water}}] \times (\rho_{\text{water}}/\rho_0) \quad (3)$$

From Equations 1, 2, and 3

$$\text{water content} = [(D_{\max_0})_a/(D_{\max_0})_{\text{water}}] \times (\rho_{\text{water}}/\rho_a) \quad (4)$$

Thus if Equation 1 is true—i.e., signal strictly proportional to volume—the error in water content due to an error in the specimen volume would be zero. However, as the magnetic field employed in the nuclear magnetic resonance measurements was not uniform, Equation 1 is only approximately true. A reasonable estimate is that the maximum error in water content is probably not greater than 1% for a 2% volume error.

The nuclear magnetic resonance absorption experiments were made at room temperature (26° ± 2° C.). The water content of the test specimens was also determined by standard vacuum oven procedures.

As in the previous paper, the peak absorption signal, $D_{\max}$, was determined for several values of the radio-frequency magnetic field intensity, H_1, in the range 2 to 6 × 10^{-4} gauss. Graphs of $D_{\max}$ *vs.* H^2 were extrapolated linearly to obtain $(D_{\max})_0$. In order to standardize the apparatus and check over-all performance, a similar determination was made for distilled water at various times

Table I. Determination of Moisture in Potatoes

Sample No.	Density, ρ, G./Cc.	$(D_{max})_0$, Arbitrary Units	% Water	
			NMR method	Vacuum oven method
1	1.033	172.0	87.2	84.4
2	1.038	162.7	82.0	84.7
3	1.048	162.5	81.2	84.5
4	1.043	169.5	85.2	84.4
5	1.058	166.0	82.2	80.7
6	1.048	157.5	78.6	79.8
7	1.050	160.5	80.0	79.9
8	1.045	159.5	79.8	85.6
9	1.053	161.7	80.3	82.6
10	1.053	153.5	76.4	79.2
11	1.057	161.6	79.8	80.5
12	1.077	147.2	71.7	76.2

Volume of specimen (jig), V_a = 9.577 cc.

during the course of measurements. The use of water provided a primary standard for the absorption measurements in addition to the checks provided by the electronic calibrator incorporated in the spectrometer (*1*).

Results and Discussion

Potato The results obtained for 12 samples of potato are shown in Table I. The table gives the density of the potato specimen calculated from the weight of the specimen and the volume of the glass jig, the quantity D_{max_0}, the water content of the potato determined by vacuum oven and calculated from D_{max_0}, and the density according to Equation 3. As was discussed previously for starch suspensions, the use of Equation 3 assumes that the nonaqueous components of the potato act essentially as inert (non-hydrogen-containing) diluents. This assumption is verified by the experimental observations on starch suspensions (*3*).

As Table I shows, the general agreement between the water content of the potato determined by the nuclear magnetic resonance absorption method and by the vacuum oven method is good. The calculated standard deviation for the group of 12 samples is 3.0%. In a search for factors which might limit the precision of the measurements it was found that significant errors in D_{max} could arise from the fact that the distribution of water in potato tissue is nonuniform and the magnetic fields involved in the nuclear magnetic resonance absorption measurement are not uniform (*4*). The combined effect of these factors was found to give a value of D_{max} which varied significantly, depending upon the orientation of the specimen in the radio-frequency coil of the nuclear magnetic resonance apparatus.

Table II shows a typical set of readings obtained for two specimens of potato. The large variation between readings for the different positions makes it clear that this is a likely source for the errors found in Table I. In an effort to reduce this error, subsequent measurements of D_{max} were performed for four different orientations of the sample in the radio-frequency coil and an average value was calculated. As a test of the procedure two groups of potatoes were investigated, one containing 11 samples, the other 17 samples. The same conditions were employed as for the samples discussed in Table I, except that D_{max_0} was obtained as the mean of measurements for four different orientations in the radio-frequency coil. The standard deviations calculated for the two groups of samples were 1.8 and 1.4%, respectively. The results for the combined groups are summarized in Figure 1. This figure shows that over 90% of the measurements differ from the vacuum oven result by 2% or less. This error is consistent with that found for starch suspensions (*3*).

Apple Table III shows representative results for a group of 12 samples of apples. The values of D_{max_0} shown are average values of measurements obtained for four different orientations of the specimen in the radio-frequency coil. The standard deviation is 7.7%, in marked contrast to the standard deviation of 1.2% obtained for a group of five samples of potato run concurrently with the apples. The standard deviation of the results for a second set of samples is 5.7%. For all samples investigated, the water content calculated from the nuclear magnetic resonance absorption is larger than the vacuum oven result.

An important factor to be evaluated in the nuclear magnetic resonance absorption method of moisture measurement is the contribution of hydrogen-containing components such as fats, lipides, water-soluble solids (sugars, etc.), or other constituents which give rise to narrow proton resonance lines and thus cannot be distinguished by the techniques used here from hydrogen nuclei in water. Apple tissue contains substantial quantities of water-soluble solids and the study of the effects of these materials (*2*) suggests that the soluble solids in apple are responsible for the results observed.

Table II. Variation in D_{max_0} with Position of Sample in Radio-frequency Coil

Position	D_{max_0}, Arbitrary Units	
	Potato 3	Potato 9
1[a]	161.5[b]	159.0[b]
2	173.0	162.0
3	173.5	162.5
4	174.5	163.5
	Av. 170.6	161.8

[a] 1. Initial position. 2. Rotated 90° clockwise from 1. 3. Inverted; aligned as in 1. 4. Rotated 90° clockwise from 3.
[b] Read only to nearest half unit.

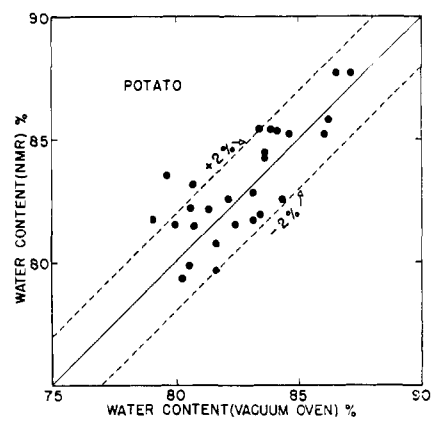

Figure 1. Determination of moisture in potatoes by nuclear magnetic absorption method and vacuum oven methods

In brief, from information in the literature on the proximate composition of apples (*5*) it was concluded that out of a total of about 16% solids in the edible portion of fresh apple approximately 12 to 14% is water-soluble, 11 to 12% being sugars. Vacuum oven measurements made in this laboratory of the total solids in the apples of Table III verify the total solids content fairly closely, the average value found

Table III. Determination of Moisture in Apples

Sample	Density, G./Cc.	D_{max_0} (Av.), Arbitrary Units	% Water	
			NMR method	Vacuum oven method
1	0.819	151.1	92.2	84.0
2	0.851	153.4	91.3	82.9
3	0.827	145.8	89.6	84.4
4	0.829	148.4	91.3	83.8
5	0.826	150.9	93.5	83.6
6	0.809	144.9	91.6	84.0
7	0.859	154.0	91.0	84.1
8	0.832	150.4	92.3	83.7
9	0.831	151.1	89.8	83.6
10	0.830	147.1	90.2	84.1
11	0.827	146.4	90.1	84.5
12	0.827	150.1	92.1	84.9

Volume of specimen (jig), V_a = 5.690 cc.

being approximately 16%. In lieu of other information, 12% was assumed for the amount of soluble sugars in fresh apple of 16% total solids and a correction was made on this basis. For the purpose of the correction it was assumed that soluble materials had the same composition as glucose (*2*).

Table IV shows the corrected results for the apple data of Table III. For the set of 12 samples the standard deviation of the corrected result is 1.3% (compared to a previous value of 7.7), and for the second group, not tabulated, the standard deviation is 2.1 for the corrected results (compared to 5.7). No information was available concerning the past history of the second set of apples, but it is known (*5*) that storage time, temperature, etc., may cause variations in water and sugar content and these factors probably account for the slightly larger standard deviation found for the second set of apples.

The over-all agreement of the corrected nuclear magnetic resonance absorption results and the vacuum oven results for apples is considered a sufficient basis for the conclusion that the difference in behavior of apples and potatoes, in so far as nuclear magnetic resonance absorption is concerned, is due to the appreciable soluble solids content of apples as compared to the negligible amount of soluble solids in potatoes.

The results obtained for potato and apple tissues show that the method of determining the water content of vegetable tissues from D_{max_0}, the maximum value of the derivative of the proton magnetic absorption curve, is satisfactory without correction only if the tissue does not contain appreciable quantities of solutes which can contribute to the proton absorption.

The correction for soluble solids applied to data in Table IV is based on the amount of solids in the individual samples of apple as determined by the vacuum oven method. It will thus be impractical to make routine determinations of water content by the nuclear magnetic absorption method unless it can be established that the soluble-solids content of each specimen of the substance under investigation is essentially the same. In this case the most direct solution would be to employ a calibration curve prepared by determining the water content of a few samples of the particular type of tissue under consideration. This procedure will be practical only if a sufficiently large number of samples is to be analyzed by the nuclear magnetic absorption method.

Other correction procedures which do not require calibration data may be developed by investigating in detail the magnitude of the correction required for a large number of different types of apple tissue, the influence of storage conditions, and other factors. The limited data taken on apple tissue in the present study are not suitable for this purpose.

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Table IV. Application of Correction for Soluble Solids to Nuclear Magnetic Absorption Data for Apples

Sample	D_{max_0}, Arbitrary Units		$\frac{(D_{max_0})_s \times \rho_w}{(D_{max_0})_w \times \rho_s}$	$\frac{12}{16} \times V_a \left(\frac{m_i}{m_0}\right)$,	$9\sum \frac{n_i}{w_i}\left(\frac{m_i}{m_0}\right)$	% Water	
	Apple	Water				NMR corrected[a]	Vacuum oven
1	151.1	200	0.922	0.120	0.072	85.0	84.0
2	153.4	197.5	0.913	0.128	0.077	83.6	82.9
3	145.8	196.8	0.896	0.117	0.070	82.6	84.4
4	148.4	196.0	0.913	0.122	0.073	84.0	83.8
5	150.9	195.5	0.935	0.123	0.074	86.1	83.6
6	144.9	195.5	0.916	0.120	0.072	84.4	84.0
7	154.0	197.0	0.910	0.119	0.072	83.8	84.1
8	150.4	195.8	0.923	0.122	0.073	85.0	83.7
9	151.1	202.5	0.898	0.123	0.074	82.4	83.6
10	147.1	196.5	0.902	0.119	0.072	83.0	84.1
11	146.4	196.5	0.901	0.116	0.070	83.1	84.5
12	150.1	197.0	0.921	0.113	0.068	85.3	84.9

[a] % water = $100 \times m_{w_s}/m_0$, where $\frac{m_{w_s}}{m_0} = \left[\frac{(D_{max_0}) \text{ sample}}{(D_{max_0}) \text{ water}} \times \frac{\rho_{water}}{\rho_{sample}}\right] - \left[9\sum_i \frac{n_i}{w_i}\left(\frac{m_i}{m_0}\right)\right]$

For notation and details of correction see (*2*).