BUDAPESTI CORVINUS EGYETEM

Élelmiszertudományi Kar

FACTORS AFFECTING THE CALIBRATION TRANSFER OF DIELECTRIC MOISTURE METERS

Thesis Book

by

Zoltán Gillay

Supervisors:

Prof. David B. Funk
Prof. András Fekete

Corvinus University of Budapest
Faculty of Food Science
Department of Physics and Control

Budapest, 2010

PhD School/Program

Name: PhD School of Food Science

Field: Food Science

Head: Prof. Péter Fodor, DSc
Corvinus University of Budapest

Supervisor: Prof. David B. Funk, PhD, DSc (hc)
Associate Director for Methods Development
USDA-GIPSA-Technical Services Division

Prof. András Fekete, DSc
Corvinus University of Budapest,
Faculty of Food Science,
Department of Physics and Control

The applicant met the requirement of the PhD regulations of the Corvinus University of Budapest and the thesis is accepted for the defence process.

....
Signature of Head of School Signature of Supervisors

1. INTRODUCTION

Grain moisture content is one of the most important factors affecting the price of the grain, so the fast and accurate moisture measurement is particularly important. One of the most challenging problems is that the different types of equipment yield diverse results; they are not uniform. This is a serious problem in the grain market. To address this problem, a given type of equipment was chosen for the U.S. official grain moisture meter. There is a master unit at the United States Department of Agriculture - Grain Inspection, Packers and Stockyard Administration (USDA-GIPSA) to which the same type of equipment used for official grain inspection are standardized. There remain disadvantages to this solution. This does not solve the international uniformity of grain moisture measurements, and the grain moisture meter manufacturers are not motivated in the development of new technology.

The USDA-GIPSA developed a new grain moisture method and calibration which is more reliable and more accurate than the recent grain moisture meters on the market. This method may be able to be the new official moisture meter technology in the U.S.; moreover the worldwide spread is intended to be promoted. Hence the development results and the calibration are not patented but they are public for everybody to use. Any of the manufacturers, with relative low cost using calibration transfer, can develop and commercialize their own designed equipment.

The new technology has multiple novelties. One of the most important things is that the measurement frequency is 149 MHz, which is higher than the frequency used by most of the recent grain moisture meters. The test cell is not a capacitive type test cell but a transverse electromagnetic (TEM) mode transmission line (Figure 2.). The measurement arrangement is reflection mode. The moisture prediction process consists of multiple steps, as figure 1 demonstrates.

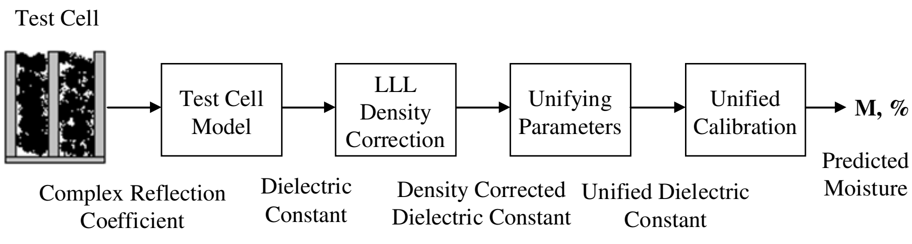

Figure 1. Schematic flow graph of the VHF-Unified Grain Moisture Algorithm

The dielectric constant is calculated from the complex reflection constant using the mathematical model of the test cell. A density correction derived from the Landau-Lifshitz-Looyenga (LLL) mixture equation reduces the effect of the density variation from loading to loading. Finally, with linear transformations on the density corrected dielectric constant, only one unified calibration is enough to predict the moisture for all grain types more accurately than most of the recent moisture meters. The temperature correction is performed on the predicted moisture.

For consideration of adoption of this method as official grain moisture meter technology, it is important that different devices made by different manufacturers have to be introduced into the market. These moisture meters should use compatible methods and calibration so they yield moisture results in a certain moisture error range relative to the master test cell at the USDA-GIPSA. The manufacturers want to design equipment with different test cells, loading methods, and measurement setups to target separate customers' needs. At the initial stage of the research, one test cell was used, called the master test cell in my dissertation. The size of this test cell was too large to make practical equipment based on it. So the research had to reveal what changes could be applied while retaining the original calibration and yielding results within the allowable error range. Furthermore, a standardization method was needed to achieve the requirements of the US standards.

2. OBJECTIVES

The PhD research was focused on the factors affecting the standardization of the dielectric type grain moisture measurement. The research included assessment of some possible alternative measurement arrangements that could be more advantageous than the original setup.

The research steps in details:

1. Determine the possible test cell arrangements and methods using the VHF-UGMA method and reveal their advantages and disadvantages. The most highlighted possibility was the method using only the magnitude of the reflection coefficient (instead of both magnitude and phase).
2. Improve the test cell model
    - to be capable of interpreting diverse type of test cells,
    - for investigation and optimization of the effects of the geometrical and electrical parameters.
3. Apply suitable calibration and standardization materials, and develop standardization methods. The most important question was to answer whether the materials with almost zero loss factor were appropriate for standardization or not.
4. Determine the effect of different practically important loading methods:
    - Assess which loading method is the more advantageous considering repeatability.
    - Determine the differences in predicted moisture using different loading methods.
    - Determine the reasons for the possible differences and develop corrections.
    - Quantify the effects of changing the loading methods and/or the size of the test cell on calibration transfer, and develop successful calibration transfer methods.

3. MATERIALS AND METHODS

3.1 Test Cells and Measurement Procedure

The original master test cell, which was used for developing the method and the calibration, was available for our research. For further investigations a prototype test cell was built, shown in figure 2. The prototype test cell was electrically similar to the master test cell but was much smaller and simpler mechanically.

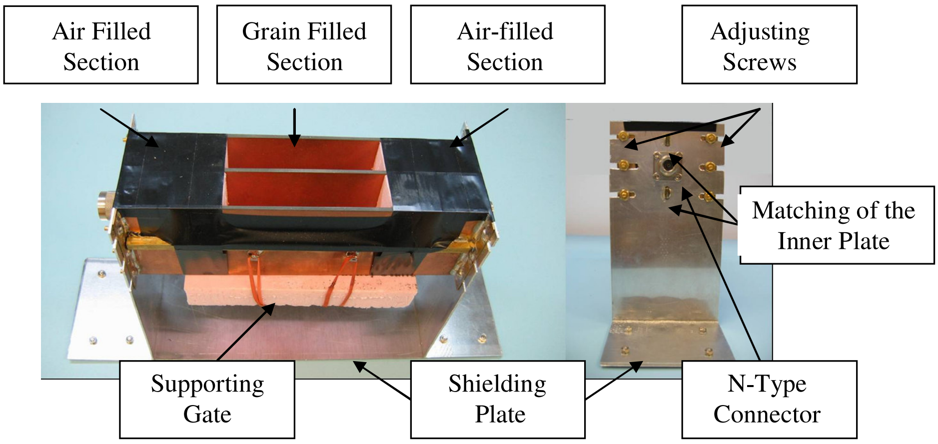

Figure 2. Prototype test cell for the investigation of calibration transfer

The prototype test cell consisted of three parallel plates similar to the master test cell. Double-sided copper-clad epoxy-glass circuit board material (3.2 mm thickness) was used because of its rigidity and solderability. Aluminum plates at both ends of the test cell supported the parallel plates and the N-type connectors. The position of the outer plates (electrode spacing) was adjustable by loosening the screws. The conductive copper cladding was removed from a 6 mm vertical strip at each end of the inner plate to provide a nonconductive support. Tabs machined on the center plate fit into two slots on each aluminum plate to fasten it rigidly. The inner plate was connected to the inner electrode of the connector at each end. A shielding plate 10 cm below from the test cell reduced the electrical coupling to nearby objects. The air-filled section was filled by polystyrene foam, defined the transverse length of the grain section increased the mechanical stability of the test cell. A polystyrene foam plate supported the grain below the test cell, also. The test cell was connected to the measurement device via 50 ohm precision cable. Different terminations could be used at the end of the test cell. The original termination was the matched case (50+0j ohm precision load). In addition, other possibilities existed, such as the extreme cases of shorted and open terminations.

The loading of the grain samples was performed by pouring the samples to overflow the test cell, then striking-off the excess by an official bushel weight stick. The complex reflection coefficient was measured between 1 and 501 MHz at 2 MHz intervals. The sample temperature and mass were measured and recorded. Funnel, manual pouring and a fast drop mechanism were used for the investigation of the loading methods.

3.2 Materials

Calibration Materials

High molecular weight alcohols were used for the dielectric calibration: decanol, hexanol, and pentanol. The permittivities of these alcohols are in the permittivity range of grains, in addition they are neither very toxic nor extremely hygroscopic. We believe that they did not gain a significant amount of moisture during the measurements. The disadvantage of using alcohols was that the test cell must be sealed against leakage. The theoretical permittivities for the used frequency range were calculated using the Debye equation with parameters found in the literature.

Standardization Materials

Standardization was distinguished from calibration because the main goal of this research was the agreement not to the reference method but to the master test cell. That implies that it is not necessary to know the permittivity of the standardization material--just the value measured by the master test cell.

Alcohols are not appropriate for routine practical standardization, hence other materials were needed. For our test cell comparison investigations, 8 grain groups of 328 samples were used. The disadvantages of standardization using grain grain were that the grain was not stable (because it lost water during the measurements) and it spoiled eventually. Granular innorganic materials were found that were mechanically and chemically stable, readily available, consistent among different lots, and with permittivities in the range of grains. Figure 3 shows the three useful materials we identified.

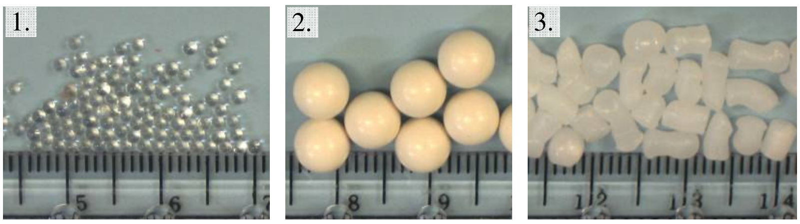

Figure 3. Standardization materials: 1. Lead free soda lime glass grinding beads, 1.2 mm average diameter; 2. Soft Air, 0.20 grams, 6 mm diameter by Crosman; 3. Stuffing plastic pellets (polyethylene)

Grain Samples Used for the Investigation of the Loading Effects

Seven Hungarian grains were used for the loading tests: barley, wheat, corn, sunflower, rapeseed, soybeans, and oats. Twenty repeats were performed for each of three loading methods: funnel, manual pouring, and fast drop. Dry and rewetted samples were used to assess moisture dependence.

4. RESULTS AND DISCUSSION

4.1 Measurement Methods

The first promising alternative method was the short termination using a conductive plate at the end of the test cell. This arrangement is more rigid and simple than the original. The tests showed that the sensitivity could be adequate for measurement if the air-filled section length is appropriate.

The second viable alternative was to use only the magnitude of the reflection coefficient (Figure 4). The original algorithm uses the complex reflection coefficient to calculate the dielectric constant (Figure 1). The dielectric constant, which is the basis for the moisture determination, can be predicted from only the magnitude of the reflection coefficient using a 5th order polynomial. The error of the predicted moisture using a polynomial to predict dielectric constant was lower than the original method if the UGMA calibration was redeveloped based on dielectric constant predicted by the polynomial equation. This method is potentially far simpler both electrically and computationally than using both magnitude and phase of the complex reflection coefficient.

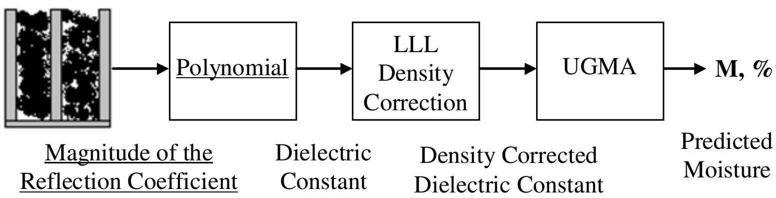

Figure 4. Schematic flow graph of algorithm using only the magnitude of the reflection coefficient

4.2 Test Cell Modeling

Improving the test cell model was an important part of the research. We applied the ABCD matrix approach to modeling this relatively large parallel plate test cell. The method used matrices for each of the sections as shown in figure 5. The ABCD matrix of the whole test cell could be given by multiplying these matrices. Using conversions on the ABCD matrix, the reflection coefficient could be calculated explicitly. The permittivity of the measured material could be calculated iteratively.

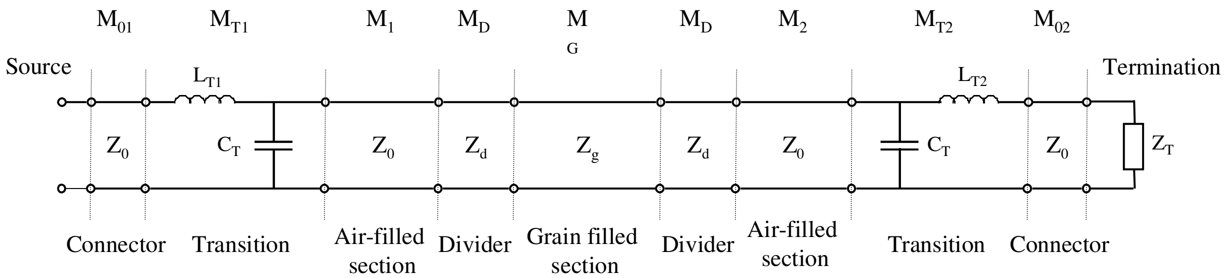

Figure 5. The sections of the ABCD matrix model

All the parameters in the model have physical meanings so they can be calculated by measurements or finite element methods--except the parameters of the transition sections. These parameters were defined by using calibration and standardization measurements. The effectiveness of the ABCD model was tested using alcohol calibration measurements. The ABCD matrix and the original signal flow graph model were optimized to fit the measurement results. The transition section parameters (L_{T1}, L_{T2}, C_T) and the empty cell correction parameter (Corr) were optimized for the ABCD matrix model, and the first air-filled section length (d_1), filling factor, and empty cell correction parameter (Corr) were optimized for the signal flow graph. The results showed that the ABCD matrix approach yielded better fit for the dielectric constant but similar fit for the loss factor. The great advantage of the new model was that effects of the test cell characteristics could be investigated theoretically.

4.3 Test Cell Standardization

Three standardization methods were tested to compare their effectiveness. The methods were: 1. several grain samples using the dielectric constant measured at 149 MHz, 2. three grain samples using the complex permittivity at frequencies between 50 and 250 MHz. (The grain samples used for the standardization were randomly chosen in 114 cases), and 3. the standardization materials shown above using the complex permittivity at frequencies between 50 and 250 MHz.

For the statistical description of the effectiveness, the RMSE values for the real part and the imaginary part, and the mean difference and the RMSE value of the moisture results relative to the master test cell were used. The results are summarized in the table 1.

Table 1. Summary of the results of the different standardization process

	Grain (55 samples)	Grain	Standardization Materials
	Single Frequency	Multiple Frequency	
RMSE (Real Part, All Samples)	0.0158	0.0132	0.0158
RMSE (Imaginary Part, All Samples)	0.0323	0.0447	0.042
Bias (Moisture), %	0	0.031	0.03
RMSE (Moisture) , %	0.048	0.064	0.061

The results show the low loss standardization material provided similarly effective standardization as the methods using grain samples.

4.4 Loading Effects on Moisture Measurement

Three practical loading methods (funnel, manual pouring, and fast drop loading) were compared to assess the variability of the measured moisture and mass using different loading methods. The other question was whether the different loading method would yield different predicted moisture results.

The standard deviation (repeatability) of the mass results showed significant increases for a few grain types, but for moisture the applied density correction reduced the variability.. The mean moisture results were surprisingly different for the different loading methods (Figure 6). Several grain types (barley, wheat and oats) showed practically significant differences. The moisture results were the highest for manual pouring and lowest for fast drop for wheat and barley and for the others close to equal. The moisture differences mostly increased with the moisture content. The investigation of possible reasons revealed that moisture differences were not correlated with mass differences but there was a good correlation with the degree of elongation of the kernels. The results for rough and milled rice showed that the hull did not play a significant rule in the moisture difference.

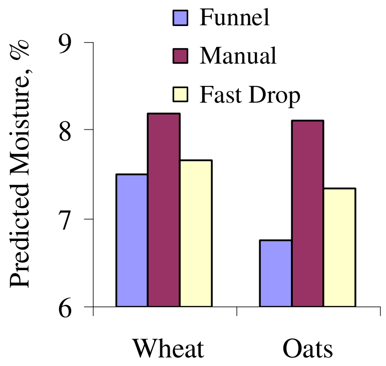

Figure 6. The averages of the predicted moisture

4.5 The Effect of the Spatial Orientation of Kernels

Based on the results it became obvious that the reason for the moisture difference was the kernel orientation caused by the loading method. That is why the orientation of the kernels in the cell was investigated in more detail. Pictures were taken (top and side views) using a transparent box. The side views showed unambiguously that the orientation of the kernels was not random but aligned in a certain order and direction. As figure 7 shows, the kernel arrangement is predominantly horizontal for funnel and manual loading and random for fast drop. For the top view, the differences in orientation of the kernels were not unambiguously visible, hence a method was introduced to quantify the order and the direction of the kernels.

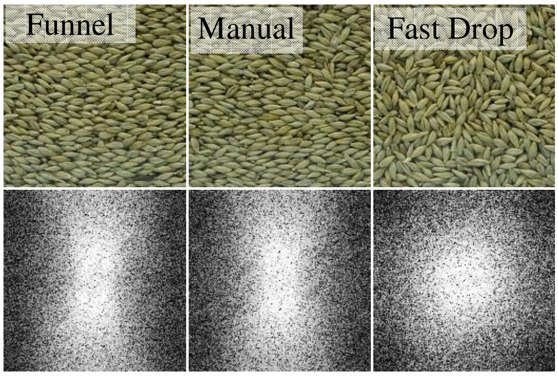

Figure 7. Side views using transparent box and their 2D Fourier-transformed pictures

The method was based on the 2-dimensional Fourier transformation. On the Fourier-transformed pictures (Figure 7) the clouds of white points characterize the direction and order of the alignment. The results derived from the top view pictures showed that the direction of the kernels tended to be parallel with the plates for funnel loading, perpendicular for manual, and random for fast drop loading. A dielectric ellipsoid most strongly decreases the electric field in the direction of its main axis, so ellipsoids aligned with the electric field would show the highest apparent dielectric constant (Figure 8.) The moisture difference was explained based on this theory and the described results.

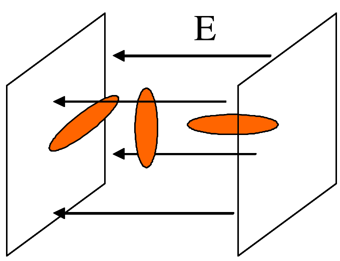

Figure 8. The main kernel directions

4.6 Moisture Calibration Transfer

The main goal of the research was to determine the effects of each factor affecting grain moisture measurement and, via this, to achieve calibration transfer. The most important question was how the size of the test cell and the loading method affect the measurement and how could resulting differences be corrected. To answer this question, 328 grain sample measurements were used. The measurements were performed on the two test cells (master and prototype) and two loading methods (funnel and fast drop). The standardization was fulfilled using all the 55 soy samples. The results showed that the change of the test cell size or the loading method caused grain group dependent moisture differences relative to the original arrangement. The average differences can be seen in figure 9.

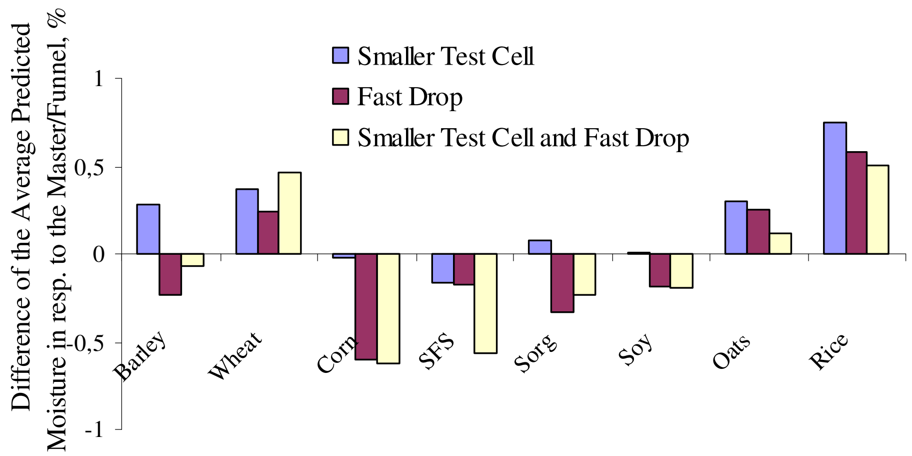

Figure 9. Differences of average predicted moisture relative to master test cell using funnel loading

There were several possibilities to introduce grain group dependent calibration transfer parameters. The first possibility was to optimize (by grain group) the target mass in the density correction to minimize the moisture differences relative to the master test cell using funnel loading. This was interesting because only one parameter was enough to be adjusted. The other plausible possibility was to use a simple linear correction with two parameters. The results showed that the optimized target mass by grain group yielded similarly good results as the two parameter linear correction.

To determine the optimum values for the correction, several grain samples were needed for each grain group, hence the possibility of the determination of these parameters from the physical and chemical parameters of the kernels was investigated. The calculations showed that there was not a significant correlation between needed corrections and any one kernel characteristic. Multivariate models were developed to predict the optimal target mass from the physical and chemical parameters. The prediction and the cross validation were successful using three variables, but the results were not scientifically explainable.

5. NEW SCIENTIFIC RESULTS

Configurations

1. I proved using rewetted wheat samples that the shorted test cell is an advantageous alternative to the matched load arrangement, because the sensitivity of complex reflection coefficient for the moisture content almost is equal to the matched load arrangement. The sensitivity increases with the increase in the distance of the shorting plate from the grain section, as I proved up to 10 cm. The sensitivity of the magnitude of the reflection coefficient is close to equal for 10 cm distance, and the phase sensitivity is four times larger compared to the matched case.

2. I proved that change of measurement frequency in a +/-30 MHz frequency range is possible using a linear correction on the dielectric constant, and the moisture results will remain in the acceptable error range. The transverse electromagnetic measurement limit was determined to be about 300 MHz, and the theoretical limit is at about 1.5 GHz.

3. I proved that using only the magnitude of the reflection coefficient is sufficient to predict the grain moisture content. A 5th order polynomial is necessary to predict the dielectric constant. Using the originally developed VHF-UGMA parameters, the calculated moisture is not equivalent to the moisture prediction derived from the complex reflection coefficient. The VHF-UGMA parameters had to be re-optimized. Then the RMSE value was equal to the value for the original algorithm.

Model

4. I applied the ABCD matrix representation successfully for for a parallel-plate transmission line type test cell with dimensions appropriate for grain moisture measurement.. In the model I developed the test cell size, the characteristic impedance, and the termination (which determined the measurement mode) were considered. Furthermore, I optimized the test cell parameters theoretically. The model gave a little better fit for the alcohol calibration, optimized transition parameters, and the empty cell correction than the original signal flow graph model.

Standardization

5. I found materials with almost zero dielectric loss to be appropriate for standardizing the test cells. The moisture errors for soybeans were in the tolerance range of the National Institute of Standards and Technology in case of standardizing the test cell by low loss materials, applied in our research, using the complex permittivity at 1, 89, 127, and 165 MHz.

Loading Effects

6.a The investigated loading methods (funnel, manual pouring, and fast drop) did not show practically significant differences in moisture repeatability using the VHF-UGMA method. The LLL density correction was effective reducing the variability of the moisture for only the manual pouring but it caused increment of the variation at few grain types for the funnel and the fast drop. The reason was for this that the variation of the moisture measurement is not only due to the variation of the bulk density but also due to the variation of the order and the direction of the kernels.

6.b I revealed that the predicted moisture is significantly different using different loading methods. The oats sample with 20% moisture content showed a 3.5% moisture difference. The moisture difference is caused not by the difference of the bulk density but by the difference of the kernel orientation. Neither the LLL density correction nor any of the other density corrections are not able to correct the effect of the changing orientation in practical use because the order and the direction of the kernel orientation are not measureable by practical methods.

6.c I proved that the moisture difference between the different loading methods is grain moisture content dependent. The hull of the kernel does not play significant role in the moisture difference. The difference of the dielectric constant is not obviously frequency dependent in the 1-250 MHz frequency range.

Spatial Orientation

7. I proved using 2D Fourier-transformed pictures that the different loading methods caused different spatial orientation tendencies for the kernels.

- With funnel loading the kernels tended to lie parallel with the plates of the test cell,
- with manual pouring perpendicular to the test cell plates, the kernels tended to lie perpendicular to the plates of the test cell, and
- with fast drop the kernels showed random orientation.

Calibration Transfer

8. I proved that moisture calibration transfer to a test cell with different geometry or loading method does not work without grain group dependent correction despite the relatively perfect standardization. The linear grain group dependent correction is exquisitely appropriate to perform the calibration transfer. The correction is possible using only one parameter; by optimizing the target mass for each grain group, we can get a practical and adequate calibration transfer.

6. PUBLICATIONS RELATED TO THE THESES

Publications with Impact Factor

1. Funk D. B., Gillay Z. (2010) Dielectric reference materials for mathematically modeling and standardizing grain moisture meters. Transactions of the ASABE, 53(1), pp. 271-281. IF: 1.042.
2. Funk D. B., Gillay Z., Mészáros P. (2007) Unified moisture algorithm for improved RF dielectric grain moisture measurement. Measurement Science and Technology, 18(4), pp. 1004-1015. IF: 1.297.

International Conference (Full)

1. Funk D. B., Gillay Z. (2009) Dielectric Reference Materials for Mathematically Modeling and Standardizing Grain Moisture Meters. ASAE Annual Meeting, Reno, Nevada, Paper No. 096651. http://asae.frymulti.com/abstract.asp?aid=27085&t=1
2. Funk D. B., Gillay Z. (2009) Strategies for Modeling Dielectric Materials and Dielectric Test Cells. ISEMA, Helsinki, Finland, pp. 23-46.
3. Gillay Z., Funk D. B. (2007) Effects of Test Cell Size and Loading Method on Calibration Transfer. ISEMA, Hamamatsu, Japan, pp. 227-234.
4. Gillay Z., Funk D. B. 2006. Adapting the Unified Grain Moisture Algorithm to Commercial Instruments. IMTC 2006 – Instrumentation and Measurement Technology Conference, Sorrento, Italy, pp. 317-322.
5. Gillay Z., Funk D. B. (2005) Analysis of Frequency Sensitivity of the Unified Grain Moisture Algorithm. ASAE Annual Meeting,Tampa, Florida, Paper No. 053047. http://asae.frymulti.com/abstract.asp?aid=19068&t=1
6. Gillay Z., Funk D. B. (2005) Calibration Transfer for the Unified Grain Moisture Algorithm. ISEMA, Weimar, Germany, pp. 473-480.
7. Funk D. B., Gillay Z., Rampton J. H., Freese L. D., Mészáros P. (2004) New Dielectric Moisture Measurement Technologies. International Quality Grains Conference, Indianapolis, Indiana. Paper on CD.
8. Gillay Z., Funk D. B., (2003) Sensitivity Analysis for VHF Dielectric Grain Moisture Measurements. ASAE Annual Meeting, Las Vegas, Nevada, Paper No. 033136. http://asae.frymulti.com/abstract.asp?aid=14073&t=1

Hungarian Conference (Full)

1. Hartyáni P., Gillay Z., Funk D. B. (2006) A betöltési módszerek hatása az új VHF gabonanedvesség-mérés hibájára. MTA AMB, Kutatási és Fejlesztési Tanácskozás, Gödöllő, pp. 155-159.
2. Gillay Z., Funk D. B. (2004) A cella geometeria hatása a nedvességmérés hibájára. MTA AMB, Kutatási és Fejlesztési Tanácskozás, Gödöllő, pp. 154-158.
3. Gillay Z., Funk D. B. (2003) Gabonanedvesség-mérő cellájának matematikai modellezése opzimalizálás céljából. MTA AMB, Kutatási és Fejlesztési tanácskozás, Gödöllő, pp. 97-100.

Hungarian Conference (Abstract)

1. Gillay Z., Funk D. B. (2005) Az egyesített gabonanedvesség-mérő módszer (UGMA) kalibrációjának átvihetősége. Lippay János - Ormos Imre – Vas Károly Tudományos Ülésszak, Budapest, pp. 258-259.
2. Gillay Z., Funk D. B. (2003) Tesztcella tervezés magasfrekvenciájú gabonanedvesség-méréshez. 5. Magyar Szárítási Szimpózium, Szeged, pp. 14-15.
3. Gillay Z., Funk D. B. (2003) Rádiófrekvenciás dielektromos gabonanedvesség-mérő matematikai modellezése. Lippay János - Ormos Imre – Vas Károly Tudományos Ülésszak, Budapest, pp. 228-229.

Citations

Funk D. B., Gillay Z., Mészáros P. (2007) Unified moisture algorithm for improved RF dielectric grain moisture measurement. Measurement Science and Technolog, 18(4), pp. 1004-1015.)

Cited by:

1. Cataldo A., Cannazza G., De Benedetto E., Tarricone L., Cipressa M. (2009) Metrological assessment of TDR performance for moisture evaluation in granular materials. Measurement, 42(2) pp. 254-263.
2. Cataldo A., Vallone M., Tarricone L., Cannazza G., Cipressa M. (2009) TDR Moisture Estimation for Granular Materials: An Application in Agro-Food Industrial Monitoring. IEEE Transactions on Instrumentation and Measurement, 58(8) 2597-2605.
3. Kaatze U. (2008) Perspectives in dielectric measurement techniques for liquids. Measurement Science and Technology, 19(11) pp. 1-4.
4. Reyes R., Castro-Giráldez M., Fito P., Reyes E. (2007) Application of Microwaves for On-Line Quality Assessment. Advances in Food Diagnostics, Blackwell Publishing, 49-79.

Gillay Z., Funk D. B. (2003) Sensitivity Analysis for VHF Dielectric Grain Moisture Measurements, ASAE Annual Meeting, Las Vegas, Nevada, Paper No. 033136.) http://asae.frymulti.com/abstract.asp?aid=14073&t=1

Cited by:

1. Benning R., Birrell S., Geiger D. (2004) Development of a Multi-Frequency Dielectric Sensing System for Real-Time Forage Moisture Measurement. ASAE Annual Meeting, Paper No. 041100. http://asae.frymulti.com/abstract.asp?aid=18474&t=1

ACKNOWLEDGEMENTS

I would like to thank:

- Dr. David Funk for providing the possibility to work on this project, and for the endless and valuable advising and help,
- Dr. András Fekete for his valuable support and advice,
- the Moisture Group at USDA-GIPSA for their contribution to this research,
- the Hungarian Education Ministry for the "Deak Ferenc" Scholarship, and
- my family for the their patience during my separation from them to write my dissertation.