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Faculty of Food Technology University of Josip Juraj Strossmayer in Osijek
European Federation of Food Science and Technology

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13th Ružička Days

"TODAY SCIENCE - TOMORROW INDUSTRY"

16th and 17th September 2010
Vukovar, Croatia

PROCEEDINGS

Hrvatsko društvo kemijskih inženjera i tehnologa
Prehrambeno-tehnološki fakultet Sveučilišta Josipa Jurja Strossmayera u Osijeku
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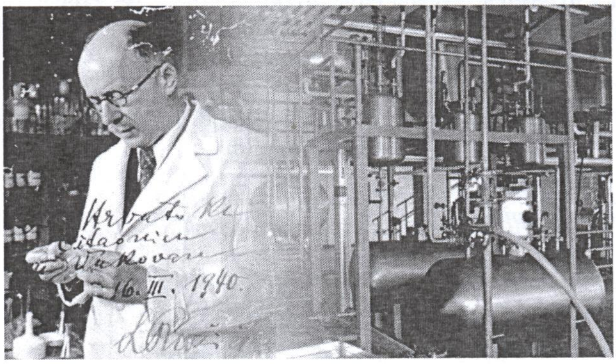

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Osijek, 2011.

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Technical criteria for the development of calibrations for moisture meters

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Summary

Dielectric (capacitive) technology used by the moisture meters for grains and oilseeds is based on the principle of the relation between the moisture content and the dielectric constant. The dielectric constant increases with the increase in moisture (water) content. Since the rate of increase is not the same for all types of grain, the unique calibration constants have to be developed for each type of grain whose moisture is measured using the moisture meter. Water content in grain is important for the storage of grain, and the accuracy of the results affects forming of the price on the market. Due to different measurement results, which are within the bounds of the maximum permissible error and occur in cases of the same model of moisture meters, there may be a lack of trust between the producers and the buyers of grain. Since it is a quick method and the instrument has a clearly defined permissible error at verification, the only solution to this problem is standardisation of calibration constants. The testing laboratory of the State Office for Metrology, which prepares samples for verification of moisture meters, has initiated the development of calibration constants for barley and wheat that render possible maximum level of homogeneity of measurement results. The laboratory has also set the technical criteria for making and testing in the first phase, followed by the implementation and the development of the constants for other agricultural cultures. For the purposes of development of calibration in this paper used the minimum number of prepared samples of barley (11) which is determined by the standard method of moisture, and has made corrections to the calibration of the existing commercial moisture meter. Measurement of the moisture meter was made before the correction, and show an error which is expressed as the mean absolute amount of the moisture meter measurements in relation to the reference method of 0.190, after correcting the calibration error is reduced to 0.038. This paper describes the process of developing calibration. The technical criteria for the development of calibrations are a group of methods consisting of sample collecting, determination of the moisture content for the grain using the standard ISO 712:2009 method, measuring with the moisture meter, computer processing of the results using the programme for calibrations and our own method of monitoring the conditions of handling the samples used for verification of moisture meters.

Keywords: moisture meters, moisture content, calibration, standardisation

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Topic: ***Food technology and biotechnology***

Introduction

One of the primary activities of the State Office for Metrology is verification of legal measuring instruments and ensuring the public guarantee concerning the safety and adequate accuracy of measurements for the purpose of free trade and consumer protection. The work of the State Office laboratory which prepares samples for verification of moisture meters is primarily dedicated to the development, monitoring and improvement of the methods of sample making as the basic tool for the verification. However, the measurements on site show that there is a difference in the results between the individual moisture meters. The reason for this lays in the non-unified calibration constants of different origins and obsolete technical requirements (OG 44/94) which do not include the software into the examination of the measuring instruments in such a way as to carry out the standardisation of the calibration constants, which are then necessary to be implemented into the legislation as part of the technical requirements.
The analysis of the measuring instruments in the Republic of Croatia which are used in the commercial transactions of agricultural cultures has shown that the model of technical criteria for the development of calibrations must be adapted to the existing situation in such a way as to be easily applicable, sustainable, understandable for all users and able to keep pace with the development of new technologies.
Taking into consideration the diversity of agroclimatic conditions of the grain-producing countries, the models corresponding to the individual geographic areas are applied (GIPSA, 1999), which follow the general instructions of the umbrella metrological organisation. A few years of analysis of grain with different moisture content from the same geographical area are needed for the development of calibration constants for moisture meters (three or more, depending on the type of the agricultural culture). The data are evaluated and rough errors eliminated, after which follow testing and implementation of the approved calibrations (OIML R 59, 2009). The US Federal Grain Inspection Service approves the official calibrations for the measuring instrument GAC 2100 manufactured by Dickey-John with nine years of validity for certain cultures. In this way the occurrence of deviation of the result is reduced to the minimum, and the calibration procedure refers to the official measuring instrument (GIPSA, 1999). The advantages of this model are a large database for the measurement data and the simplicity of the procedure (everyone uses the same model of instrument). In spite of the uniformity of results, this model shows some defects in terms of the lack of flexibility towards the other measuring instruments manufacturers and some changes are therefore being prepared (Pierce, 2009). The verification model applicable for the use of moisture meters produced by various manufacturers is used in France where it is possible for the manufacturer of the measuring instrument to develop the calibration constants if it is verified by the competent institution (NLE, France), which is periodically controlled by the relevant regional office (DRIRE, France).

Since there are no manufacturers of moisture meters in Croatia and there are few models of measuring instruments with type approval in use, the calibration constants on the instruments are mostly factory settings or their modifications. At the occasion of measuring instrument verification, Agrionica d.o.o., company for moisture meter servicing has been using the unified calibration constants over the last three years (applied to 90 moisture meters manufactured by Dickey-John). The constants have been approved by the State Office and the analysis has shown that the repeatability of measuring is satisfactory, which has rendered possible the writing of guidelines referring to the technical criteria for the development of calibrations. Besides, a correction of calibration constants has been carried out in cooperation with Chopin Technologies, the moisture meter manufacturer from France, and Agrionica d.o.o. service, with the samples in the laboratory of the State Office on the Aqua TR meter, which is new on the Croatian market and technologically very advanced. The results obtained have confirmed the procedure and unified it within the technical criteria for the development of the calibration for moisture meters, which is shown in this work.

Materials and methods

Material

The used samples of barley have been gathered by the Inspecto d.o.o. inspection company at the time of harvest (2010) in the silos in Đakovo and Osijek, in accordance with the HRN ISO 950:1999 method.
No more than 24 hours should pass from the time of sample gathering to the time of processing and storing in the controlled conditions. During the transportation the samples must be kept from major temperature changes which would lead to the loss of moisture.

Working conditions in the laboratory

The manipulation of samples and the measurements are carried out in controlled laboratory conditions. The laboratory temperature: from 20 to 23 °C, maximum change of 1 °C/h, air humidity from 30 to 55 %. The measuring instruments for monitoring of the conditions (thermometer, hygrometer) are calibrated periodically (once a year).

Preparation of samples and storage

Admixtures and damaged grains are removed from the samples by means of mechanical sifting through adequate sieves. The sample is accepted if it is completely purified from admixtures and without dust, and if the grain is healthy, without odour and of a colour characteristic for its type. The mass of the sample is from 1000 to 1500 g. Purified and marked sample (year of harvest, origin, number of samples) is vacuumed-packed in plastic foil and stored in a refrigerator (storage temperature is from 5 to 8 °C) or set to laboratory conditions for 12 hours and then used. Vacuum-packing of the sample has proven to be the best way of keeping, compared with the storage in a plastic container with a lid or a jar with a ground stopper, because it reduces the contact of the sample with the air and prevents the development of microorganisms. In case of samples with low content of moisture, this manner of storage delays the date of expiry up to one year.

Humidity determination using the curing shed method

The moisture of the sample is determined by means of the ISO 712:2009 method for grain in accordance with all the requirements of the method. The analysis is carried out in controlled laboratory conditions (*Storage conditions and environment*). The analysed sample is taken from the vacuum bag, so that its part needed for moisture determination can be taken out (c. 30 g) and vacuum-packed again. The sample exposure to the air and to the temperature changes has to be reduced to the minimum. The samples are hermetically sealed all through the grinding procedure, until their weighing.

Measuring of moisture using moisture meters

The moisture meter used in this case was Aqua TR with the factory calibration constant for barley, manufactured by Chopin technologies from France. The moisture meter was turned on and acclimatized under the laboratory conditions (for 12 hours, in accordance with manufacturer's instructions), and the measuring of barley was carried out (Table 1). The results achieved are within the limits of the permissible error for the initial verification of the measuring instrument (OG 44/1996).

Table 1. Measuring moisture of barley using a moisture meter

Recorded Measurements

N° 1001 (C2)
MAIN 1.0I [35DB] [0674]
MES. 01.00[7] [F780]

Number of Meas.
11/ 1000

Rec.	Date / Time	Num.	Calibrations	H%	HLW(kg/hl)
0001	2010/08/25 08:4...	007	JECAM	11.50	68.00
0002	2010/08/25 08:5...	007	JECAM	15.38	66.97
0003	2010/08/25 08:5...	007	JECAM	13.60	57.03
0004	2010/08/25 08:5...	007	JECAM	11.75	68.34
0005	2010/08/25 08:5...	007	JECAM	15.19	62.02
0006	2010/08/25 08:5...	007	JECAM	10.23	66.11
0007	2010/08/25 09:0...	007	JECAM	14.40	68.69
0008	2010/08/25 09:0...	007	JECAM	9.58	63.83
0009	2010/08/25 09:0...	007	JECAM	12.70	67.21
0010	2010/08/25 09:0...	007	JECAM	16.27	67.57

Erase Export Quit

Results and discussion

After the measuring with the moisture meter using the existing calibrations (linear), the value measured using the curing shed method is added to the results in the table of the program for the calculation of calibration delivered by the manufacturer of the measuring instrument (Table 2). The correction of the calibration coefficient is carried out based on the calculation from the resulting parameters and the calibration curve (Fig. 1).

Table 2. Calculation (Cal) of coefficients for calibration constant

Cal			
H% Linear	**H% Reference**	**H% Calculated**	**Difference**
11.5	11.99	-1.7	13.7
15.38	14.93	2.8	12.1
13.6	13.6	0.8	12.8
11.75	12.16	-1.4	13.6
15.19	14.72	2.6	12.1
10.23	10.86	-3.4	14.3
14.4	14.06	1.7	12.3
9.58	10.41	-4.3	14.7
12.7	12.93	-0.2	13.2
16.27	15.87	3.7	12.2
10.17	11.34	-3.5	14.8
Average	12.99	-0.27	13.26
Minimum	10.41	-4.29	12.14
Maximum	15.87	3.71	14.82
Nb value	11		

Calibration AQUA-TR / AGRI-TR

	a0	**a1**	**a2**	**a3**
Product :	-21	2	-4	3

$H=a0+a1*lin+a2/100*(lin)^2+a3/10000*(lin)^3$

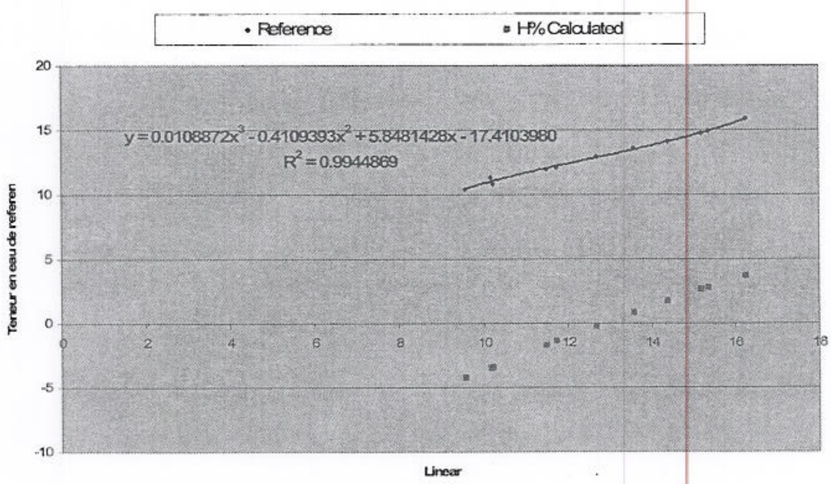

Fig. 1. Calibration curve

The average difference of the deviation value for barley between the moisture measured by means of a curing shed and measured using the moisture meter is 0,19 (Table 3). The value for correction is entered into the settings of the moisture meter, after which it is sent to the manufacturer of the measuring instrument who includes the new value of the calibration constant into the calibration base. The measuring shows the reduced result error.

Table 3. Moisture (H%) measured by use of moisture meter and reference value before and after the correction

1ST MEASUREMENT			2ND MEASUREMENT		
H% Linear 1	**H% Reference**	**Difference1**	**H% Linear2**	**H% Reference**	**Difference2**
11.5	11.99	-0.45	11,83	11.99	0,16
15.38	14.93	0	15,44	14.93	-0,51
13.6	13.6	0.41	13,83	13.6	-0,23
11.75	12.16	-0.47	12,24	12.16	-0,08
15.19	14.72	0.63	15,19	14.72	-0,47
10.23	10.86	-0.34	10,59	10.86	-0,57
14.4	14.06	0.83	14,63	14.06	0,47
9.58	10.41	0.23	9,94	10.41	-0,02
12.7	12.93	-0.4	12,95	12.93	-0,44
16.27	15.87	1.17	16,31	15.87	1
		0.1909090			-0,0381818

After the correction, the calibration constant is entered into the measuring instrument (Fig. 2) and the measuring is repeated with the same samples. The calibration constant is marked by its name and number, and stored in the memory of the measuring instrument.

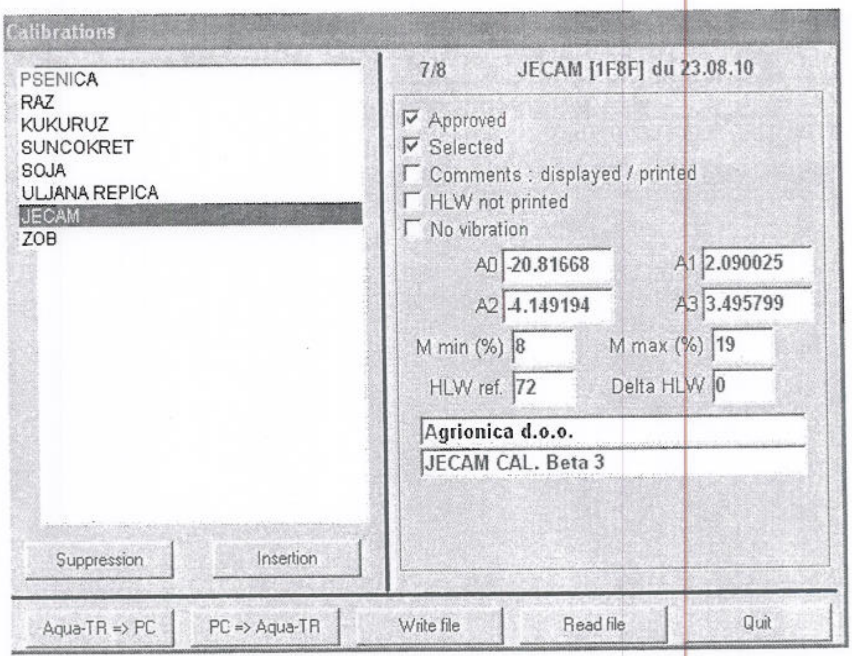

Fig. 2. Calibration constant on the moisture meter

Conclusions

The measuring has been carried out on eleven samples of barley with the moisture range from 10.41 % to 15.87 %, which represents the minimum necessary for the correction of the calibration constant, despite the fact that there is an obvious reduction of the error, which is the objective of this work. The procedure is also completely applicable in case of the measuring instruments by other manufacturers of moisture meters used for measuring moisture in grains and oilseeds in Croatia. The experience has shown that the manufacturers cooperate on the development of the calibration constants because the reliability of their measuring instruments is in their best interest, and the reduction of the error means the most to the users of the measuring instruments because it guarantees fair trade. Furthermore, it is obvious from this example that the possible correction of the existing calibration is successful. The described procedure of sample preparation and measurement for the complete development of the new constant, when necessary, is the same with the greater number of samples (it is defined for each agricultural culture separately) taken from at least three harvests for the same culture (USDA, 1999). According to the information by the State Office, the standardisation of the existing calibrations in the Republic of Croatia is implementable to the majority of meters in use in the period of two years, in the sequence determined by the criteria of representation in the agricultural production. The technical criteria for the development of calibration constants and their implementation should be included into the Ordinance on metrological requirements for moisture meters, as well as the definition of the correct calibration and the conditions under which it is corrected or completely replaced with a new one.

Acknowledgments

Measurements using the moisture meter of Aqua TR model manufactured by Chopin technologies and the program support have been enabled by the company Agrionica d.o.o. from Požega.

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