DESIGN AND TESTING OF AN INSTRUMENTTO MEASURE EQUILIBRIUM MOISTURE CONTENT OF GRAIN

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ABSTRACT. *Two instruments to measure the equilibrium moisture content (EMC) of grain were designed and tested under different grain conditions to determine their measurement time and suitability for quick spot measurements. An initial prototype was developed and tested and used to refine a second prototype. Both used a relatively inexpensive digital relative humidity and temperature sensor. Objectives were to determine factors affecting measurement performance and methods to minimize the effect of the factors which degraded performance. Specifically, the time response of the sensor was considered a major obstacle in obtaining quick measurements. Results showed that airflow over the sensor (0.60 m^3/h) was required to reduce measurement times to an acceptable level. Modeling of the initial measured data with an exponential equation helped to predict when the sensor readings were in equilibrium with the grain environment, and reduce measurement time, but significant error can occur between predicted EMC and actual EMC values. Error correction methods were developed that reduced the error significantly but the methods are potentially sensitive to changes in the operating parameters of the instrument.*

Keywords. *Equilibrium moisture content, Grain, Quality, Storage.*

Moisture measurement and management is critical prior to storing grain but does not assure that grain quality will not deteriorate during storage. Stored grain can undergo changes in moisture due to moisture migration by natural convection of the interstitial air or by water infiltration due to structure leakage. Typically, convection problems occur in the top or bottom of a bin where warm moist air may come in contact with cool headspace and ducting surfaces and condense, or, along sidewalls where external heating and cooling can also cause condensation (Navarro et al., 2002). Problems that develop due to excessive moisture levels during storage, coupled with inadequate temperature management, are damaging insect infestations and the development of molds, yeasts, and in extreme cases bacteria. Once a problem occurs, it can be augmented by the metabolism of either the insects or microflora, as both tend to increase grain temperature and release additional moisture as they develop. Moisture measurement during storage would help reduce damage by measuring the dynamic changes that are occurring and to take timely corrective actions.

Several direct and indirect moisture measurement methods have been studied and have been commercially developed to replace standard oven drying methods. Grain electrical properties have been exploited extensively using techniques such as radio-frequency dielectrics, microwave transmission (Kandala et al., 1993; Kim et al., 2006), simple capacitance or resistance methods, and time-domain reflectometry (IMKO Micromodultechnik, GmbH, 2006). Sinar Technology (Berkshire, UK) uses a proprietary 'electric field' technology to measure grain moisture. Typically these type of instruments measure dielectric property changes due to moisture and are accurate and fast but require good calibration development. Near-infrared methods are also popular for moisture measurement which is partly due to the ability to also measure grain composition such as protein, oil, etc. All of these methods are beneficial in specific applications. Grain equilibrium moisture content (EMC) prediction is particularly attractive in some applications considering the availability of inexpensive and reliable sensors to measure relative humidity (RH) and temperature (T). Currently there are no systems or instruments available which utilize RH and T measurement to predict EMC, although there are instruments which measure water activity and T (Decagon Devices, 2006) and probes which will measure RH and T (Models HMP46/HMI41,Vaisala, Woburn, Mass.).

Equilibrium moisture content and temperature determines the environmental conditions of the interstitial storage air, which, in turn, is the primary factor for favorable or unfavorable conditions for insect or mold development. The physiological response of different stored product insect species and molds is most commonly related to T and RH or water activity (a_w). This is especially true for microflora where various species respond differently to T and RH (Lacey et al., 1980), while insect species respond predominantly to T (Navarro et al., 2002). EMC can be expressed as a function of RH and T using common equations such as the Chung-Pfost, Henderson, and Oswin relationships. Although EMC relationships are grain-type,

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hybrid, or variety specific, and are affected by agronomic conditions, the ease of measuring RH and T with modern sensors makes the use of these relationships attractive for monitoring stored grain. A disadvantage of using RH and T for quick MC measurement can be the slow time response of the sensor. Chen (2001) determined a measurement time of 10 min was required for the RH and T sensor he studied to equilibrate to the grain environment for accurate measurement. Tests were conducted in a stagnate air environment. Other research (Bunn and Buschermohle,1986, Chung and Verma, 1991) found accuracy of EMC methods diminish at high humidity. Young (1991) made the following observations about EMC prediction: (1) accurate EMC/ERH/T relationships need to be established; (2) grain equilibration with the environment needs to be established when the measurement is made; (3) RH measurement error, at high RH, results in a large MC error; and (4) RH measurement error itself can be significant.

From a practical view, the accuracy of predicting the EMC is more important for grain trading while predicting a_w and T are better indicators of storage conditions. Uddin et al. (2006) examined the effect of RH and T measurement accuracy on EMC prediction and found that sensor error contributed to a maximum dry basis MC error of about ±0.65%; at high relative humidity error increases substantially. Uddin (2005) also determined regression coefficients for the Chung-Pfost equation for 47 varieties of wheat. The standard error of residuals from regression ranged from 0.31% to 0.63% MCdb (dry basis MC) for these EMC relationships. These studies help define the source of error in predicting MC using RH and T measurements from EMC relationships.

The objective of this work was to design and evaluate a handheld grain moisture instrument that uses RH and T sensors to predict the EMC of grain. The instrument was designed as a probing device for spot measurements with the capabilities for long-term spot monitoring. It uses an inexpensive commercial RH and T sensor that has excellent cross-sensor inter-changeability. Specific objectives were to examine implementation of the instrument/sensor system for EMC prediction, determine the time response of the instrument and implement improvements addressing the time response issue.

Experimental Procedures

Development of the instrument involved the examination of an early prototype and enhancements made to a second prototype. Procedures were thus divided into two sections. The initial tests were conducted on the first prototype to quantify operating characteristics. Knowledge gained from these tests was used to modify a second prototype. Comparison studies were then performed between the first and second prototypes.

Initial Tests - First Prototype

Equipment

The first prototype (fig. 1) was designed and constructed for the measurement of the interstitial RH and T of the air in the grain. Interstitial grain air was drawn through the probe tube, across the sensor, and exhausted to ambient air. Forced air was used to improve the response time of the instrument. The sensor had combined RH and T sensing capabilities and transmitted data digitally. The sensor (SHT75, Sensirion AG, Staefa, ZH, Switzerland) had a rated RH accuracy of ±1.8% RH and T accuracy of ± 0.3°C. Actual RH accuracy diminishes at extreme RH values. At RH >85% to 90% the grain is in serious danger and realistically the storage manager needs to take significant steps to preferably reduce RH to at least the mid to high 60% range which is within the sensor's higher accuracy range. The sensor was mounted in the probe tip and wiring from the sensor was routed through the probe tubing to a USB data acquisition device (USB-1208LS, Measurement Computing, Norton, Mass.) connected to a PC. The PC was used for experimental purposes only; a low cost embedded controller would suffice in a commercial instrument. An air concentrator was used to force air into intimate contact with the sensor. Twelve (2.5-mm dia.) round air inlets allowed air entry. The sensor was protected from dust by a porous cylindrical filter material (Porex®, Porex Corp. Fairburn, Ga.). Air was drawn across the sensor by a small compressor and was measured and controlled with a rotameter and needle valve. The drop in static pressure across the sensor probe was measured using a Magnahelic® (Dwyer Inst., Michigan City, Ind.). Pressure drop was measured for future design purposes of selecting a sampling fan to replace the air compressor and to determine the amount of pressure drop resulting from grain dust accumulating on the filter. The tube length and insertion depth was based on the maximum airflow that would be tested (0.60 m^3/h), the maximum time of testing (20 min.) and the subsequent volume of air extracted from the grain during the measurement. Sampling air for too long of a period would eventually start to draw air from outside the test grain volume. An insertion depth of 600 mm was determined adequate for a 20-min measurement based on an 18 bushel grain volume and porosity of 35%. A custom program was written in Visual Basic 6.0 (Microsoft Corp, Redmond, Wash.) to communicate with the sensor and record data via the USB data acquisition device. The four sensor wires were connected to two input-output (I/O) ports, a five volt power source and ground on the USB device. One I/O port is used to clock the other I/O port which sends commands to and retrieves data from, the sensor. Decimal RH, T, and EMC were recorded every 4 s. EMC (dry basis) was computed using the modified Chung-Pfost relationship (eq. 1) for hard red winter wheat. The Chung-Pfost coefficients used were from ASAE Standard D245.5 (2005a) where a = 610.34, b = 0.15526, and c = 93.213.

$$MC = -\frac{1}{B}\ln\left[\frac{-(T+C)\ln(RH)}{A}\right] \qquad (1)$$

Wheat Samples

Hard red winter wheat was conditioned to high, moderate and low moisture levels of 22%, 13.8%, and 11.6% MCdb, respectively. Initial wheat MC was determined by oven drying (ASAE Standard S352.2, 2005b) and distilled water was gradually added to the wheat as it was stirred in a large mixing chamber. Three containers (approximately 18 bushels) were placed in a cooler at 15°C and filled with the grain at the different MCs. Three similar containers were filled with the grain and left at ambient conditions (approximately 25°C) next to the cooler. These grain

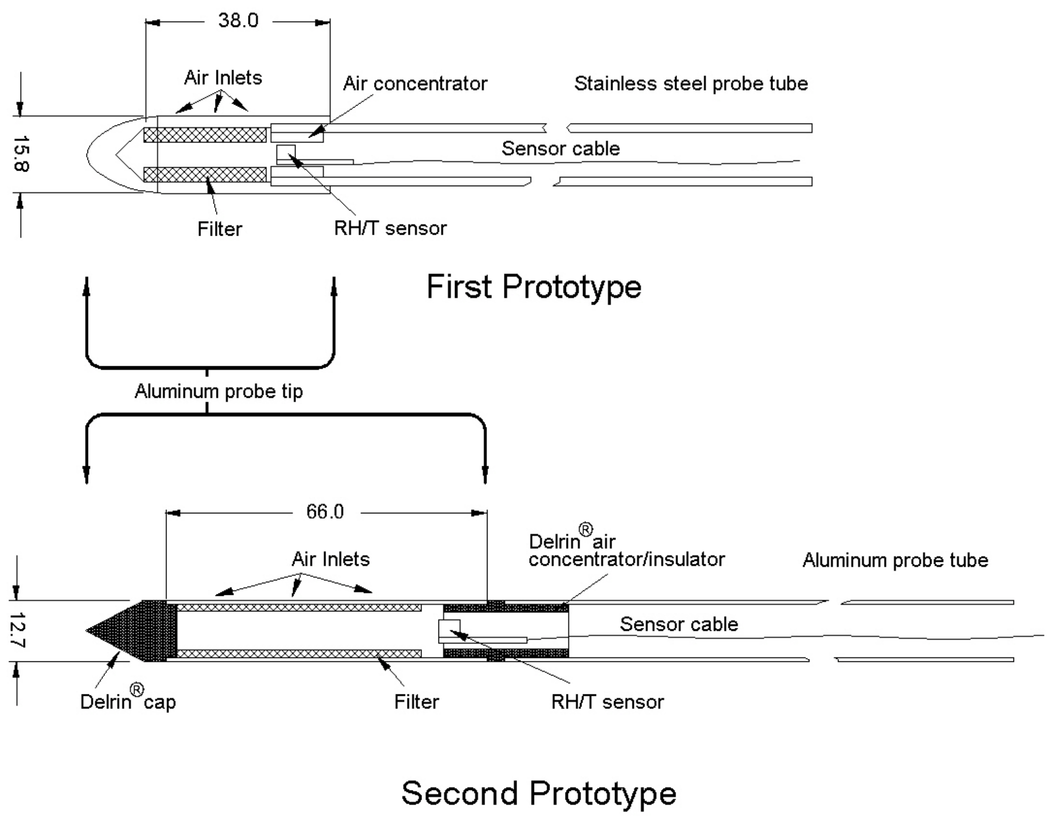

Figure 1. First and second prototypes instruments for EMC measurement (dimensions are in mm).

conditions were used to approximate the extreme temperature and MC conditions that the instrument probe might be subjected to. Actual MC, as specified above, was determined by oven drying (130°C for 19 h) after the sample had equilibrated for seven days.

EMC Measurements

EMC measurements were collected on grain using the first prototype, alternating measurements between those taken inside the cooler and outside the cooler. Measurements at an airflow of 0.15 m^3/h were taken sequentially on the warm 11.6% MC grain, the cool 13.8 %, warm 13.8%, cool 11.6%, warm 22%, and cool 22% grain. This was repeated for 0.30- and 0.60-m^3/h airflows. The entire sequence was then repeated after replacing sensors with a different sensor. Recording for each measurement was stopped after 20 min. The time to reach an equilibrium EMC value was determined from data. Equilibrium was defined as the time at which the time rate of change in EMC was equal to or less than 0.15%/min.

EMC time response modeling was then performed using non-linear regression to determine if equilibrium values could be predicted using a short time interval of the initial data. Details of the modeling analysis are presented in the results. Non-linear regression analysis was then programmed into the controlling program to predict equilibrium EMC values as probe measurements were made. One hundred additional measurements were collected on wheat samples at five moisture contents of 11.5%, 13.4%, 16.7%, and 22% MCdb, and at temperatures of 8°C, 20°C, and 35°C using the prediction model. The samples were mixtures of the original conditioned wheat and were about half the volume as measurements were limited to a 10-min sampling period. A single EMC measurement was also taken with 1) no airflow using an exposed sensor (no probe housing), 2) the probe under static air conditions, and 3) the probe with 0.60-m^3/h airflow. This provided a simple measure of the effect of the probe housing and airflow on sensor response.

Second Prototype Tests

Equipment

Modification of the probe (fig. 1) was done using performance characteristics obtained from the first prototype. The probe function was essentially the same as the first probe with the following differences: the filter element was replaced with a thinner element to reduce pressure drop and a smaller thin walled (0.83-mm) aluminum tube was used to reduce thermal mass. Thirty-five (2.5-mm dia.) air inlets were used.

EMC Measurements

The performance of the second prototype was compared to that of the first prototype. Measurements were collected with both instruments on wheat samples at four nominal moisture contents of 10%, 13%, 18%, and 22% MCdb, at temperatures of 8°C, 20°C, and 35°C, with different initial probe temperatures (8°C, 19°C, and 35°C), to determine the response time performance of each instrument. Measurements were made with the first prototype and then the second on the same wheat sample; the next measurement was made on a different wheat sample using the same probe measurement order, i.e. the first then second prototype. The

Table 1. Sequence of measurements.

Initial Probe T (°C)	19	8	20	8	35
Final Probe T (°C)	20	20	8	35	8
MCdb	Measurement Sequence				
10	1	5	6	7	8
13	2	15	16	9	10
18	3	17	18	11	12
22	4	19	20	13	14

sequence of measurements, listed by grain and probe conditions in table 1, was designed to simulate conditions requiring maximum equilibration times. Measurements were performed in triplicate.

Results and Discussion

Initial Tests - First Prototype

Static Air Measurments

The EMC response times with 1) an exposed RH/T sensor with no probe housing and no airflow, 2) with the probe housing at 0.60-m^3/h airflow (fig. 1.), and 3) with the probe housing and no airflow were 600, 1300, and 3000 s, respectively (fig. 2). The response time was considered to be the time required to reach steady-state equilibrium. Equilibrium was determined by the same method described previously, i.e. the time rate of change in EMC was ≤0.15%/min. The stated time response, by the manufacturer, to reach 63% of the steady state value, is 4 s for RH and from 5 to 30 s for temperature with a small airflow. This equates to equilibrium being reached in approximately 12 s for RH and up to 180 s for T. Temperature time response is most likely affected by the thermal mass of the sensor and the rate of heat exchange to the sensor controlled primarily by the flow rate past it. The plastic filter media and the restricted flow around the sensor in the instrument, under static conditions, should impede the movement of air to the sensor. While the slow response time may be adequate for long term monitoring, quick measurements (< 5 min) are not feasible without airflow. Results from Chen (2001) showed similar periods of time are required for measurement. He found the accuracy of measuring ERH in stagnant air improved after 20 min of equilibration, compared to 10 min, using a Shinyei THP-B7T humidity transmitter (Shinyei Kaisha Co., Tokyo, Japan). Probes manufactured by Vaisala (HMP75, 6, and 7series, Woburn, Mass.) are claimed to have a 90% response time of 8 to 40s. No other information is given to characterize their response.

Dynamic Measurements and Prediction Modelling

EMC versus time at different flow rates is shown in figure 3. Transitions from cool to warm grain for lower airflows of 0.15 and 0.30 m^3/h resulted in high initial values of EMC. This was thought to be caused by the cool temperature of the sensor housing elevating the relative humidity of the incoming warm air. As the sensor housing warmed to ambient grain conditions, the relative humidity lowered quickly. Inspection of actual RH values supports this as they remain high and relatively constant, approx 95%, before falling (fig. 4). Temperature consistently increases. EMC values do not show this behavior for 0.60-m^3/h airflow and could be the result of the probe tip approaching ambient grain temperature conditions quickly due to the higher airflow. Data shows that 0.60 m^3/h airflow provided a more predictable response from the probe although considerable time is still required to attain equilibrium conditions.

The pressure drop, as measured from the sampling tube to ambient pressure, showed a value of approximately 340 ± 10 Pa. This did not vary significantly from the beginning to the end of tests. A noticeable amount of grain dust had collected on the filter but did not seem to affect the pressure. Some loose particles and dust were cleaned from the probe tip by wiping with a soft cotton rag when it was apparent it could be easily removed. Replacement of the sensor element did not noticeably affect the response of the instrument upon examination of response curves under the same transitioning conditions.

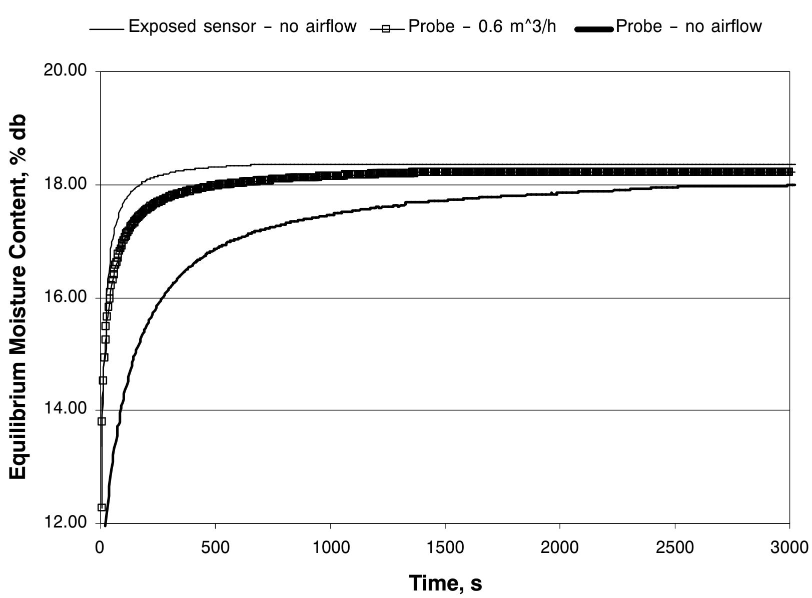

Figure. 2. EMC values vs. time for the first prototype with no airflow, 0.60 m^3/h airflow and for an exposed sensor.

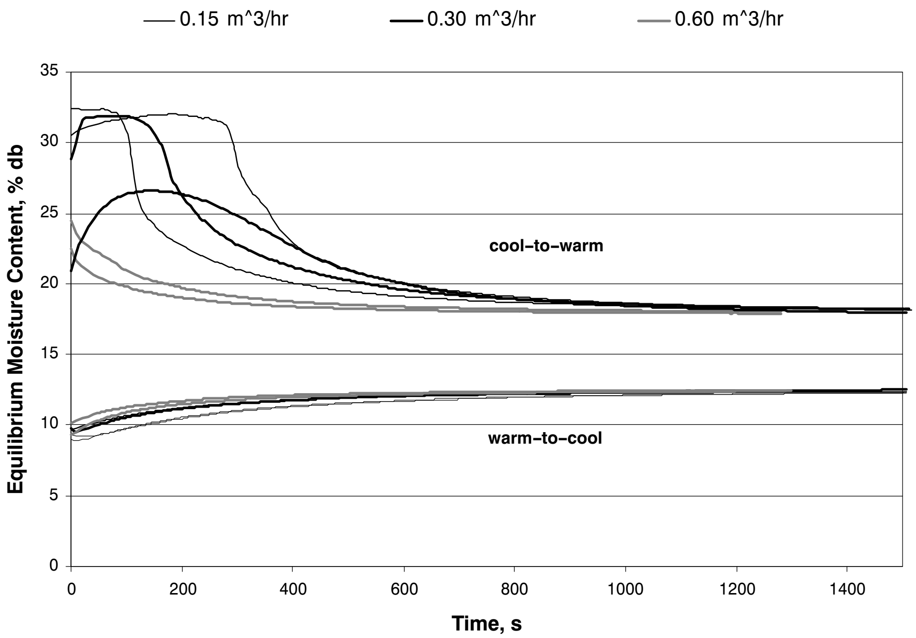

Figure 3. Measured EMC values vs. time for different airflow rates. Two replicates are shown for each airflow. The instrument was transitioning from a cool to warm or warm to cool grain.

EMC Time Response Modelling

The behavior of RH for airflows at 0.15 and 0.30 m^3/h over the initial few hundred seconds of data indicated that modeling this data to predict equilibrium values would be unreliable and was thus not considered in modeling. Response modeling was thus performed using non-linear regression for 0.60 m^3/h airflow only, over a time window of 200 s. The actual data used was the period between 10 to 210 s. A 200-s period was considered a feasible time for spot measurements to be made. Regression was performed using a statistical program which fit a large number of non-linear curve types to the data (TableCurve 2D, 5.01, SYSTAT Software Inc., San Jose, Calif.) Curves were examined for goodness of fit based on correlation values and then extrapolated to determine how accurately they predicted the true EMC value. True EMC values were determined by averaging the last 20 end points of data. Of the curves examined, equations 1 and 2 were chosen for more extensive evaluation. These equations were chosen as they will fit either an increasing or decreasing EMC value and approach equilibrium or steady state values.

$$EMC_{pred} = (a+bt+ct^2+dt^3) / (1+et+ft^2+gt^3) \qquad (1)$$

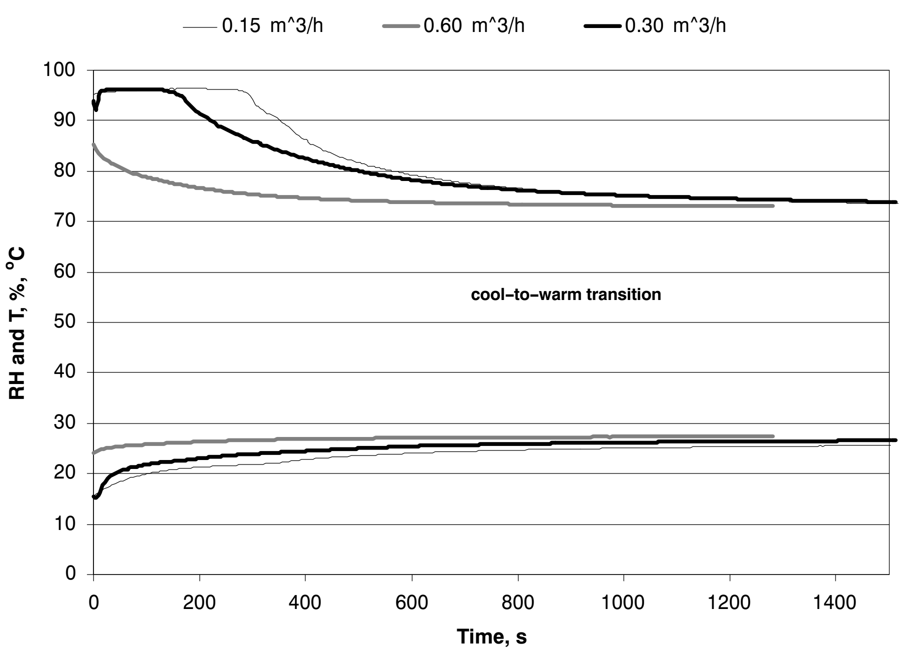

Figure 4. Measured RH and T used to predict EMC values in figure 3 for lower airflows with the instrument transitioning from cool to warm grain for replicate 1.

$$EMC_{pred} = a(b - e^{-ct}) \text{ or } a(b + e^{-ct}) \quad (2)$$

Equation 1 fit the data very well ($r^2 > 0.99$, F-statistic minimum = 120,456) better than equation 2 ($r^2 > 0.98$, F-statistic minimum = 6,456), but was highly unstable in a few cases due to poles occurring in the denominator. For this reason, its use was not pursued. Regression coefficients for equation 2 were determined for each measurement and the steady state value was determined for each case. In all cases, this equation type predicted EMC values that were higher than the true EMC if the curve was decreasing or lower, if the curve was increasing (fig. 5).

Because the error was fairly large, methods were examined to see if the predicted equilibrium value from equation 2 could be reliably corrected to the true EMC value using the difference between EMC at 200 s and the EMC at 160 s (ΔEMC). Error was plotted against ΔEMC and a reasonable linear fit was observed although conditions where a large temperature difference occurred between the initial probe temperature and final temperature at 200 s caused the most deviation from the linear relationship. To minimize the temperature effect, ΔEMC was recalculated at the time (t) where the temperature difference between the current temperature and temperature at t-170 s was less than 3°C. This was done to lessen the effects of temperature on measurement. The linear relationship developed from this and used to determine corrected EMC values is shown as equation 3 (F-statistic = 835, $p = 0.0001$). This equation was developed from the 100 measurements made on wheat ranging from 10% to 22% MCdb, and at temperatures ranging from 8°C to 35°C. A correlation of $r = 0.95$ was obtained between error and ΔEMC.

$$\text{EMC Corrected} = 0.1784 \cdot (\Delta EMC) - 0.0211 + EMC(t) \quad (3)$$

Using this protocol, most measurements were completed between 200 and 300 s with the longest requiring 432 s. Although this method reduced the error in all cases, there were still a few cases where the error was substantial. Table 2 shows statistics of absolute measurement error using equation 2, and equation 3 to correct the equation 2 error.

Table 2. Statistics of error in predicted EMC values from equation 2 and correcting equation 2 values with equation 3.

Prediction Method	Average Absolute Error	Standard Dev. Error	Minimum Error	Maximum Error
Eq. 2	1.23	0.85	0.14	4.23
Eq. 2 and eq. 3 correction	0.35	0.37	0.00	1.29

In general, the correction reduced error substantially except for a few cases as indicated by the maximum error (1.29%) observed in table 2. Changes in airflow rate should cause the correction relationship in equation 3 to change as it relies on the rate of change of EMC. Without monitoring airflow, the reliability of this correction is questionable. Monitoring pressure drop across the sensor could be a method to determine if airflow rate has changed due to constrictions from grain dust in the filter or some other physical change in the instrument.

Second Prototype Tests

Instrument Comparisons

The pressure drop for the second prototype, also measured from the sampling tube to ambient pressure, showed a value of approximately 140 ± 10 Pa and did not vary significantly from the beginning to the end of tests.

Equilibrium and measurement time were determined at the time when the rate of change in EMC was equal to or less than 0.15%/min. Measurement time for each instrument is shown graphically in figures 6 and 7 for conditions where the sensor head was approximately 9°C, prior to measurement, and the grain temperature was 21°C and, the sensor head was approximately 19°C prior to measurement and the grain temperature was 21°C, respectively. Each probe responded consistently as shown in figure 7 and replications clearly define differences in probe response. Comparison of measurement times between instruments showed that the

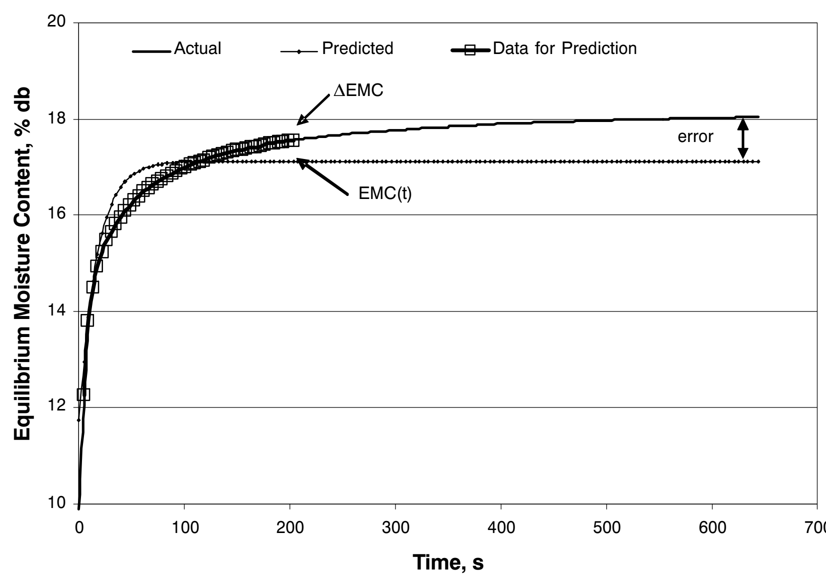

Figure 5. Modeled response of instrument using equation 2 from the first 200 s of data and actual response.

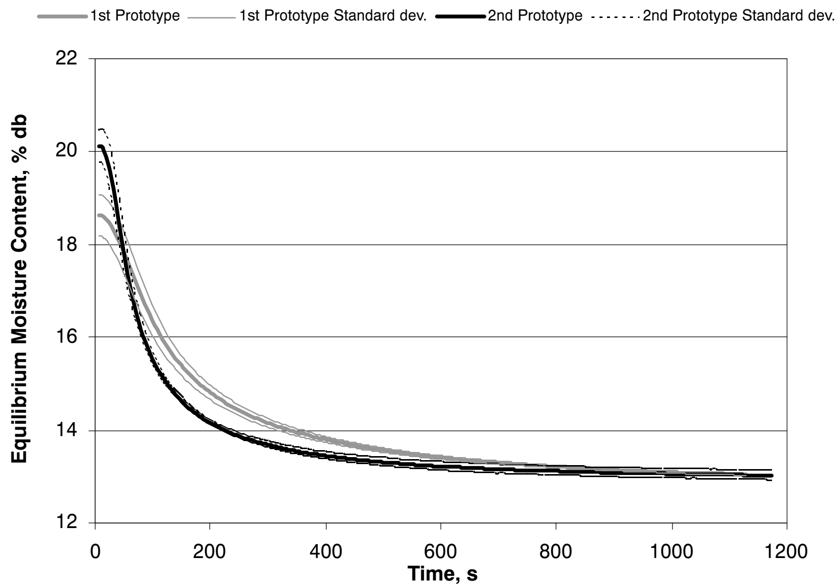

Figure 6. Measurements comparison between the first and second prototype instruments. The sensor probe was approximately 9°C, prior to measurement, and the grain temperature was 21°C.

second prototype performed better than the first (table 3). This was primarily attributed to the lower thermal mass of the sensor head. Measurement times are substantially longer for both prototypes when the probe undergoes a large temperature swing. There was minimal difference, within a prototype, between the 8-20°C and 8-35°C measurement times.

The measurement times shown are the actual times required for EMC equilibrium to be attained by the instruments and can be fairly long when large temperature differences are encountered. Correction methods developed for the first prototype were also applied to the second prototype data resulting in an EMC correction relationship shown in equation 4 (F-statistic = 421, p = 0.0001).

$$\text{EMC Corrected} = 0.2928 \cdot (\Delta\text{EMC}) + 0.0118 + \text{EMC}(t) \quad (4)$$

Statistics of EMC correction (table 4) show improved performance over the first prototype (table 2). Overall the second prototype performed measurements quicker and more accurately than the first prototype. The actual times required for measurement using the prediction and correction equations ranged from approximately 240 to 300 s.

As previously stated, a problem with using this type of correction is that airflow changes may cause the correction relationship in equations 3 and 4 to change as it relies on the rate of change of EMC. Monitoring airflow would be highly desirable.

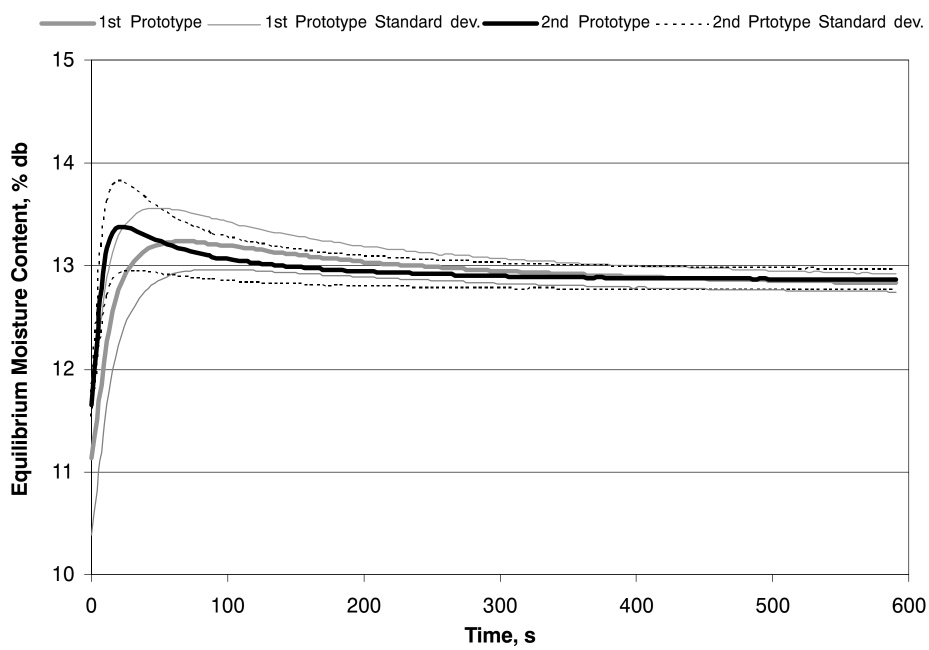

Figure 7. Measurements comparison between the first and second prototype instruments. The sensor probe was approximately 19°C prior to measurement and the grain temperature was 21°C, respectively.

Table 3. Average and standard deviation of the response time for the first and second prototype instruments.

		MCdb (%)	Average, Standard Dev. of Measurement Time (s)				
First prototype		10	291, 28	852, 37	880, 68	863, 62	862, 76
		13	278, 73	924, 18	936, 43	889, 69	932, 33
		18	293, 53	888, 45	913, 34	885, 56	871, 56
		22	213, 34	936, 63	938, 24	925, 65	904, 37
Second prototype		10	136, 53	721, 30	700, 65	713, 39	703, 55
		13	140, 23	731, 23	687, 55	778, 71	652, 35
		18	135, 45	724, 47	746, 57	748, 51	709, 40
		22	167, 29	748, 24	679, 41	732, 25	746, 64
Probe T (°C)	Initial		19	8	20	8	35
	Final		20	20	8	35	8

Table 4. Statistics of error in predicted EMC values from equation 2 and correcting equation 2 values with equation 4.

Prediction Method	Average Absolute Error	Standard Dev. Error	Minimum Error	Maximum Error
Eq. 2	0.39	0.28	0.01	0.92
Eq. 2 & eq. 4 correction	0.12	0.11	0.00	0.44

CONCLUSIONS

EMC measurement with the prototype instruments required a forced airflow to reduce measurement time to an acceptable level for spot measurements. The temperature differential between the instrument probe prior to insertion into the grain and grain T has a significant effect on response and measurement time. The response time of the second prototype was somewhat better than the first prototype and was likely the result of a smaller thermal mass. The sensors used in the study did not appear to degrade in measurement accuracy after extensive use of being subjected to several hundred measurements. The filter methods used seem adequate in protecting the sensor.

Modeling response time can reduce measurement time by predicting equilibrium conditions but the exponential model used was not entirely adequate. Correcting for errors in the model using an empirical linear correction factor based on observed errors reduced error significantly but should be used with caution as changes in airflow may likely affect response behavior. Compared to results from Chen (2001), measurement times were generally reduced substantially by using forced air.

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