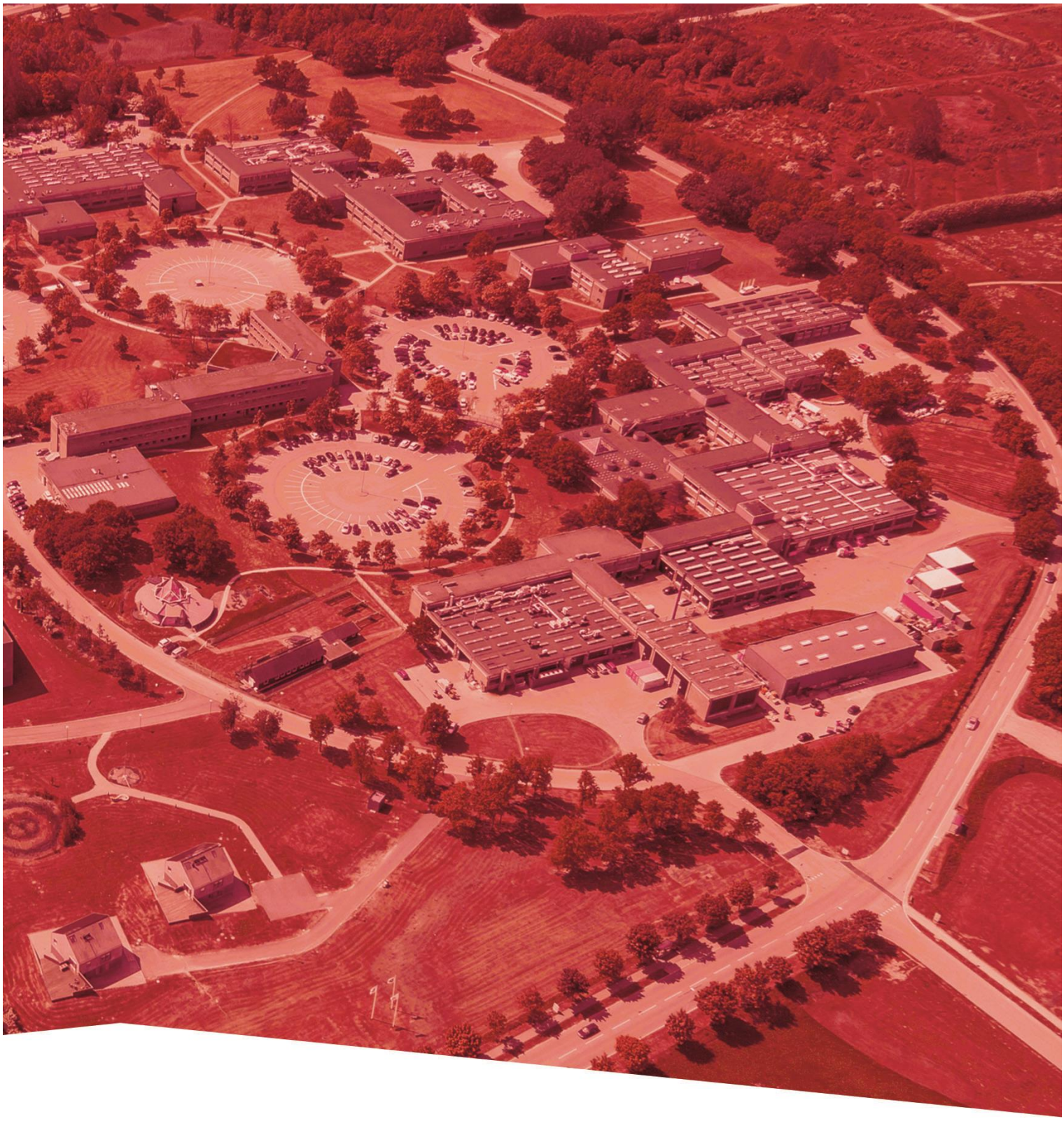

Calibration of Setup for Online Measurement of Moisture Content

Kalibrering af Online Fugtmåleudstyr

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Kalibrering af Online Fugtmåleudstyr

Udarbejdet af
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1. Introduction

The present report describes a moisture content calibration test for the microwave (MW) system at VERDO's Combined Heat and Power (CHP) plant in Randers, Denmark. The test was conducted in October 2022 in a collaboration between VERDO and Danish Technological Institute (DTI). In addition, CETIAT was present at the facility and conducted related work, and CMI and IMBiH received fractions of the sample material employed.

Woodchips are an important fuel for CPH plants. The water content in woodchips is significant, typically constituting 10 – 60 %, and has a substantial impact on economy and boiler control for CPH plants. However, woodchips constitute a heterogenous material, and therefore the measurement of the water content gives rise to many challenges. Traditional Loss-on-Drying (LoD) measurements are time consuming and in addition usually give rise to sampling issues. Online measurements have significant advantages over traditional manual measurements based on LoD. Firstly, the measurements are conducted automatically. The measurements are very rapid, and consequently the results can be employed as input for control of boilers. Also, it is possible to conduct a large number of measurements and in this way measure on a large fraction of the biofuel. One effect of this is, that the effort required for accurate sampling is significantly reduced.

The main issue with online measurements is related to the quality control of the measurements, in the form of tests, calibration, and adjustment. The goal of the present work is to develop and demonstrate a procedure for calibration of an online system for water-content measurements on solid biofuels (woodchips). In brief, the work consisted of three parts.

1. Preparation of woodchip test material
2. Test measurements with VERDO's MW system
3. Reference measurements at DTI.

The work resulted in a calibration curve for VERDO's MW system for the range of 10 – 60 % water content. The uncertainty of the calibration result was about 3.1 % absolute ($k = 2$). The best results were obtained by using both attenuation and phase data from the MW system.

2. Principle for setting up the measurements

The basic scheme employed to test the water content results of the MW system is to compare measurements on the same sample with measurements conducted using a reference method. While in the principle being straight forward, there are several details which must be considered carefully. The actual MW measurement system is described in the next section.

In the present case, the method employed for the reference measurements is the DTI's reference method for water mass fraction. This method is based on the principle of Evolved Water Vapour principle and detects the amount of water by measuring the dew point and the flow (EWV-DP). To test the stability of the sample material the Loss on Drying (LoD) method is employed. The measurements were conducted according to ISO/EN 18134-2. The LoD method measures the weight loss resulting from drying a sample of the woodchips in an oven at 105 °C until stable weight. This is defined as the moisture content. However, in addition to water a small amount of VOC may also evaporate during the process, and consequently the moisture content may be slightly different that the water content. In other words, the LoD method cannot measure the water content SI traceable.

A critical part of the setup was the preparation of test samples of woodchips. The sample material chosen was a mixture of *Picea Abies* (Norway Spruce) and *Picea Sitchensis* (Sitka Spruce). The trunks had the bark removed and was subsequently chopped into relatively small woodchips. See Figure 1. The fine chopping was a significant advantage in relation to the handling and measurement process because it was easier to mix and pack homogenously.

It is a strict requirement that the sample material spans the entire measurement range. Therefore, the goal was to prepare 5 different levels of moisture content ranging from 10 % to 60 %. Duplicates were produced of each moisture level and in addition two samples of the original (i.e. unprepared) woodchips were included. Thus, in total 12 samples

Figure 1. The woodchip test material.

Figure 2. The prepared test samples. The photo shows 10 of the 12 samples.

were prepared. It was estimated that about 30 L was required for each measurement at Verdo, and the sample-size goal was therefore 40 L.

The different levels of moisture content of the test samples were produced by pre-drying and re-moisturizing fractions of woodchips. This process was adopted for two reasons, firstly to reach the required moisture levels, and secondly to secure homogenous samples. The process was as follows. From a batch of about 1 m^3 woodchips fractions of 40 L were extracted. Each sample fraction was spread out over a large surface while being dried in an oven at 40 °C for approximately 24 hours. Then the resulting moisture level was measured using LoD, and the amount of water required to reach the desired water-content level was estimated from the LoD result and the sample weight. The fractions were subsequently stored in sealed boxes until about 10 days prior to the VERDO measurements. At that point the additional water was added while mixing the sample. The test samples produced are shown in Figure 2.

3. The MW system

The principle of the MW system at VERDO's CPH plant in Randers, Denmark, is shown in Figure 3. In addition to the MW transmitter and receiver it also includes a subsystem for radioactive load

Figure 3. Left: A principal sketch of the tested MW measurement system, including firstly a radioactive load measurement and secondly the microwave transmitter (bottom) and receiver (top) (from https://www.berthold.com/). Right: A photo of the actual setup at VERDO.

measurements. To determine the moisture content, M, from the measured parameters $load$, $attenuation$, and $phaseshift$, a linear regression using based on the following formular is employed:

$$M = a \cdot \frac{phaseshift}{load} + b \cdot \frac{attenuation}{load} + c. \quad (1)$$

During normal operation the measured data are continuously feed into the control system of the CPH plant.

4. Measurements with online setup at site

The basic idea behind the tests described in this report was to place the prepared test sample material on the conveyor belt, perform a moisture measurement using the MW system, and immediately thereafter collect a fraction of the material for reference measurements. Subsequently, the reference measurements were conducted at DTI.

The first step of measurement process is illustrated in Figure 4 and Figure 5. The conveyor belt was stopped during the entire testing process, which lasted about 2½ hour. To avoid unnecessary interruption of the CPH plant, the measurements were scheduled to coincide with planned maintenance work.

Figure 4. Sample material place at the conveyor belt at VERDO's CPH plant for the test measurements using the MW system. The sample material was prepared in advance at DTI, as described in the text.

For each sample, the following procedure was adopted.

1. The belt was cleaned.
2. Two support blocks were place on the belt, and the space between the blocks was filled with woodchip test material. See Figure 4.
3. The woodchip layer was carefully levelled, as illustrated in Figure 5.
4. A measurement using the MW system was performed.
5. Woodchip sample material was collected for subsequent reference materials at DTI. The material was mainly collected from the central area of the MW system.

The total handling and measurement time was about 10 minutes per sample.

The repeatability of these MW measurements contributes significantly to the total calibration uncertainty. There are three main sources of variability. The first is the inherent inhomogeneity of the sample material, resulting from moisture and other material-properties variations. The second origins from the irregular packing of the sample material during the measurement process. The third is the MW instrument itself. Several measures were taken to improve the repeatability. Mixing and other actions were conducted to achieve homogenous moisture content. In addition, special care was taken to produce a homogenous layer of sample material on the belt.

Figure 5. The woodchip test material is carefully level, to secure a constant layer thickness.

The uncertainty contribution from repeatability on the same batch of sample material was measured 4 times in a row. This test indicated relative magnitude of the standard deviation of about 38 % for the phase shift, 15 % for the attenuation and 7 % for the load, respectively. The last number agrees roughly with expectations – the latter being based on the

uncertainty of sample-material layer thickness, which is estimated to be better than 1 cm out of a total layer thickness of 10 cm. The other numbers are larger than expected for unknown reasons.
The actual quantification of this uncertainty contribution was performed as part of the regression process, as discussed below.

5. Laboratory reference measurements

The reference measurements of the water content were of course a central part of the calibration procedure. The measurements were performed using the reference method developed by DTI during the ERMP project METefnet (Bell et al., 2015). The method has been described by Østergaard and Nielsen (2018) and is based on the evolved water vapor principle. In short, the samples are dried in an oven using a flow of N_2 gas, and the total amount of water is determined from the dew point and the flow of the gas. Photos of the setup are shown in Figure 6.

The reference measurements were challenged by the effect of biological activities during the storage of the samples before the reference measurements. The reference setup typically only allowed for measurement of one sample per day. The sample material was collected after the MW measurements at VERDO's CPH plant and stored in sealed aluminium bags until the reference measurements could be performed. These bags were diffusion tight, with the goal to avoid exchange of humidity between the atmosphere and sample. On the other hands, the bags do not prevent biological degradation of the woodchips. To quantify the effect of the biological activity on the water content LoD measurements were performed on fractions of the collected samples before and after the reference measurement campaign, and the standard deviation of the difference between the before/after LoD results was calculated. The LoD measurements were conducted according to ISO/EN 18134-2. In brief, the samples were dried in an oven at 105 °C ± 2 °C until stable weight, i.e. for about 20 hours.

The uncertainty of the reference measurements has three main contributions.

Figure 6. The setup for reference water-content measurement at DTI. Left: The heated sample chamber; Right: A sample being placed in the chamber.

1. Homogeneity, i.e. variation in water content within a single sample. To quantify this, two fractions of each sample bag were taken right after the MW measurements at VERDO. LoD was then used to measure the moisture content, and the uncertainty contribution was determined using the standard deviation of the differences between each pair of results. Magnitude: 0.26 % absolute ($k = 1$).
2. Stability. The uncertainty contribution from stability was discussed above. To quantify this contribution, two fractions of each sample bag taken right after the MW measurements at VERDO were compared with fractions taken after all the reference measurements had been performed. LoD was then used to measure the moisture content, and the uncertainty contribution was determined using the standard deviation of the differences between each set of before/after results. Magnitude: 0.94 % absolute ($k = 1$).
3. Reference measurement. This contribution is due to uncertainties related to measurement of flow, pressure, dewpoint, and temperatures in the reference setup (see Østergaard & Nielsen, 2018), and a typical value is 1 % of the water content ($k = 1$). The uncertainty of the water mass fraction depended on the water content, and as an example about 0.5 % absolute would result for a woodchip sample with 50 % water content.

It can be observed that the dominating contribution to the reference measurements originates from the sample-stability contribution. The total expanded uncertainty ($k = 2$) is about 2.1 % absolute on average.

6. Results

The results of the MW measurements at the CPH plant and the reference measurements at DTI are presented in Table 1. The data analysis is described in the sections below.

Table 1. Data for moisture and other measured parameters from test of the VERDO MW system. "blank" refers to measurement of empty conveyor belt (no sample) and "Verdo" to a test sample of "normal" woodchips recorded several days after the test measurements. The parameters measured by the MW system are Phase (Phase shift), Att. (Attenuation) and Load (Load signal). Ref. is the reference moisture measurements. The contributions to the standard uncertainty are "u ref" (measurement uncertainty of the reference method), "u hom" (homogeneity), "u stab" (sample-material stability), and their combined value, "u (k=1)". The total expanded uncertainty is "U (k=2)".

#	Box	Phase	Att.	Load	Ref	u ref	u hom	u stab	u (k=1)	U (k=2)
1	10	-86.13	2.21	10.36	11.26%	0.14%	0.26%	0.94%	1.0%	2.0%
2	9	-90.28	1.9	9.98	9.53%	0.13%	0.26%	0.94%	1.0%	2.0%
3	8	-73.31	5.15	10.41	19.40%	0.24%	0.26%	0.94%	1.0%	2.0%
4	7	-71.06	4.93	10.19	24.09%	0.28%	0.26%	0.94%	1.0%	2.0%
6	5	-54.61	9.4	10.71	28.80%	0.42%	0.26%	0.94%	1.1%	2.1%
7	4	76.1	7.78	11.94	35.85%	0.39%	0.26%	0.94%	1.1%	2.1%
8	3	71.37	7.38	10.74	34.09%	0.39%	0.26%	0.94%	1.0%	2.1%
9	2	119	15.52	11.62	43.94%	0.48%	0.26%	0.94%	1.1%	2.2%
10	1	9.87	15.38	11.37	48.53%	0.52%	0.26%	0.94%	1.1%	2.2%
11	blank	1.29	0.19	7.88						
12	11	145.86	17.66	11.4	56.10%	0.57%	0.26%	0.94%	1.1%	2.3%
13	12	22.95	20.4	11.51	56.97%	0.61%	0.26%	0.94%	1.1%	2.3%
14	12	37.68	21.87	12.16						
15	12	30.49	23.61	11.63						
16	12	14.43	16.51	11.81						
17	Verdo	-43.16	6.48	9.35	39.74%	0.55%	0.26%	0.94%	1.1%	2.2%

7. Analysis

7.1. Load

The load signal is the measure of the area density of the load and can be measured in kg/m^2. This signal is essential, because both signals of the MW system (attenuation and phase shift) will be proportional to the load. Consequently, these signals need to be load corrected before they can be used to determine the moisture content, as indicated in equation (1). On the other hand, a calibration of the load signal is

not required for measuring the water content of the woodchips, and therefore this section can be considered as a kind of a test.

In the present case the load consists of dry matter (i.e. wood) and water. The height of the test samples was kept constant during the measurements and furthermore the volume is basically not influence by water content. If the density of dry wood is designed ρ_{dry}, the volume V and the area A, the load can be described by

$$Load = \frac{m}{A} = \frac{m_{dry} + m_{water}}{A}. \quad (2)$$

Since the water content, MC, is given by

$$MC = \frac{m_{water}}{m_{dry} + m_{water}} \Rightarrow m_{water} = m_{dry} \cdot \frac{MC}{MC - 1} \Rightarrow m_{dry} + m_{water} = m_{dry} \cdot \left(1 + \frac{MC}{MC - 1}\right) = \frac{m_{dry}}{1 - MC}, \quad (3)$$

the load variation can be calculated by

$$Load = \frac{V \cdot \rho_{dry}}{A} \cdot \frac{1}{1 - MC} \propto \frac{1}{1 - MC}. \quad (4)$$

Therefore, plotting $1/(1 - MC)$ versus the load signal should result in a straight line through $(0,0)$. This is tested in Figure 7, and indeed the expected relationship is observed. However, the relative magnitude of the standard deviation of the regression is 17 %, which is larger than expected (see discussion of repeatability above). At present, it is not evident whether this uncertainty is due to the calibration process or to the measurements system at VERDO. The accuracy of the calibration may possibly be improved by using a wider range of loads for the test samples, e.g. 5, 10, 15, and 20 cm load.

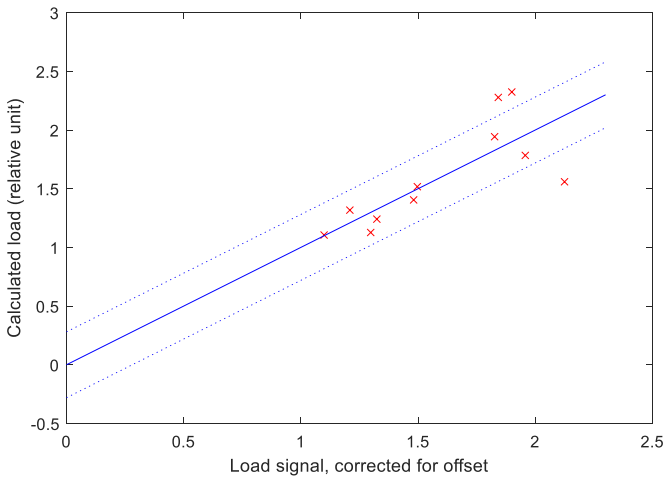

Figure 7. Relative load calculated by equation (4) vs. load signal (corrected for offset). Measured points: red crosses; Regression and standard uncertainty: solid and dotted blue lines, respectively. See text.

An offset of 7.88 was subtracted from the load signal. This offset was determined by measuring the load signal with an empty conveyor belt. It should be remarked that this offset is time-dependent, because of the decay of the radioactive source in the load sensor. The control box of the MW

system should in the principle automatically adjusts for this decay, however for some reason the adjustment appears to not be working properly. For that reason, the load-calibration offset is not correct for historic data. It is strongly recommended to address this problem, either by proper software correction or, alternatively, the signal from the belt weigher could be employed, thus negating the requirement for the radioactive load sensor.

7.2. Moisture content

As previously mentioned, the MW system detects the phase shift and attenuation of microwaves passing through the sample material on the conveyor belt. Assuming that these two signals can be considered as the sum of the signals of the dry matter (i.e. of dry wood) and the water, the water content can be determined by equation (1), i.e.

$$M = a \cdot \frac{phaseshift}{load} + b \cdot \frac{attenuation}{load} + c. \tag{5}$$

In the following three versions of this relationship will be tested.

1. Using only phase shift, i.e. setting $b = 0$
2. Using only attenuation, i.e. setting $a = 0$
3. Using both phase shift and attenuation

The results of the three different analyses, based on measurements of the phase shift, attenuation, and both. The standard deviation is a measure of the repeatability of the MW measurements and is given in percent absolute.

Method	a	b	c	σ (st.dev.)	$u(rep)$	Plot
Phase	0.46289		34.795	10 %	3.1 %	Figure 9
Attenuation		9.7228	4.3563	6.1 %	1.8 %	Figure 8
Phase and attenuation	0.19937	7.2763	12.246	3.6 %	1.1 %	Figure 10

The results of these three analyses are summarized in the table above. It is evident that for the investigated test samples the best result is obtained by using both the phase and the attenuation measurements of the MW system.

The standard deviation, σ, is calculated by from the residuals of the regression. The repeatability contribution to the calibration uncertainty is estimated by

$$u(rep) = \frac{\sigma}{\sqrt{n}}, \tag{6}$$

with $n = 11$ being the number of calibration measurements. The standard calibration uncertainty is obtained by combining the uncertainty contributions from reference measurements, sample stability and homogeneity and repeatability. The average uncertainty of the reference has been employed, and therefor the uncertainty will be slightly large for high water content and slightly smaller for low water content.

Method	$u(cal)$, standard calibrations uncertainty ($k = 1$)	$U(cal)$, expanded calibrations uncertainty ($k = 2$)
Phase	3.3 %	6.6 %
Attenuation	2.1 %	4.2 %
Phase and attenuation	1.5 %	3.1 %

7.3. Measurement uncertainty in normal operation

In normal operation there will be an extra contribution to the measurements uncertainty resulting from different material properties of different types of woodchips. This may result from variation in size, species, or impurities. To quantify the magnitude of this extra contribution test measurements must be made during normal operation, comparing MW measurements with reference measurements. Such measurements are already being performed, but due to the issue with drift of the load measurements resulting from radioactive decay they cannot provide the basis of a reliable comparison. Three test samples obtained in normal operation shown in Figure 10 appear to show a satisfactory level of agreement.

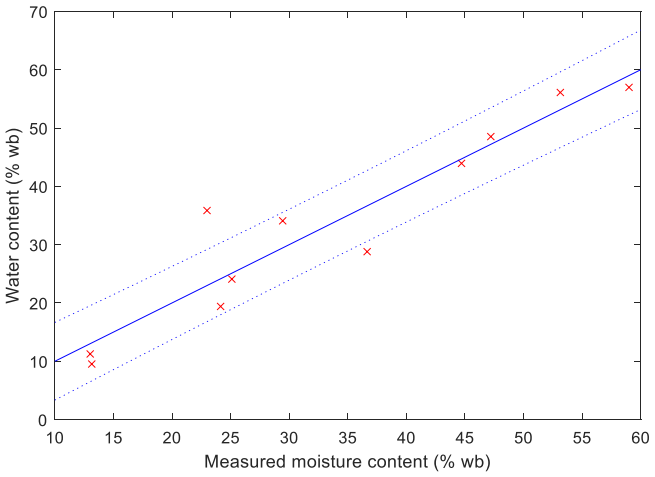

Figure 8. The reference moisture content vs. MW-measured moisture content based on the load-corrected phase signal. Measured points: red crosses; Regression and standard uncertainty: solid and dotted blue lines, respectively.

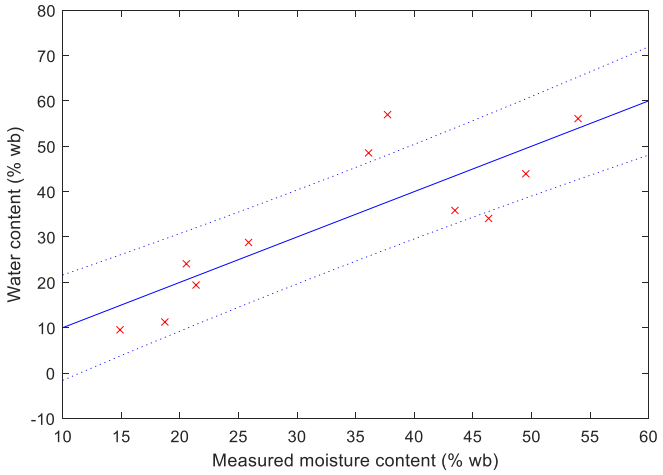

Figure 9. The reference moisture content vs. MW-measured moisture content based on the load-corrected phase signal. Measured points: red crosses; Regression and standard uncertainty: solid and dotted blue lines, respectively.

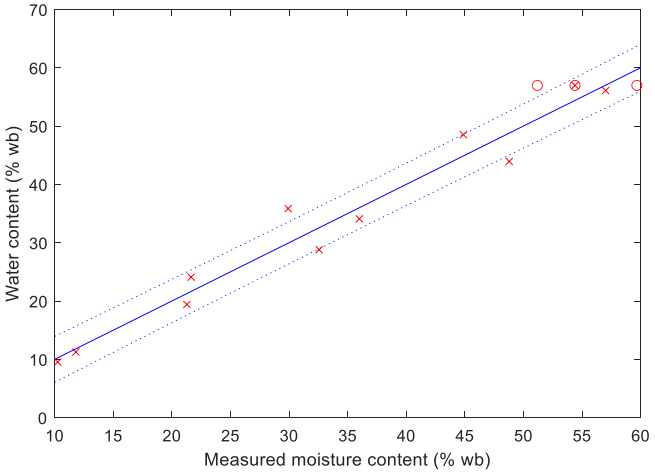

Figure 10. The reference moisture content vs. MW-measured moisture content based on both phase and attenuation signals (both load-corrected). Measured points: red crosses; Regression and standard uncertainty: solid and dotted blue lines, respectively; Test points: red circles.

8. Conclusion and outlook

In conclusion, the MW system used at VERDO's CPH plant in Randers, Denmark has been calibrated for water fractions in the range of 10 – 60 %. To perform the calibration, a batch of 10 particularly prepared woodchip samples with different water-fraction levels were prepared. The reference method for water content at Danish Technological Institute (DTI) was employed to provide traceable results. The best calibration result was obtained using both phase-shift and attenuation measurements in combinations, and the resulting calibration uncertainty was about 3.1 % absolute ($k = 2$, corresponding to a 95 % confidence interval).

As result of the present work the following suggestions / recommendations are made.

1. Load measurements (see appendix). Due to the problems mentioned with the radioactive load sensor significant drift of the load – and thus the water-content measurement – using the MW system occurs. There are two possible solutions.
    a. Software: The half-life constant of the radioactive ^{137}Cs source is well-known, and consequently it is possible to correct for that effect – either using the control box of the MW system or other software.
    b. Belt weight: Instead of using the radioactive sensor the belt weight (in combination with belt speed) can likely provide a much more accurate load measurement.
2. Reference-sample stability. To improve the stability of the reference samples the biological activity should be reduced. This could be obtained by storing the samples at low temperature, either in a refrigerator (probably sufficient) or in a freezer.
3. If a calibration is not required – and an adjustment is considered sufficient – reference measurements using the LoD method are faster and less cumbersome the DTI reference method but will still yield similar results.
4. A smaller but higher frame to contain the sample material may lead to better repeatability and thus smaller uncertainty.

9. Support

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EMPIR EURAMET
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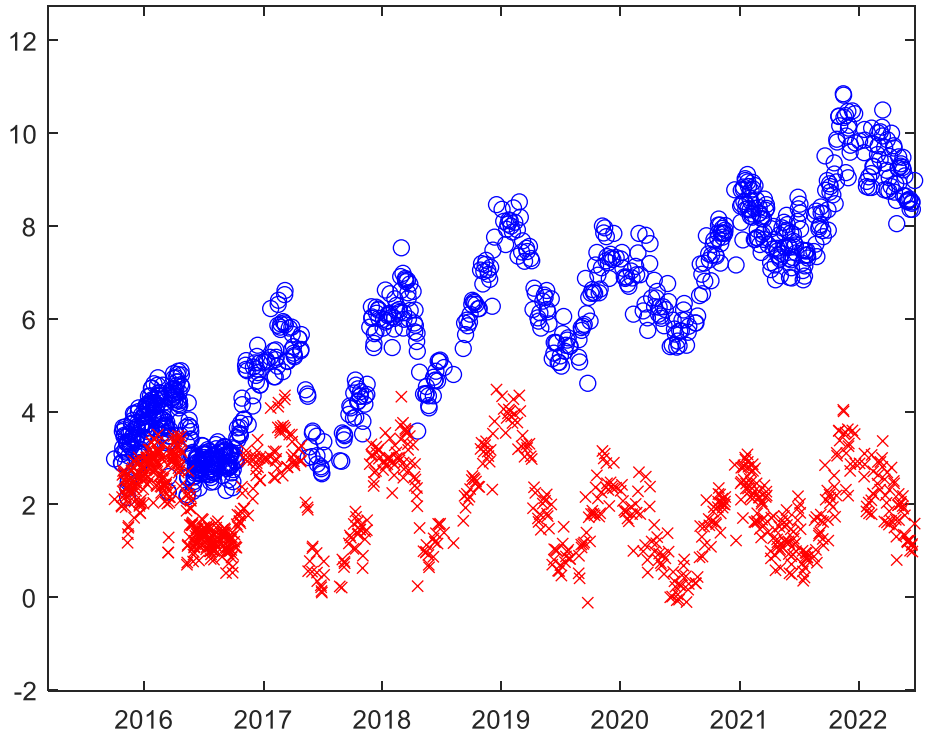
11. Appendix

Load signal (in g/cm2) measured since 2016. The blue curve is the results supplied by VERDO, the red crossed are corrected for a decay of about -1 g/cm2 per year.



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