

# Variability in determination of the single kernel moisture content of grain by means of TD-NMR spectroscopy

T.M. Schinabeck<sup>1,2\*</sup>, F. Weigler<sup>1</sup>, E. Flöter<sup>2</sup> and J. Mellmann<sup>1</sup>

<sup>1</sup>Department of Post Harvest Technology, Leibniz Institute for Agricultural Engineering and Bioeconomy, Max-Eyth-Allee 100, 14469 Potsdam, Germany; <sup>2</sup>Department of Food Process Engineering, Technische Universität Berlin, Seestraße 13, 13353 Berlin, Germany; [tschinabeck@atb-potsdam.de](mailto:tschinabeck@atb-potsdam.de)

Received: 19 May 2017 / Accepted: 24 October 2017

© 2017 Wageningen Academic Publishers

## RESEARCH ARTICLE

### Abstract

Detailed information on the moisture distribution of grains is important to predict its storability, because there can be a significant variance in the moisture content among individual grain kernels. Hence, it is important to determine the single kernel moisture content. For it, the time domain nuclear magnetic resonance (TD-NMR) spectroscopy was applied and adopted for the non-destructive measurement at different types of grain with the focus on wheat. The TD-NMR is widely used in food industry to determine the moisture content precisely. The influence of the single kernel position in the measuring cell was examined more closely. As could be demonstrated, the particle moisture varied significantly depending on the angular position in the TD-NMR spectroscope. When the kernel was turned by 90°, the moisture deviated by up to 0.9% wet basis, and this deviation increased as the grain water content increased. In addition, the experiments showed that the dry mass of the kernel has an impact on the received moisture content. Hence, the kernel should always have the same position in the NMR-spectroscope. Also the calibration should be carried out in this position and a wide range of kernel weight should be selected.

**Keywords:** wheat, moisture-determination, angular position, data variability, calibration

### 1. Introduction

In the grain markets as well as in grain preservation and storage, the term 'grain moisture content' refers to the average moisture content of a sample of kernels. Studies have found that a significant variance in the moisture content exists among individual grain kernels (Liu *et al.*, 1997). The moisture content of grains is one of the most important parameters in measuring quality and predicting storability. However, common meters are unable to detect any differences in moisture content between kernels, because they only measure the mass average moisture content. The detailed moisture distribution is, however, important for better prediction of grain storability. The lack of uniformity of the moisture distribution within a grain mass is known to affect its storability (Brusewitz, 1987).

Many methods have been developed to measure the moisture content of grains and their products. The best known methods are: oven drying; vacuum oven drying, distillation, chemical reaction with the Karl Fischer reagent, electrons devices/ moisture meters, nuclear-magnetic-resonance (NMR), near-infrared spectrophotometry (NIR). These methods are classified as: fundamental, routine and practical references. The fundamental reference methods are considered to measure 'true' moisture and are used to verify the measurements obtained with the routine reference methods (Christensen *et al.*, 1992). Among these various methods, only a few can be used to measure the moisture content of individual grains. If the individual grain is needed for further analysis, the method may have to be non-destructive (Chambers *et al.*, 1989). For example, the methods of hygrometry, NIR, and time domain nuclear magnetic resonance (TD-NMR) fulfil these requirements.

However, the hygrometry method requires an apparatus which is not widely available. The disadvantage of NIR is the potential error due to the limited measuring depth into the kernel centre. Hence, the TD-NMR method was selected for single-kernel moisture determination.

The use of NMR for moisture measurement of cereal samples weighing several grams is well established (Miller and Kaslow, 1963; Miller *et al.*, 1980; Morley *et al.*, 1984; Pohle and Gregory, 1968). At the individual grain level, pulse NMR methods have been used to ascribe the signals of resonating protons to oil, water, or solids (Persyn and Rollwitz, 1971) and to study the binding of water (Askochenskaya and Golovina, 1981). Peglow *et al.* (2011) used the method of NMR spectroscopy to determine the mass of water in a single solid particle to investigate the distributed properties of a population of granular materials after drying in fluidised bed dryers. In recent decades, NMR has become a widely appreciated measurement tool within food science and technology (Van Duynhoven *et al.*, 2010). In the food industry, TD-NMR is widely used for quality control/quality assurance (QC/QA) applications where precise determination of moisture and water is of fundamental interest (Todt *et al.*, 2006). Many of the pulse sequences in benchtop NMR relaxometry found their first application already decades ago. The free induction decay (FID) is the most basic transversal relaxometric experiment, but at current low-field benchtop NMR instruments its applicability is compromised by the strong inhomogeneity of commercial permanent magnets now in use. For phases of a higher molecular mobility such as the most semi-solid and liquid phases, the Hahn spin-echo (SE) of Carr-Purcell-Meiboom-Gill sequences is typically used (Van Duynhoven *et al.*, 2010).

Chambers *et al.* (1989) stated that it was not necessary to adjust the horizontal orientation of the grain in the NMR device while measuring the moisture content of individual wheat grains. However, the vertical position of the grain within the spectrometer was found to be critical. But Tiwari *et al.* (1974) as well as Srinivasan (1979) reported a strong influence of the shape of the particle and its position on the NMR moisture signal while measuring the oil content of single seeds of, among others, peanuts and sunflower seeds. They showed that the angular dependence is pronounced at larger delay time for FID and also at larger  $\tau$  (time between pulses) for SE.

Tiwari *et al.* (1974) tried to eliminate the influence of seed orientation by adjusting the delay time while measuring the oil content of peanuts by FID at four sample positions (0°, 90°, 180°, 270°). They found, that at a delay time of 200  $\mu$ sec, the same signal intensity was attained at all angular positions. Srinivasan (1979) did not observe this latter behaviour. But both authors found that the influence of the seed position can be expected to decrease in a sample

where the oil is distributed more uniformly. They showed that in the FID method the angular dependency at higher delay times of 200-1,200  $\mu$ sec. is more pronounced than at high  $\tau$  values (2-12 msec.) for the SE method. In our application, a proven  $\tau$  for measuring moisture in seeds is given (3.5 msec.). Chambers *et al.* (1989) adjusted each grain for maximum signal to eliminate the influence of the position, but this is a very time-consuming way. Hence, it is necessary to know the coherency to avoid measuring errors due to different kernel positions in the TD-NMR. Also Kraszewski *et al.* (1991) have observed that a single corn kernel of arbitrary shape which is rotated about its x axis in a moisture measurement device based on Microwave Resonator Technique, the transmission factor and the frequency shift followed a  $\cos^2\phi$  function, where  $\phi$  is the angle of rotation. The authors also noted that when the measured objects are of nearly uniform shape, the determination of moisture with Microwave Resonator Technique, independent of volume is possible by adapting a shape factor (Kraszewski *et al.*, 1989). For highly nonuniform objects like corn kernels this is not possible (Kraszewski *et al.*, 1991). To this end, the influence of the angular position of the single kernel is examined more closely for moisture determination in this study. Also the influence of the particle weight is examined.

## 2. Materials and methods

### Materials and sample preparation

To investigate the influence of the grain kernel position within the TD-NMR spectroscope, wheat (18, 15 and 12% wet basis (wb), shelled), barley (13% wb, unshelled), oats (14% wb, hand peeled), and rye (11.5% wb, shelled) were used. The peeling of the oats kernels was necessary to allow a horizontal position of the kernels in the test tubes. Except for the wheat, all grains had shelf-life moisture content and were examined by TD-NMR spectroscopy after a few months of storage. All samples of one type of grain were from the same batch. In the case of wheat, one batch with approx. 18% wb was used. Parts of it were dried to 15 and 12% wb, respectively, and used for subsequent measurements. The drying was performed in a cabinet dryer at 90 °C. Further sample preparation was not necessary for the TD-NMR spectroscopy. To study the influence of the dry kernel mass, a more recent wheat batch is used (14% wb). All moistures in this study refer to wb.

### NMR-spectroscopy

The single grain moisture content was determined by a Time-Domain NMR Benchtop System (minispec mq40; Bruker, Billerica, MA, USA). This is a 40 MHz system with a possible sample volume of 0.75 ml and a test tube volume of 10 ml (inner diameter: 8.5 mm). The system is recommended for single kernel measurements. A

normalised application was used based on spin-echo pulse sequences (duration of 3.5 msec between 90° and 180° pulses) to determine the moisture content in products such as seeds. The function of the TD-NMR and the Hahn-echo pulse sequence used have been explained elsewhere (see for example Cunäus, 2010; Van Duynhoven *et al.*, 2010; Hahn, 1950; Peglow *et al.*, 2009; Rutledge, 2001; Todt *et al.*, 2006).

### Oven method

With the NMR spectroscopy, the water content can only be determined indirectly. Therefore, calibration with a direct technique is necessary (Cunäus, 2010). Since the NMR spectroscopy is a non-destructive measuring method that does not change the properties of the sample, it is possible to determine the moisture content of a kernel after the NMR measurement, e.g. by means of the oven method and to calibrate the NMR with these results. According to the American test method (ASAE, 2006), a sample size of 10 g unground grain is required for wheat, barley, oats, and rye, which is dried in the oven and weighed again after cooling down in a desiccator. The percentage of the particle moisture  $u_p$  (wb) can then be calculated as:

$$u_p = \frac{m_t - m_d}{m_d} \times 100 \quad (1)$$

where  $m_t$  is the grain mass before drying and  $m_d$  the mass after drying. Replicate determinations should check within 0.2% wb of moisture. This method was slightly modified for the single particle moisture determination. Instead of the prescribed 10 g of unground grains, only single kernels were weighed out. Consequently, double determination was not possible in that case.

Nevertheless, the oven method is suitable for the calibration due to its low susceptibility to errors and the high number of kernels that can be used. Every batch of grain needs its own calibration.

### Influence of the grain kernel position in the TD-NMR measuring cell

Each grain was weighted and placed evenly in a test tube with the crease downwards (Figure 1). The hairs of brush were defined as 'front' and the germ as 'back' (Figure 1A). The test tubes were sealed with caps. The single grain was oriented at eight angular positions with a distance of 45° using a template, and the intensity was measured. The measurement of one single kernel in the eight positions takes a few minutes while the grain is in a 40 °C warm unit. To minimise the statistical error due to dehydration of the grains over time, therefore, the sequence of the measurements was reversed after half of the grain positions, beginning from the last position at 315° backwards. The wheat with 12% wb was analysed at 16 positions with an angular distance of 22.5° in a similar manner.

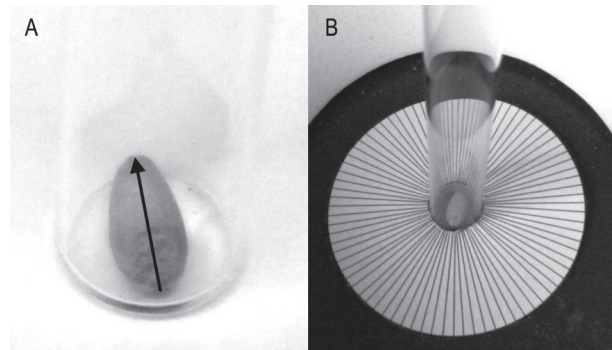


Figure 1. (A) Grain kernel in the test tube; (B) angle template to adjust the grain.

## 3. Results and discussion

The measurements of the NMR intensity of the single grain kernels were calibrated against the particle moisture content which was determined by the oven method. At the example of wheat, the results are illustrated in Figure 2 where the values of the NMR intensity of 239 kernels with different moisture contents are depicted over the particle moisture content. The calibration curve obey a strong linearity.

As can be seen from the graphs in Figure 3A, the NMR intensity also depends on the dried kernel mass ( $m_d$ ) although all kernels were taken from the same batch and were in equilibrium with the same relative atmospheric humidity. For all kernels, the oven-method measurements revealed the same scattering of around 12%. In contrast, the NMR intensity was slightly higher for the heavier kernels. In Figure 3B, the NMR intensity is depicted over the mass of water ( $m_w$ ) of the single kernels. The experimental results for 283 kernels at different moisture contents fit well a linear equation with the parameters  $m_w$  and  $m_d$  as:

$$\text{NMR intensity} = 180.1 + 37,515.8 m_w - 4,354.9 m_d \quad (2)$$

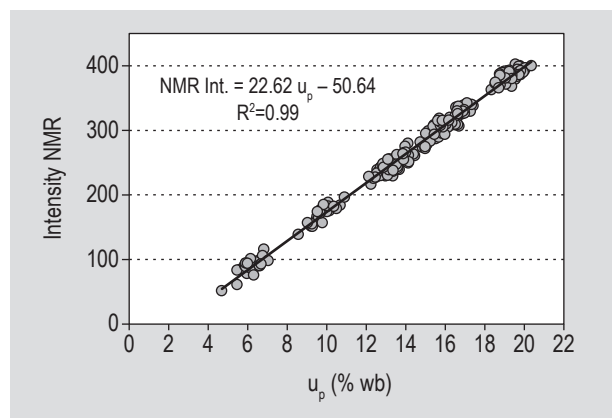


Figure 2. Calibration curve for wheat: nuclear magnetic resonance (NMR) intensity as a function of the particle moisture content measured by the oven-method (n=239).

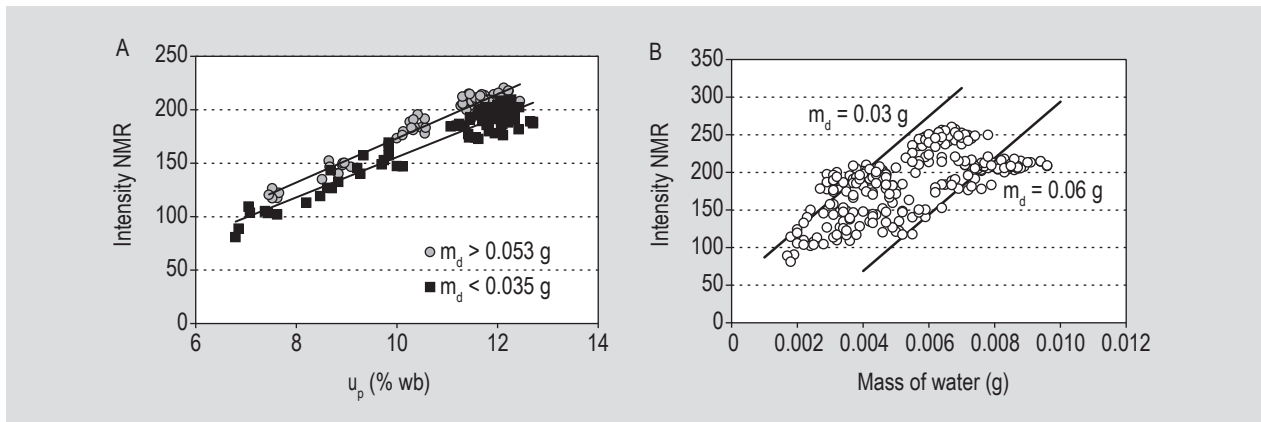


Figure 3. (A) Nuclear magnetic resonance (NMR) intensity as a function of the particle moisture content ( $u_p$ ) measured by the oven-method displaying a dependence on the dry grain mass ( $m_d$ ) ( $n=171$ ); (B) relation between NMR intensity measured for wheat and the mass of water in the single kernels depending on the dry grain mass ( $n=283$ ) (for kernels in the range  $0.017 < m_d < 0.073$ ).

with a correlation factor  $R^2=0.87$ . The relation (2) shows, that not only the mass of water in the particle plays an important role but also the dry mass of the particle has an impact on the measured NMR intensity. A similar dependency was observed by Kraszewski *et al.* (1991) for the average frequency shift while measuring the moisture of single corn kernels by the microwave resonator techniques. However, the transmission factor was essentially independent of the mass of the dry kernel. In contrast, Miller *et al.* (1980) stated that pulsed nuclear magnetic resonance measurements were not affected by the particle size and that weighing was not necessary if the sample packing did not vary significantly from the one used in the calibration. However, single kernels can widely vary in size and weight, thereby, influencing the measurements.

To achieve confidence level, 30 kernels were randomly selected from the respective batch for each type of grain. Then, the moisture content of each kernel was examined in dependence on its angular position in the NMR spectroscope. After that, the arithmetic mean was calculated from the 30 individual measurements at each kernel position. Firstly, the particle moisture distribution was determined at eight angular positions with an angular distance of  $45^\circ$  for different types of grain. The results are shown in Figure 4 where the particle moisture content is illustrated as a function of the grain angular position in the NMR spectrometer. Each dot in the diagram represents the mean value of 30 individual grains which were measured in any of the eight positions. As can be seen from the figure, the particle moisture distribution consistently follows a pronounced sinusoidal curve for all types of grain.

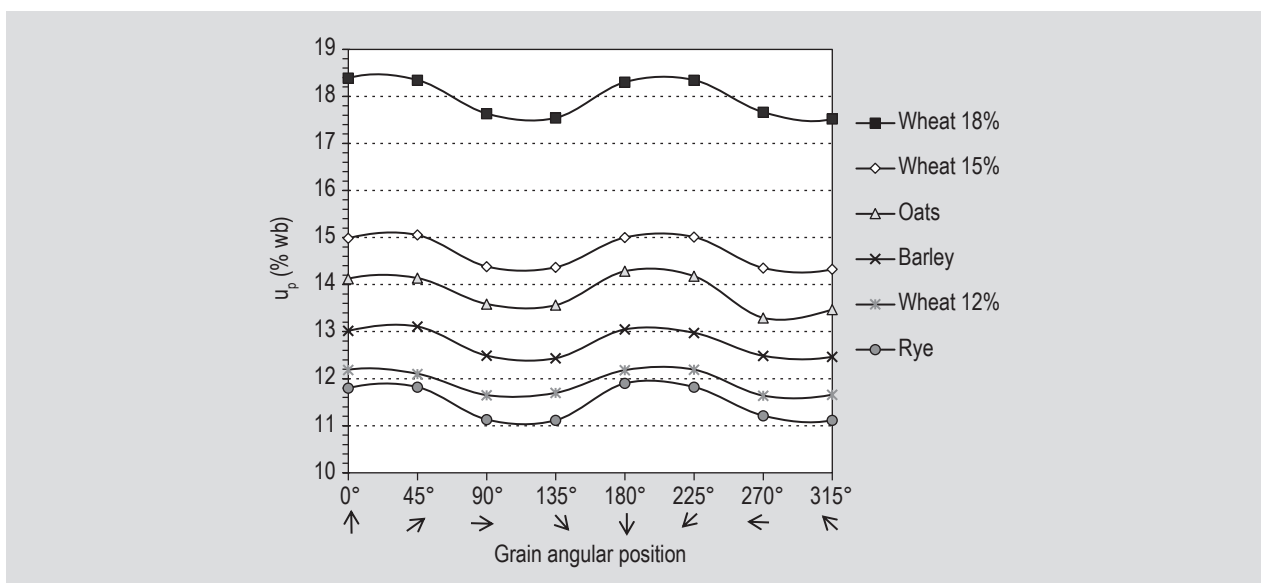


Figure 4. Particle moisture content ( $u_p$ ) as a function of the grain angular position measured for the grain types: barley, rye, oats, and wheat (mean values from  $n=30$  kernels each).

This tendency is also visible in Figure 5, where the particle moisture distribution of wheat with 15% water content is depicted in a higher resolution. At the ordinate, the differences of the measuring values from the arithmetic mean (14.7% wb) are plotted. At the example of this test series, an error calculation was performed to analyse the accuracy of the particle moisture determination using TD-NMR spectroscopy. The error bars in the diagram include the scattering caused by the different masses of water per kernel. Kernels with higher moisture content can have greater scattering values (Table 1). From the results obtained it can be inferred that the orientation of the longitudinal axis of a single kernel is decisive. However, it is irrelevant whether the germ is in front or back. The moisture values obtained at the positions 0°, 45°, 180° and 225° are clearly higher than those at 90°, 135°, 270° and 315°.

In a second experimental series with wheat of 12% moisture, the particle moisture distribution was examined at a higher angular resolution. The experiments were performed at 16 kernel positions with an angular distance of 22.5°. The results are shown in Figure 6. As can be seen, the measurements follow a pronounced sinusoidal curve as a function of the rotation angle. Due to the finer subdivision, it was possible to determine the minima and maxima from the moisture course. In addition, the arithmetic mean of all single measurements of  $\bar{u}_p = 11.9\%$  is depicted in the diagram as a solid line corresponding to the zero line. The maximum of the  $\Delta\bar{u}_p$  distribution was 0.37% wb obtained at a rotation angle of 202.5° (Figure 6). The moisture minimum was -0.38% wb at a rotation angle of 292.5°. The absolute moisture difference between maximum and minimum was 0.75% wb.

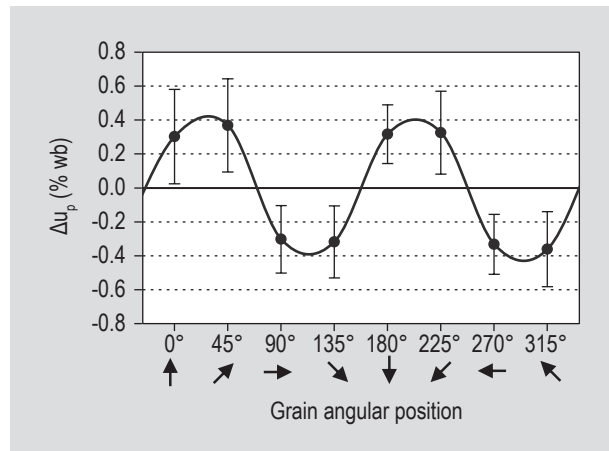


Figure 5. Particle moisture content ( $u_p$ ) of wheat with 15% moisture vs the grain angular position. The zero line depicts the average moisture content of  $\bar{u}_p = 14.7\%$  wb. The error bars show the standard deviation of the 30 single kernel values.

Table 1. Minima and maxima of the particle moisture distributions as well as the differences between them obtained for wheat (rounded to one decimal place).

Wheat sample	Moisture content	Minimum	Maximum	Difference
1	12%	11.5%	12.3%	0.8%
2	15%	14.3%	15.1%	0.8%
3	18%	17.5%	18.4%	0.9%

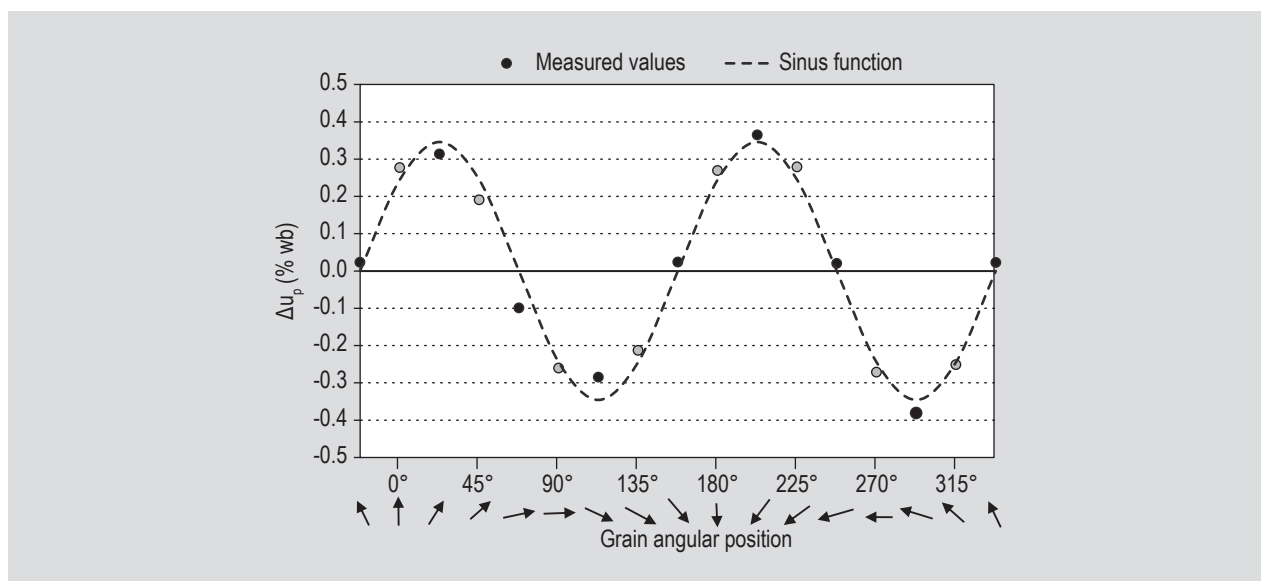


Figure 6. Particle moisture content ( $u_p$ ) of wheat with 12% moisture vs the grain angular position. The zero line denotes the average moisture content of  $\bar{u}_p = 11.9\%$  wb. Dots represent mean values from  $n=30$  single kernels. The dashed line depicts the particle moisture calculated with Equation 3.

As the results show, the measuring values are strongly affected by the angular position of the single grain in the TD-NMR. On average, the values of the NMR intensity differed by 5.6% when the grain was turned by 90°. This corresponds to a deviation in the moisture of 4.7% wb. These moisture differences increased as the grain moisture content increased. Table 1 shows the minima and maxima of the particle moisture for wheat samples of different moisture content depending on the kernel position as well as the differences between minima and maxima. The values for wheat with 15 and 18% grain moisture were estimated from the courses of the individual curves. As the table illustrates, the moisture difference can reach up to 0.9% wb. However, the reference method ASAE S352.2 FEB03 (ASAE, 2006) selected only allows a deviation of 0.2% wb between the prescribed double determinations and the ICC 110/1 (ICC, 1976) standard for moisture content determination for grounded grain even only allows a difference of 0.15% wb. Also in case of the DIN EN ISO 10565 (ISO, 1998) standard for simultaneous determination of oil and water contents in oilseeds by pulsed nuclear magnetic resonance, the water content must have a repeatability of 0.2-0.3% wb (m/m). This makes clear that the angular position of a grain is very important for single grain moisture measurements in TD-NMR. To keep the error small, it is practicable to measure all grains in the same position and also calibrate the method at this position.

The measured particle moisture distributions as illustrated in Figure 4, 5 and 6 can be described by a sinus function as:

$$u_p = a \times \sin 2 \times (\phi + b) \quad (3)$$

where  $u_p$  is the particle moisture,  $a$  denotes the amplitude of the function,  $\phi$  the rotation angle of the grain kernel and  $b$  denotes the phase shifting angle, in our case  $b=22.5^\circ$ .

For wheat with 18% moisture, the value of the amplitude is  $a=0.534$  ( $R^2=0.985$ ), for wheat with 15% moisture  $a=0.465$  ( $R^2=0.995$ ) and for 12% moisture  $a=0.346$  ( $R^2=0.974$ ). Accordingly, the amplitude increased as the mean moisture content was increased. This tendency coincides with the increasing moisture differences presented in Table 1.

In the NMR spectroscope, all protons of a sample are reached by the external magnetic field (Figure 7). This is the main advantage of the NMR as compared to other measuring methods with a limited penetration depth such as the NIR technique. Therefore, one would suppose that the cross-section of the grain stretched in the magnetic field and, hence, its angular position should not influence the measurements. This assumption was confirmed by examinations of the position of the germ, which has a higher concentration of protons due to a higher moisture content (Klingler, 2010). These measurements revealed the same moisture values when the kernel was turned by 180°. This effect was also observed when measuring the particle moisture distribution of wooden cylindrical sticks with an almost homogeneous moisture profile over the length. However, the moisture contents differed from each other when the kernel was rotated by 90°. This was the case when comparing the values measured for example at the 0° and the 90° position, but also at the 45° and the 135° position (Figure 5).

In Figure 7, the arrangement of the external magnetic field of the NMR unit is schematically depicted for different angular positions of a grain kernel. As is clear from the figure, the magnetic field transits the same cross-section of the kernel both in the 45° position and in the 135° position. As expected, the particle moisture content measured at these two angular positions should be the same or at least comparable. This was not the case as explained above. Hence, there must be another reason for the moisture differences observed.

#### 4. Conclusions

For the non-destructive measurement of the single particle moisture content, the TD-NMR spectroscopy was applied and adopted. The TD-NMR is widely used in food industry for QC/QA applications where precise determination of moisture and water is of fundamental interest. Although the TD-NMR has become an appreciated measurement tool in food science and technology, there is only limited information about the influence of the position of the single kernel in the measuring cell while measuring the moisture

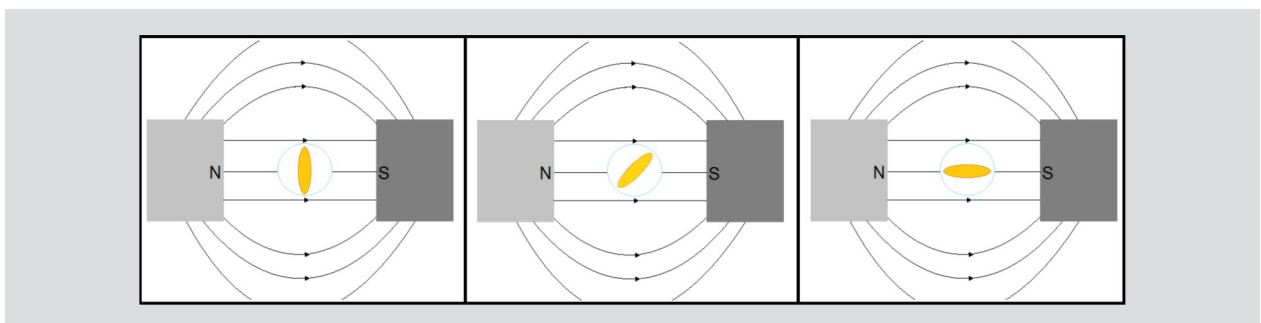


Figure 7. Schematic of the external magnetic field for different grain kernel positions (0°, 45°, 90°).

content. This influence has been examined more closely in this study for different types of grain such as wheat, barley, rye, and oats. As could be demonstrated, the particle moisture significantly varied depending on the angular position in the TD-NMR spectroscope. For all types of grain examined, this relation consistently obeyed a pronounced sinusoidal curve. When the kernel was turned by 90°, the moisture of wheat grains significantly deviated by up to 0.9% wb. This deviation increased as the grain water content increased. It should be noted that also the dry mass of the single kernels slightly affected the moisture measurement in the TD-NMR device. The effect of the kernel angular position is possibly device-specific and most likely caused by inhomogeneities of the magnetic field. For example, commercial permanent magnets which are used in low-field benchtop NMR instruments are limited in their applicability due to strong inhomogeneity of the magnetic field.

As a consequence, the kernel should always have the same position in the NMR spectroscope, and also the calibration should be carried out in this position. To minimise the effect of the different dry mass of single kernels, it is important to calibrate the TD-NMR in a wide range of kernel weight. The method developed will be used to investigate the single kernel moisture distribution in postharvest processes of grain.

## Conflict of interest

The authors declare that they have no conflict of interest.

## References

- American Society of Agricultural and Biological Engineers (ASAE), 2006. Moisture measurement – unground grain and seeds. ASAE S352.2 FEB03. ASAE, St. Joseph, MI, USA.
- Askochenskaya, N.A. and Golovina, E.A., 1981. State of water in seeds and its alterations under various actions. *Studia Biophysica* 85: 19-20.
- Brusewitz, G.H., 1987. Corn moisture variability during drying, mixing, and storage. *Journal of Agricultural Engineering Research* 38: 281-288.
- Chambers, J., McKeivitt, N.J., Scudamore, K.A. and Bowman, C.E., 1989. Non-destructive determination of the moisture content of individual wheat grains by nuclear magnetic resonance. *Journal of the Science of Food and Agriculture* 49: 211-224.
- Christensen, C.M., Miller, B.S. and Johnston, J.A., 1992. Moisture and its measurement. In: Sauer, D.B. (ed.) *Storage of cereal grains and their products*. American Association of Cereal Chemistry, Saint Paul, MN, USA, pp. 39-54.
- Cunäus, U., 2010. Populationsdynamische beschreibung der kontinuierlichen wirbelschichttrocknung. PhD thesis, Otto-von-Guericke-University, Magdeburg, Germany, 136 pp.
- Hahn, E.L., 1950. Spin echoes. *Physical Review* 80: 580-594.
- International Association for Cereal Science and Technology (ICC), 1976. Standard method 110/1: determination of the moisture content of cereals and cereal products (practical method). ICC, Vienna, Austria.
- International Organization for Standardization (ISO, 1998). ISO 10565:1998. Oilseeds – simultaneous determination of oil and water contents – method using pulsed nuclear magnetic resonance spectrometry. ISO, Geneva, Switzerland.
- Klingler, R.W., 2010. *Grundlagen der getreidetechnologie*. Behr's Verlag, Hamburg, Germany, 356 pp.
- Kraszewski, A.W., Nelson, S.O. and You, T.S., 1991. Moisture content determination in single corn kernels by microwave resonator techniques. *Journal of Agricultural Engineering Research* 48: 77-87.
- Kraszewski, A.W., You, T.S. and Nelson, S.O., 1989. Microwave resonator technique for moisture content determination in single soybean seeds. *IEEE Transactions on Instrumentation and Measurement* 38: 79-84.
- Liu, Q., Montross, M.D. and Bakker-Arkema, F.W., 1997. Stochastic modelling of grain drying: part 1. Experimental investigation. *Journal of Agricultural Engineering Research* 66: 267-273.
- Miller, B.S. and Kaslow, H.D., 1963. Determination of moisture by nuclear magnetic resonance and oven methods in wheat, flour, doughs, and dried fruits. *Food Technology* 17: 142-145.
- Miller, B.S., Lee, M.S., Hughes, J.W. and Pomeranz, Y., 1980. Measuring high moisture content of cereal grains by pulsed nuclear magnetic resonance. *Cereal Chemistry* 57: 126-129.
- Morley, J.H., Fletcher, P.D. and Morgan, A.G., 1984. The estimation of moisture in whole grain wheat and barley using nuclear magnetic resonance spectroscopy. *Journal of the National Institute of Agricultural Botany* 16: 437-442.
- Peglow, M., Cunäus, U. and Tsotsas, E., 2011. An analytical solution of population balance equations for continuous fluidized bed drying. *Chemical Engineering Science* 66: 1916-1922.
- Peglow, M., Metzger, T., Lee, G.H., Schifter, H., Hampel, R., Heinrich, S. and Tsotsas, E., 2009. Measurements of average moisture content and drying kinetics for single particles, droplets and dryers. In: Tsotsas, E. and Mujumdar, A.S. (eds.) *Modern drying technology, Volume 2: experimental techniques*. Wiley-VCH, Weinheim, Germany, pp. 1-71.
- Persyn, G.A. and Rollwitz, W.L., 1971. Transient NMR quantitative measurements. *Journal of the American Oil Chemists' Society* 48: 67-69.
- Pohle, W.D. and Gregory, R.L., 1968. Application of wide-line NMR to analysis of cereal products and fats and oils. *Journal of the American Oil Chemists' Society* 45: 775-777.
- Rutledge, D.N., 2001. Characterisation of water in agro-food products by time domain-NMR. *Food Control* 12: 437-445.
- Srinivasan, V.T., 1979. A comparison of different pulsed sequences in the nondestructive estimation of seed oil by pulsed nuclear magnetic resonance technique. *Journal of the American Oil Chemists' Society* 56: 1000-1003.
- Tiwari, P.N., Gambhir, P.N. and Rajan, T.S., 1974. Rapid and nondestructive determination of seed oil by pulsed nuclear magnetic resonance technique. *Journal of the American Oil Chemists' Society* 51: 104-109.

Todt, H., Guthausen, G., Burk, W., Schmalbein, D. and Kamlowksi, A., 2006. Water/moisture and fat analysis by time-domain NMR. *Food Chemistry* 96: 436-440.

Van Duynhoven, J., Voda, A., Witek, M. and Van As, H., 2010. Time-domain NMR applied to food products. *Annual Report in NMR Spectroscopy* 69: 145-197.