Uncertainty of Moisture Measurements Methods for Grains

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Abstract: It is important for food scientists to be able to reliably measure moisture content as it is one of the most commonly measured properties of food materials especially grains. In addition, the moisture monitoring techniques will provide for better control of processing for value added applications and improved end product quality. They will provide more precise moisture information that can be used in optimizing grain drying with resulting higher quality products and significant energy savings in grain drying operations. A number of techniques and electronic meters have been developed for this purpose, which vary in their accuracy, cost, speed, sensitivity, specificity, ease of operation, etc...The uncertainty in the moisture content measured by drying method, which is the highest standard method in grain moisture measurements, was evaluated. The uncertainties for other electronics meters to be used as secondary standard for determination of moisture in grain, which are very fast and easy to be used, were calculated and its traceability to Inter-National System of units(SI) was established.

Key words: Electronic moisture meters uncertainty, Calibration of moisture meters, Moisture measurements

INTRODUCTION

Foods are heterogeneous materials that contain different proportions of chemically bound, physically bound, capillary trapped or bulk water. In addition, foods may contain water that is present in different physical states: gas; liquid or solid which can be problematic for the food analyst trying to accurately determine the moisture content of foods.

Moisture is generally refers to the presence of water, often in trace amount. It is important to food scientists for a number of different reasons. First, it is the single most important quality characteristic that determines the safe storage potential for cereal grains and oilseeds (Wikipedia, 2003; Grain Moisture, 1995). It is also important for harvesting, storage transport, shelf lives, package for storage, energy savings in grain drying and processing. There are legalities to the maximum or minimum amount of water that must be present in (FAQS, 1969) certain type of food. The coast of many foods depends on the amount of water they contain as the manufacturers often try to incorporate water as much as possible in a food without exceeding some maximum legal requirements because water is an inexpensive ingredient.

There are many techniques used for the determination of moisture in grains the most important techniques used are discussed below (NIST, 2006; Moisture, 2007; German, 2006; Develops, 2008; On-Line 2003; Adam Moisture Balances, 2003).

1-evaporation Method (Oven Dry Method or the Standard Method):

Its principle relies on measuring the mass of water in known mass of sample. The moisture content is determined by measuring the mass of a grains before and after the water is removed by evaporation. The basic principle of this technique is that water has a lower boiling point than the other major components within grains, e.g., proteins, carbohydrates and minerals.

To obtain an accurate measurement of the moisture content of a grain using evaporation methods it is necessary to remove all of the water molecules that were originally present, without changing the mass of the grain matrix. This is often extremely difficult to achieve in practice because the high temperatures or long times required to remove all of the water molecules would lead to changes in the mass of the grain matrix, e.g., due to volatilization or chemical changes of some components. For this reason, the drying conditions used in evaporation methods are usually standardized in terms of temperature and time so as to obtain results that are as accurate and reproducible as possible. Using standard method of sample preparation and analysis helps
to minimize sample-to-sample variations within and between laboratories.

**Evaporation Devices:**

Thermal energy used to evaporate the water from a grain sample can be provided directly (e.g., transfer of heat from an oven to grain) or indirectly (e.g., conversion of electromagnetic radiation incident upon a grain into heat due to absorption of energy by the water molecules using microwave oven or infrared lamp). The three techniques used in this study are:

**Vacuum oven:**

Weighed samples are placed under reduced pressure (typically 25-100 mm Hg) in vacuum oven for a specified time and temperatures until constant weight and their dried mass is determined. The thermal energy used to evaporate the water is supplied directly to the sample via the metallic shelf that it sits upon. There is an air inlet and outlet to carry moisture lost from the sample out of the vacuum oven, which prevent the accumulation of moisture within the oven. The boiling point of water is reduced when it is placed in vacuum. Drying grains in vacuum oven therefore has a number of advantages over drying techniques. If the sample is heated at the same temperature, drying can be carried much quicker. Alternatively, lower temperature can be used to remove the moisture (e.g., 70°C instead of 100°C), and so problems associated with degradation of heat labile substances can be reduced.


Moisture content was determined by this method with accuracies of better than one half of one percent. It offers good potential for saving considerable time and labor in moisture testing required when drying grains and testing them for safe storing and marketing. Weighed samples are placed in a microwave oven for a specified time and power-level and their dried mass is weighed. Alternatively, weighed samples may be dried until they reached constant final mass. The water molecules in the food evaporate because they absorb microwave energy, which causes them to become thermally excited. The major advantage of microwave methods over other drying methods is that they are simple to use and rapid to carry out.

**Infrared lamp drying (Determination of moisture content, 2007; Development, 2006):**

The sample to be analyzed is placed under an infrared lamp and its mass is recorded as function of time. The water molecules in the food evaporate because they absorb infrared energy, which causes them to become thermally excited. One of the major advantages of Infrared lamp drying methods is that moisture contents can be determined rapidly using inexpensive equipments, e.g., 10-25 minutes. This is because the IR energy penetrates into the sample, rather than having to be conducted and converted inwards from the surface of the sample.

**The Following Practical Considerations Were Taken into Account in Our Study:**

The first is sample dimension as the rate and extent of moisture removal depends on the size and shape of the sample. The second is the decomposition of other grain component because if the temperature of drying is too high, or the drying is carried out for too long, there may be decomposition of some of the heat-sensitive components in the grains. This will cause a change in the mass of the sample matrix and lead to error in the moisture content determination. It is therefore normally necessary to use a compromise time and temperature, which are sufficient to remove most of the moisture, but not too long to cause significant thermal decomposition of the grain matrix. Besides most evaporation methods stipulate a definite temperature or power level to dry the sample so as to standardize the procedure and obtain reproducible results. Finally it is important to use appropriate pans to contain samples, and to handle them correctly, when carrying out a moisture content analysis. Typically aluminum pans are used because they are relatively cheap and have a high thermal conductivity. Pans were handled with tongs because fingerprints can contribute to the mass of a sample. Also pans were dried in an oven and stored in desiccator prior to use to ensure that no residual moisture is attached to them.

**Advantages and Disadvantages of Evaporation Method:**

These methods are Precise, relatively cheap, easy to use, and officially sanctioned for many applications. Many samples can be analyzed simultaneously. But they are destructive and time consuming.
Uncertainty of Oven Dry Method (Standard Method):
The Two Ways to Evaluate Individual Uncertainty Contributions Are:

1- Type (A) (UA) uncertainty:

After ~ ten measurement results the mean was obtained from the relation:

\[ q = \frac{\sum_{i=1}^{n} q_i}{n} \]  

(1)

Where

- \( q \) is the mean
- \( n \) is the number of readings =10
- \( q_i \) is the i value and
- \( i \) is the number of the reading

Then the variance is equal to

\[ S_q^2 = \frac{1}{n-1} \sum_{i=1}^{n} (q_i - q)^2 \]  

(2)

The standard deviation (\( S_q \)) (Type A of uncertainty) is equal to

\[ \text{UA} \text{ or } \bigcup_{\text{REP}} = S_q = \sqrt{S_q^2} \]  

(3)

2-Calculation of type (B) (UB) uncertainty:

Type (B) uncertainty was calculating from the following table.

Table 1: Calculation of type (B) uncertainty UB.

<table>
<thead>
<tr>
<th>Symbol</th>
<th>Source</th>
</tr>
</thead>
<tbody>
<tr>
<td>UT</td>
<td>Uncertainty caused by the temperature distribution in the dryer oven.</td>
</tr>
<tr>
<td>Um0</td>
<td>Uncertainty of the combined mass of the grain sample and the weighing can before drying.</td>
</tr>
<tr>
<td>Um1</td>
<td>Uncertainty of the combined mass of the grain sample and the weighing can after drying.</td>
</tr>
<tr>
<td>Us</td>
<td>Uncertainty in the calibration of the weighing balance.</td>
</tr>
</tbody>
</table>

Then the total uncertainty

\[ U_{\text{tot}} = \sqrt{UB^2 + UA^2} \]


According to the previous disadvantages of the evaporation method physical methods are used. A number of physical methods have been developed to determine the moisture content of grains that are based on the fact that water has appreciably different bulk physical characteristics than the grain matrix, e.g.; density, electrical conductivity or refractive index. One of these methods is using the electronic meters. These meters are calibrated to give the moisture content values directly. They are done calibrated using samples which were dried using the oven standard method. The data of uncertainty of the dry samples were used in the determination of moisture content and uncertainty budget of the electronic meters. Then the total uncertainty of the meter is calculated.

Uncertainty in Calibrations of Electronic Meter (Working Standard):

Many national standards laboratories have chosen to maintain a secondary standard as their primary reference for moisture measurements. The traceability to a primary standard is realized by calibrating the secondary standard(s) with the national standard of the laboratory.

Experimental Procedure:

The procedure used to evaluate the uncertainty of measurement of electronic meters used in our laboratory is as follows:

Samples of grains were prepared with moisture content covering the range of moisture meter scale. The preparation were discribed in a privious paper (N.I.El-Sayed, Mostafa M. Makawy, 2010). Four types of grains were used which are; Adas, Rice, Lobya and Beans; Each sample was divided into five portions. Three were used for the determination of moisture content using oven dry method, microwave oven; and the third was used with the IR appratous. The two other samples were used for moisture measurement by two electronic meters with resolution of 0.1 and 0.2 respectivly. A comparison was done between the results of each sample using
the five measuring methods. This process was repeated for four kinds of grains and the results are shown in table (3).

**Sources of Uncertainty:**

The uncertainty of a calibration is contributed by the uncertainty of the reference value (i.e. uncertainty sources related to the measurement standard and calibration equipment) and uncertainty sources related to the electronic meters.

Uncertainty of moisture meter considered here includes:
- Repeatability type (A) uncertainty
- Resolution
- Non-linearity/interpolation error is taken into account if calibration curve or function is reported (usually taken from the manufacturer catalogue).
- Ambient temperature changes affecting the display unit
- Long-term stability is usually added by the user of the meter after a period of using time.

The repeatability type A uncertainty (UA) was calculated using the previous equation (3). Type (B) uncertainty was calculated using table (2).

**Table 2:** Uncertainty of electric meter type (B).

<table>
<thead>
<tr>
<th>Symbol</th>
<th>Source of Uncertainty</th>
<th>Probabilty distribution</th>
<th>Divisor</th>
</tr>
</thead>
<tbody>
<tr>
<td>R_s</td>
<td>Calibration of moisture meter</td>
<td>Normal</td>
<td>2</td>
</tr>
<tr>
<td>R_d</td>
<td>Drift of moisture meter</td>
<td>Rectangular</td>
<td>√3</td>
</tr>
<tr>
<td>S_s</td>
<td>Ambient temperature changes affect</td>
<td>Rectangular</td>
<td>√3</td>
</tr>
<tr>
<td>T_t</td>
<td>Resolution of moisture meter</td>
<td>Rectangular</td>
<td>3</td>
</tr>
</tbody>
</table>

Then the total uncertainty

\[ U_{total} = \sqrt{UB^2 + UA^2} \]

**Experimental results:**

**Table 3:** The moisture content with its uncertainty for four grain samples.

<table>
<thead>
<tr>
<th>Moisture Value</th>
<th>Moisture using oven St. method 7.0 ± 0.1%</th>
<th>Moisture using oven St. method @ 11.0 ± 0.1%</th>
<th>Moisture using oven St. method @ 15.0 ± 0.1%</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Microwave</td>
<td>IR ± 0.6</td>
<td>Meter</td>
</tr>
<tr>
<td>Adas</td>
<td>7.6</td>
<td>7.9</td>
<td>7.7</td>
</tr>
<tr>
<td>Rice</td>
<td>7.4</td>
<td>7.6</td>
<td>7.1</td>
</tr>
<tr>
<td>Lobiya</td>
<td>6.8</td>
<td>6.7</td>
<td>6.5</td>
</tr>
<tr>
<td>Beans</td>
<td>7.5</td>
<td>7.8</td>
<td>7.3</td>
</tr>
</tbody>
</table>

**Conclusion**

In this study, the uncertainties in the grain moisture content measured by the five apparatus; oven dry method (st), microwave oven and the IR apparatus and the other two electronic meters were evaluated and the data were referred to the SI units. The uncertainty of the oven dry method was ±0.1%. The uncertainties of the Microwave oven was (± 0.5) while for the IR is (± 0.6) and the electronic meters were found to be (± 0.8) and (± 1.2). It is clear that its values are a bit bigger because it includes the draft of the meter, its resolution and type A uncertainty beside the uncertainty of the st. method (± 0.1).

It is also clear from a general analysis of propagation of uncertainty in moisture measurement that the uncertainty data measured by different meters contain the uncertainty of the standard.

\[ \text{Uncertainty of standard} + \text{resolution of the meter} + \text{repeatability of the meter} + \text{draft of the meter (long term stability)} \]

It is clear that the dominating component affecting the uncertainty is the resolution, temperature effect and the drift. For meters has low resolution may be mainly affected by the drift of the instrument and temperature effect.
Although the uncertainty of the electric meters are high but it is widely used according to its easier in use nondistractive, gives fast results and some meters has long pins which enables the measurements in a deep lot of grains especially during export and import of grains.

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