Successful Moisture & Water Activity (aW) measurements in Pet Food Kibble utilizing the Liebherr, Litronic FMS-II "In-line" Analyzer

A: General Information

"In-situ" real-time constituent measurements provide many benefits to a manufacturing process. Accurate data will result in product consistency, savings on rework, labor and scrap, while providing improved sustainability and customer satisfaction. This report summarizes our in-house **moisture**, **water activity** (aW) and **density** testing on pet food kibble samples collected after the dryer. The moisture ranged from ~ 1.0 to 14%, aW from 0.10 to 0.76 and density from 0.38 to 0.43 grams/cc.

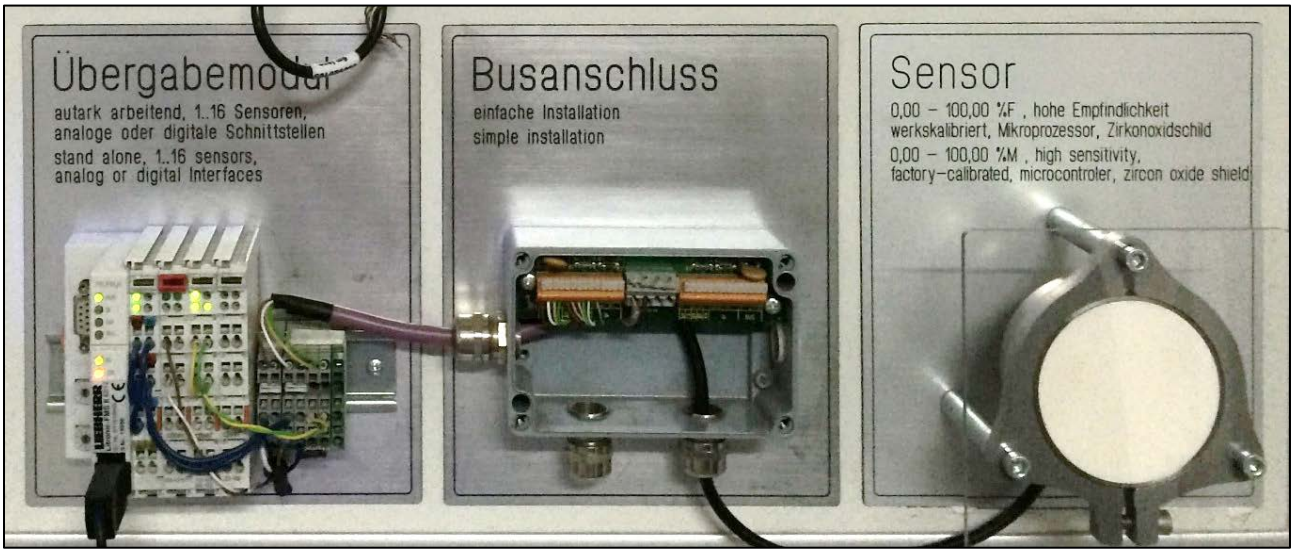
System and sensor description: A single Liebherr FMS-II controller was used for testing the optional **P78** (3-inch, 78mm diameter) and smaller **P30** sensor heads configured in a "static" bench-top configuration.

The Liebherr FMS-II Litronic is a robust in-line/at-line analyzer that measures moisture and other constituents in food, beverage, petro-chem, slurries and aggregates. Current sensor locations include conveyor belts, chutes, silos, webs and pipelines with up to 16 optional heads per single controller at distances of up to 3,900 feet. Click here for a technical description of the technology.

The Litronic has no moving parts. The sensor interface options are: **1)**, food grade "PEEK" or **2)**, industrial grade Zirconium Alumina Oxide (ZrO_2 - Al_2O_3). Explosion and Dust Proof versions are also available. With 10,000 plus sensor heads installed over 35 years, reliability is ensured.

A complete Liebherr system consists of **a)**, a micro-controller that outputs data to a customer interface system such as a PLC via RS232, Ethernet, Profibus, 4-20mA or 0-10V DC to **b)**; an optional length-extender RS485 buss to **c)**, an **"intelligent"** sensor head that is installed in the customer's process and connected back to the controller.

a. **b.** **c. (P78 shown)**



B. Objectives, Test Highlights and Preliminary Summary

Objectives:

1. To determine the feasibility of this application.
2. To determine instrument parameters for the measurement with regards to this product.
3. To obtain a preliminary indication of the sensitivity and precision of the instrument measuring the representative test product and unknown "blind" samples.

Test highlights:

Both sensor heads have excellent correlation (r2) to moisture content and aW in pet food kibble however there was no correlation to density.

NOTE: Because the sensor scans 35x per second, sample errors and packing density can be substantially mitigated with consistent product flow and signal smoothing using the Liebherr controller or an end users' PLC/SCADA control system.

Preliminary Summary:

Based on the results of this report, moisture and aW measurements utilizing the Liebherr in-line analyzer on this type of product would be an excellent application for this technology.

C. Calibration and Unknown "Blind" Sample Descriptions

Eleven samples of approximately .350" x .400" inch sized kibble was used to develop the **base calibration.** Ten unknown "blind" samples of various sizes and formulations were then measured to see how well the Liebherr could predict **moisture** for kibble **not used in the calibration.** These ten samples varied in size from the smallest at 0.270" x 0.200" inches to the largest at 0.350" x 0.600".

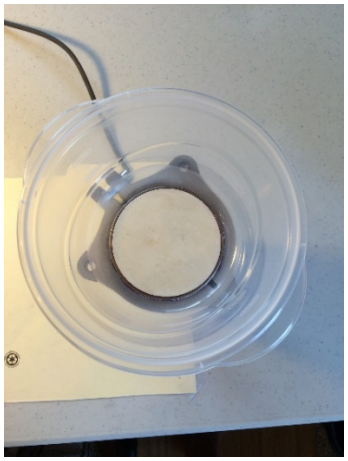
NOTE: There are many shapes and sizes of kibble produced in a manufacturing facility. The Liebherr can store hundreds of different curves giving our customers the flexibility to add as many recipes as necessary. We feel confident that several different products could be combined into fewer curves based on grouping kibble size; however, more testing would be needed to confirm this.

D. Test environment and Sample Procedure

The **FMS-II Liebherr Litronic** in-line analyzer was tested in a static configuration at ambient temperature (74F), pressure and humidity conditions.

1. Calibration sample #1 was poured into a large bowl and gently folded using a slotted spoon. This was to ensure the samples were homogenous for presentation to the Liebherr instrument.
2. Each sample was then "loosely" poured into the 500ml sample bowl affixed to each of the P30 and P78 heads respectively.
3. The Liebherr ***"Digit"*** value was recorded for each head that is used to predict %H2o, aW and density.
4. The sample was then lightly shaken for 3-seconds to partially settle the sample and the new Liebherr digit value was recorded.
5. The sample was then firmly "tapped" 10 times from the bottom to further settle the kibble and the new Liebherr digit value was recorded. The sample was then quickly returned to its airtight container.
6. Steps two through five were repeated for all calibration and "blind" samples.

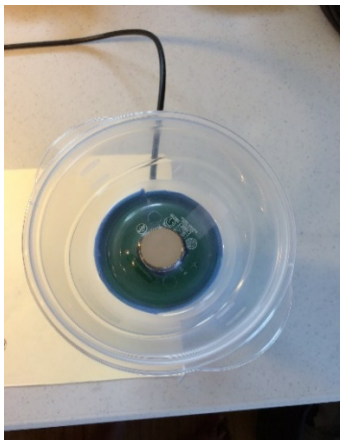
Sensor Setup



P78 top view

P78 side view

P78 filled view



P30 top view

P30 side view

P30 filled view

E: Moisture and Water Activity Results and Graph Explanations

1. The **ten calibration** "Digit" readings were used to generate coefficients used for moisture and aW using Y = (m*x) + b for linear predictions. The moisture curves are shown in **F1 & F2** for both sensors.

2. The equation strings were uploaded to both sensor heads and actual values were then recorded for **validation** using the same fill and dump methods used in section D for calibration. These measured **moisture** results are shown in **G1 & G2**.

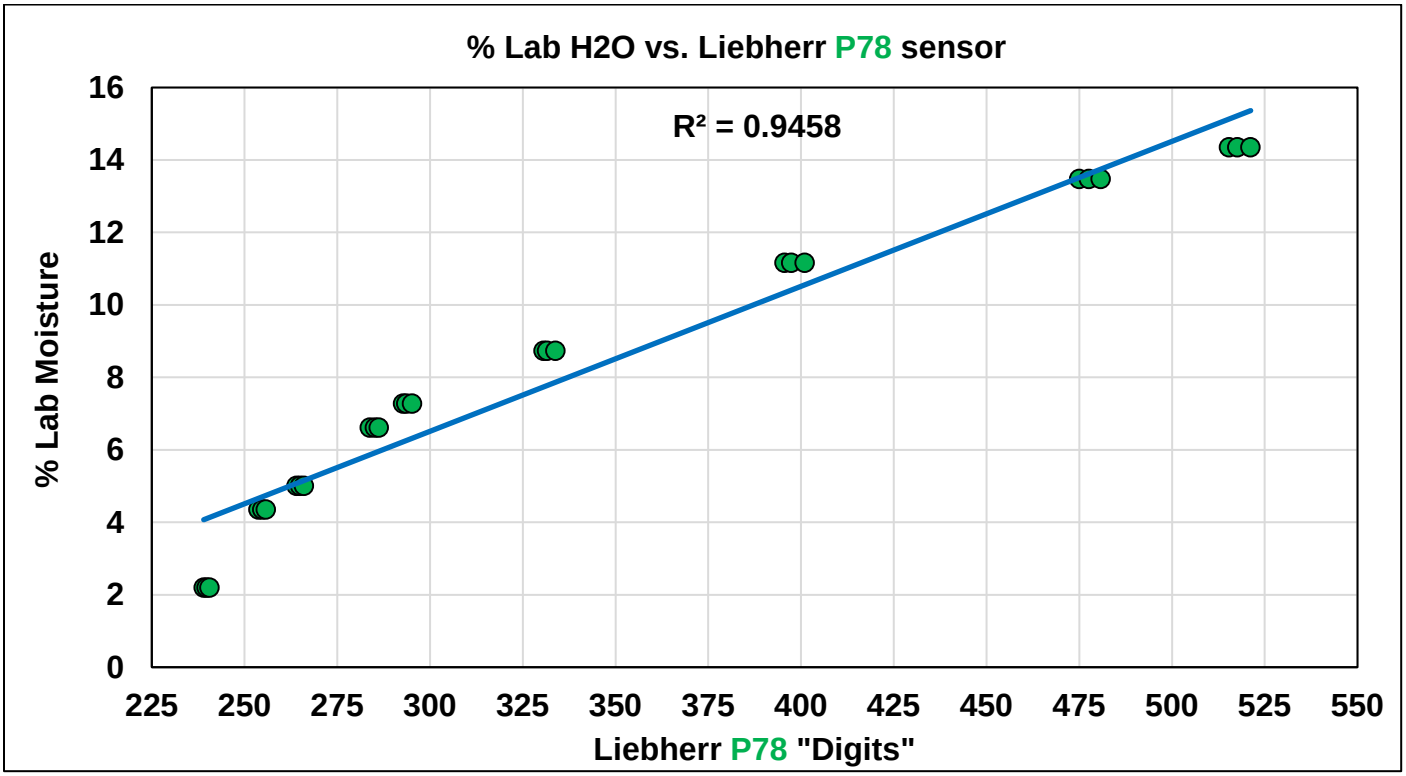
3. The unknown **"blind"** samples were then analyzed as in step 2 above using only the "10X Tap" fill method with the measured **moisture only** results shown in **H1 and H2**. *As previously stated, the blind samples were not used in the calibration that explains the lower r2 and higher standard error.*

4. **Predicted aW** results for both heads are shown in **I1 and I2**.

5. **Measured aW** validation results for both heads are shown in **J1 and J2**. (The same procedure was used as in #2 above with obvious different equations)

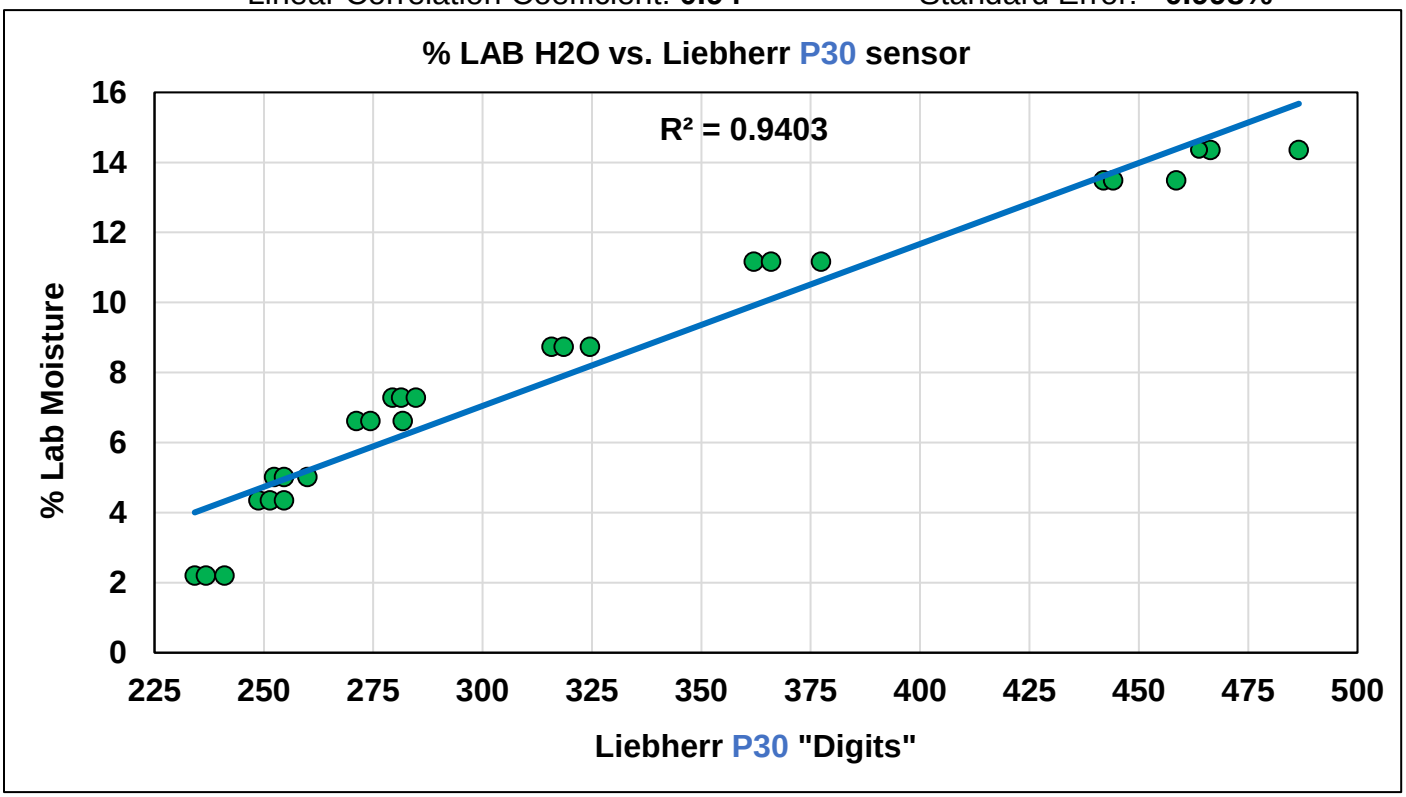
F1. Calibration Lab Samples % H2o Test Results, P78 sensor head

Linear Correlation Coefficient: **0.95** Standard Error: **0.95%**



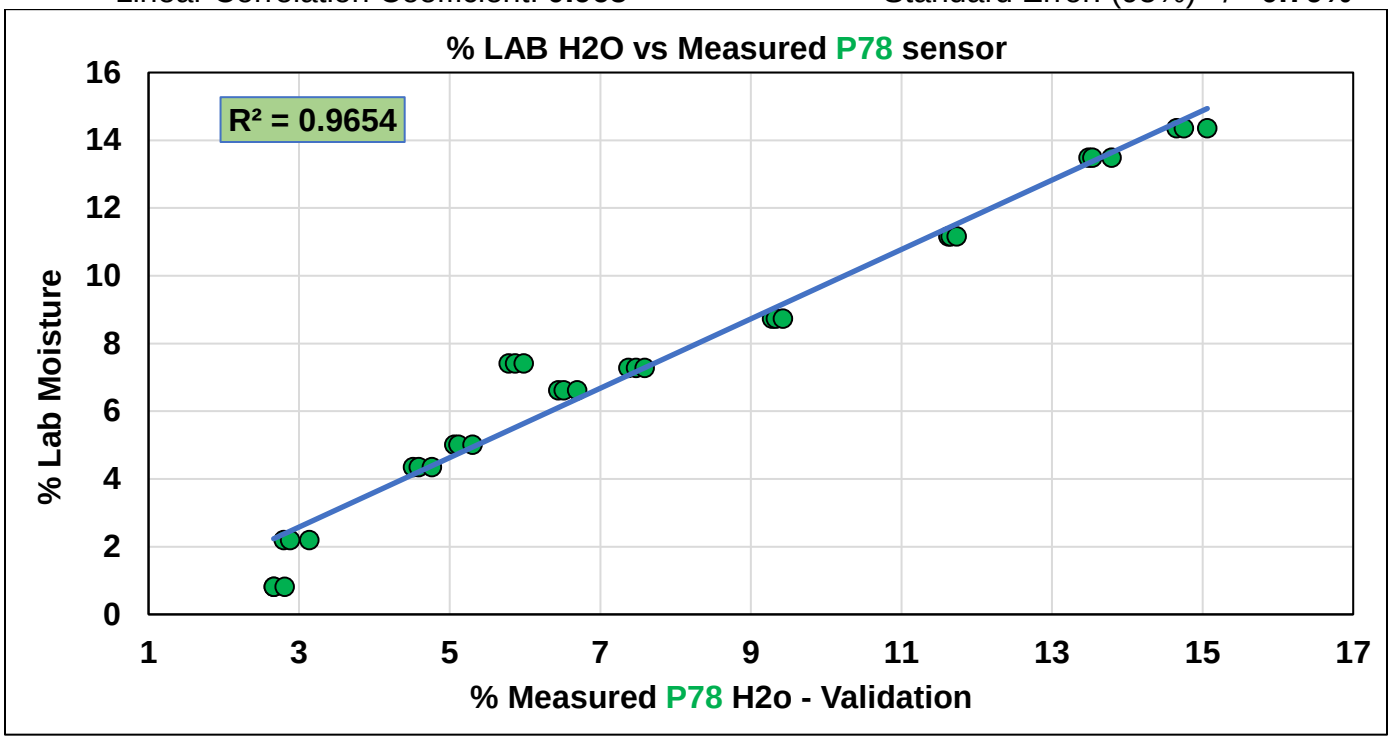
F2. Calibration % Lab Samples H2o Test Results, P30 sensor head

Linear Correlation Coefficient: **0.94** Standard Error: **0.998%**



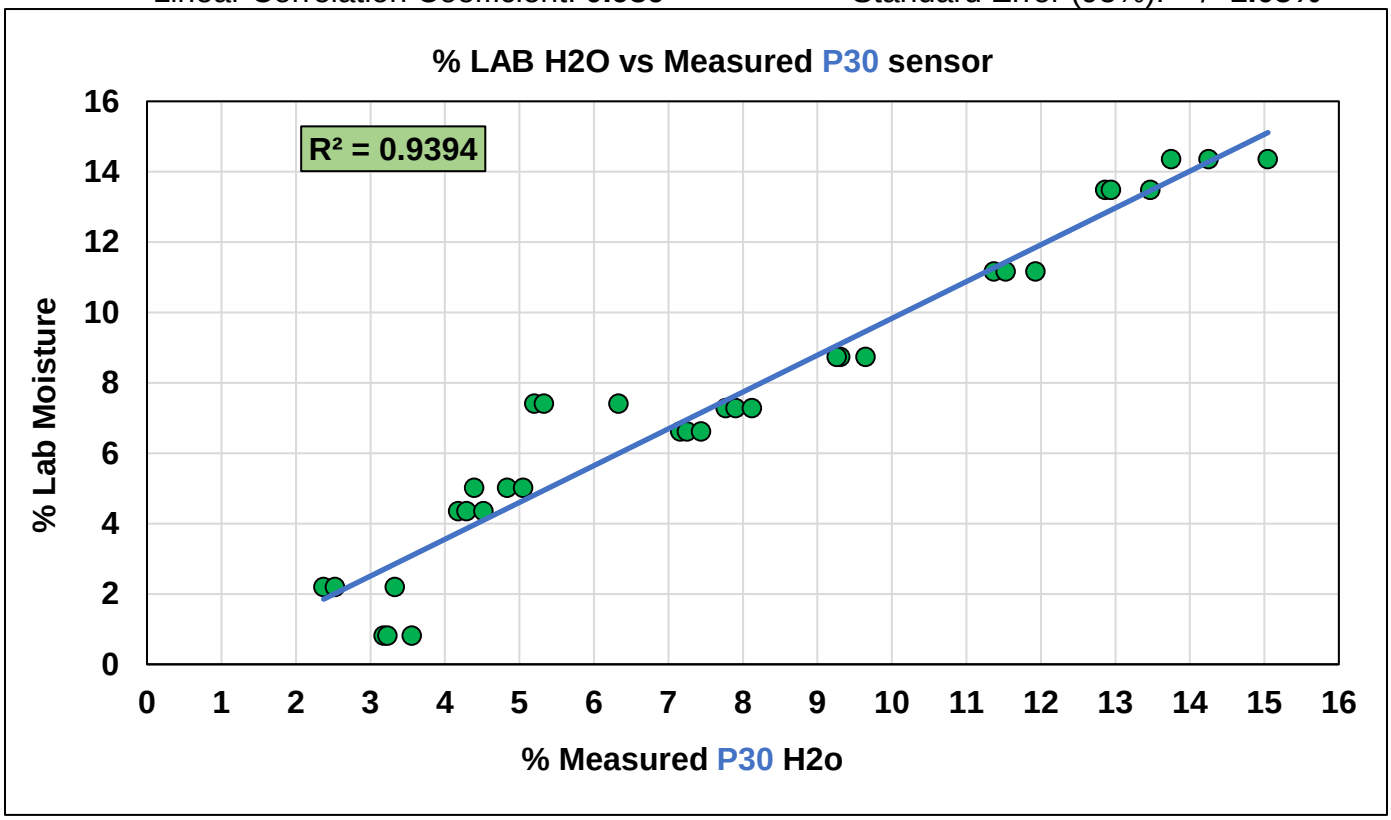
G1. Validation % Lab Samples H2o Test Results, P78 sensor head

Linear Correlation Coefficient: **0.965** Standard Error: (95%) +/- **0.79%**



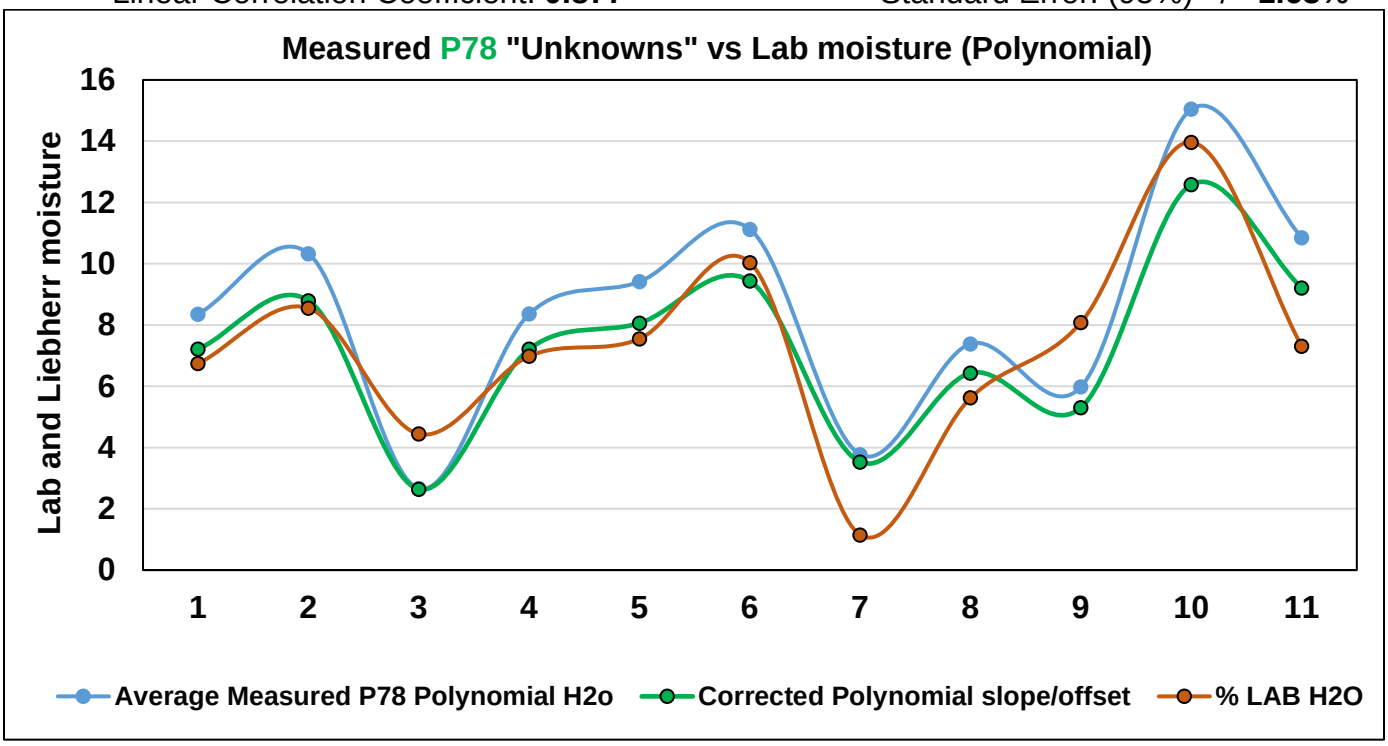
G2. Validation % Lab Samples H2o Test Results, P30 sensor head

Linear Correlation Coefficient: **0.939** Standard Error (95%): +/- **1.05%**



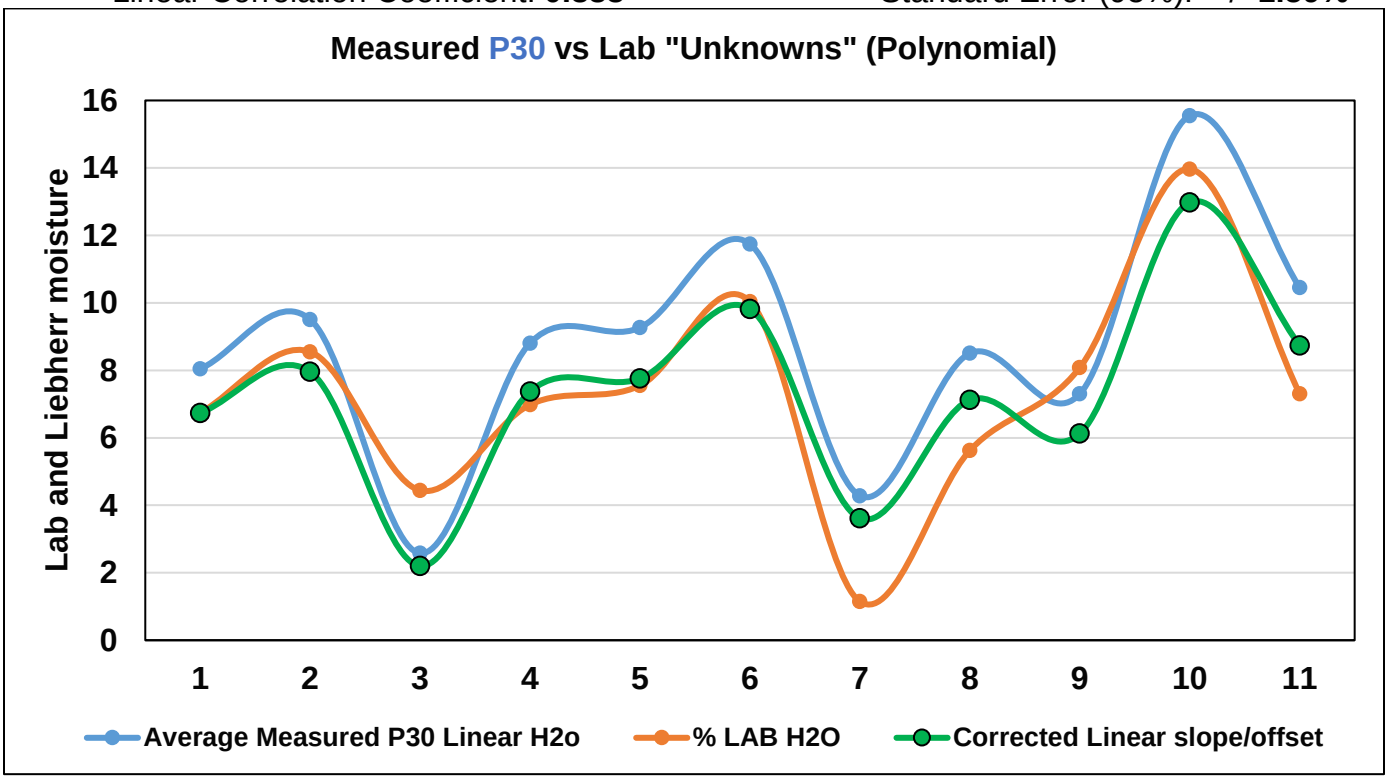
H1. “Blind” Sample % H2o Measured Results, P78 sensor head

Linear Correlation Coefficient: **0.877** Standard Error: (95%) +/- **1.63%**



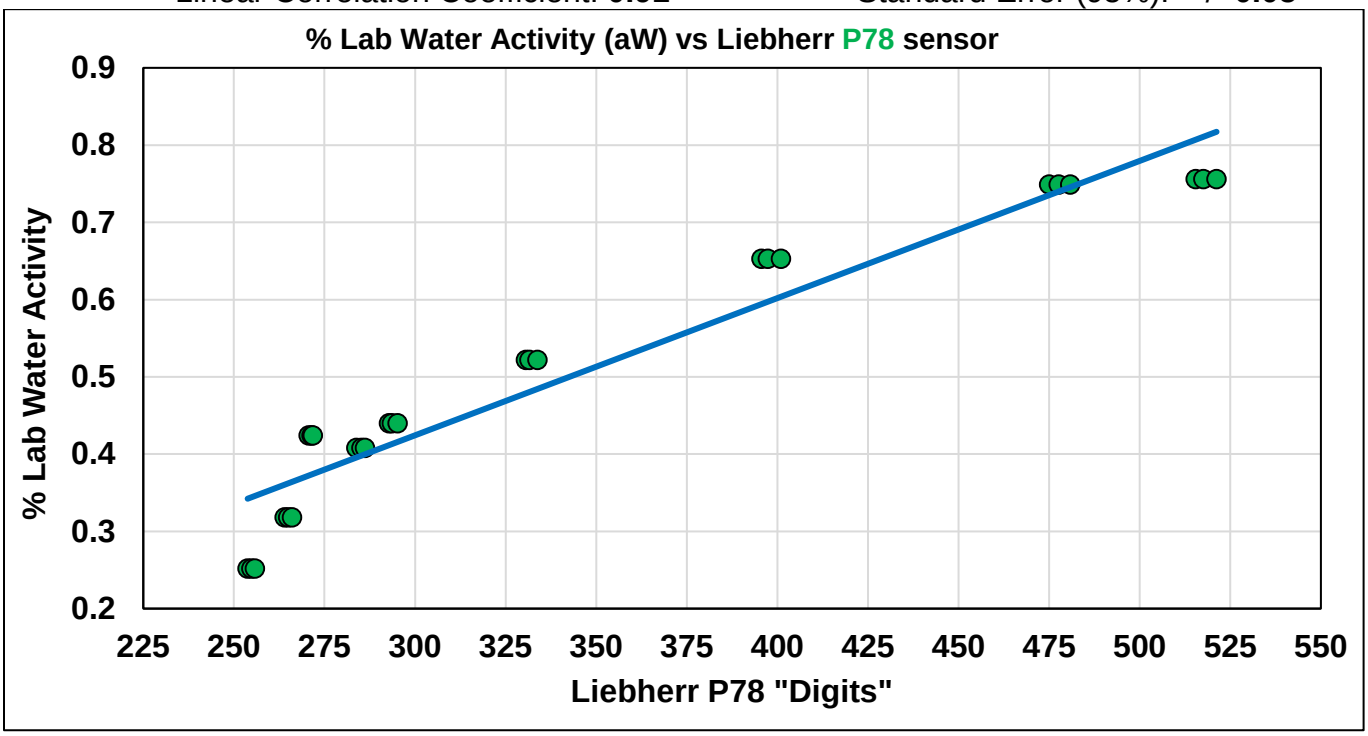
H2. “Blind” Sample % H2o Measured Results, P30 sensor head

Linear Correlation Coefficient: **0.883** Standard Error (95%): +/- **1.59%**



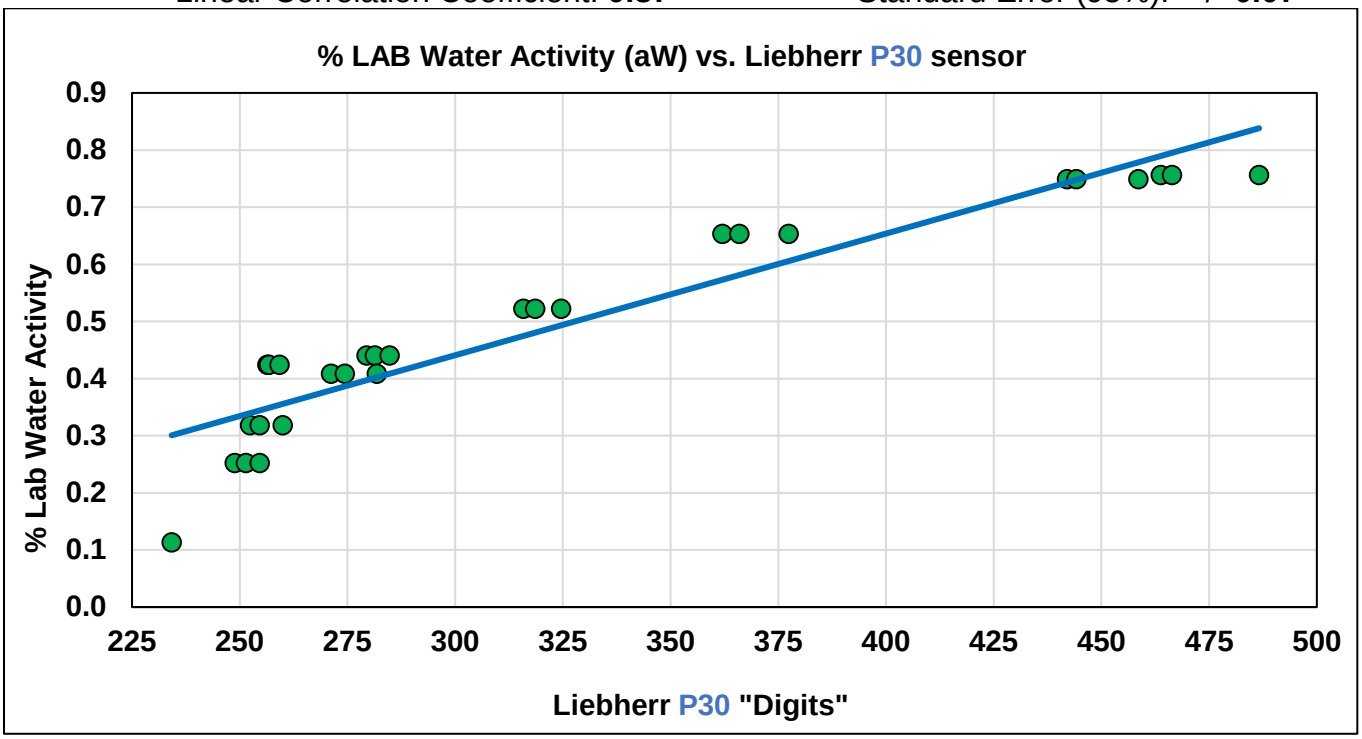
I1. Calibration Water Activity (aW) Results, P78 sensor head

Linear Correlation Coefficient: **0.91** Standard Error (95%): +/- **0.05**



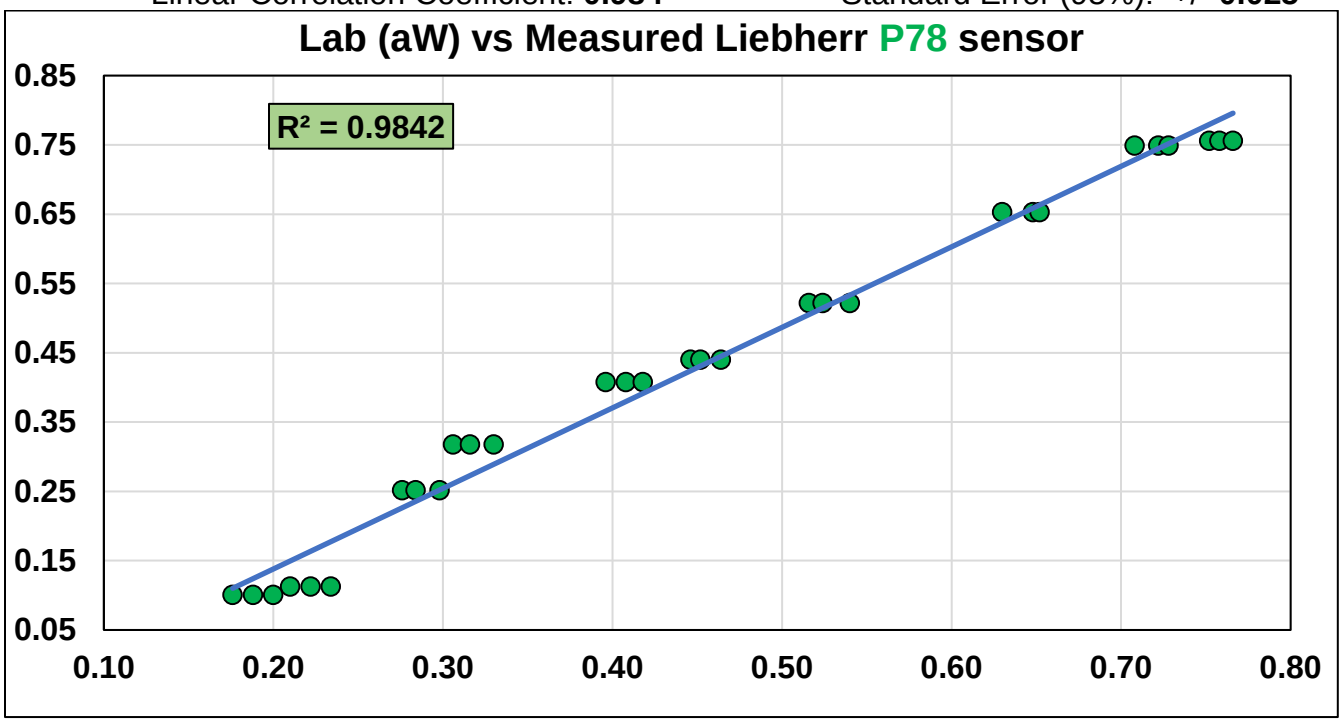
I2. Calibration Water Activity (aW) Results, P30 sensor head

Linear Correlation Coefficient: **0.87** Standard Error (95%): +/- **0.07**



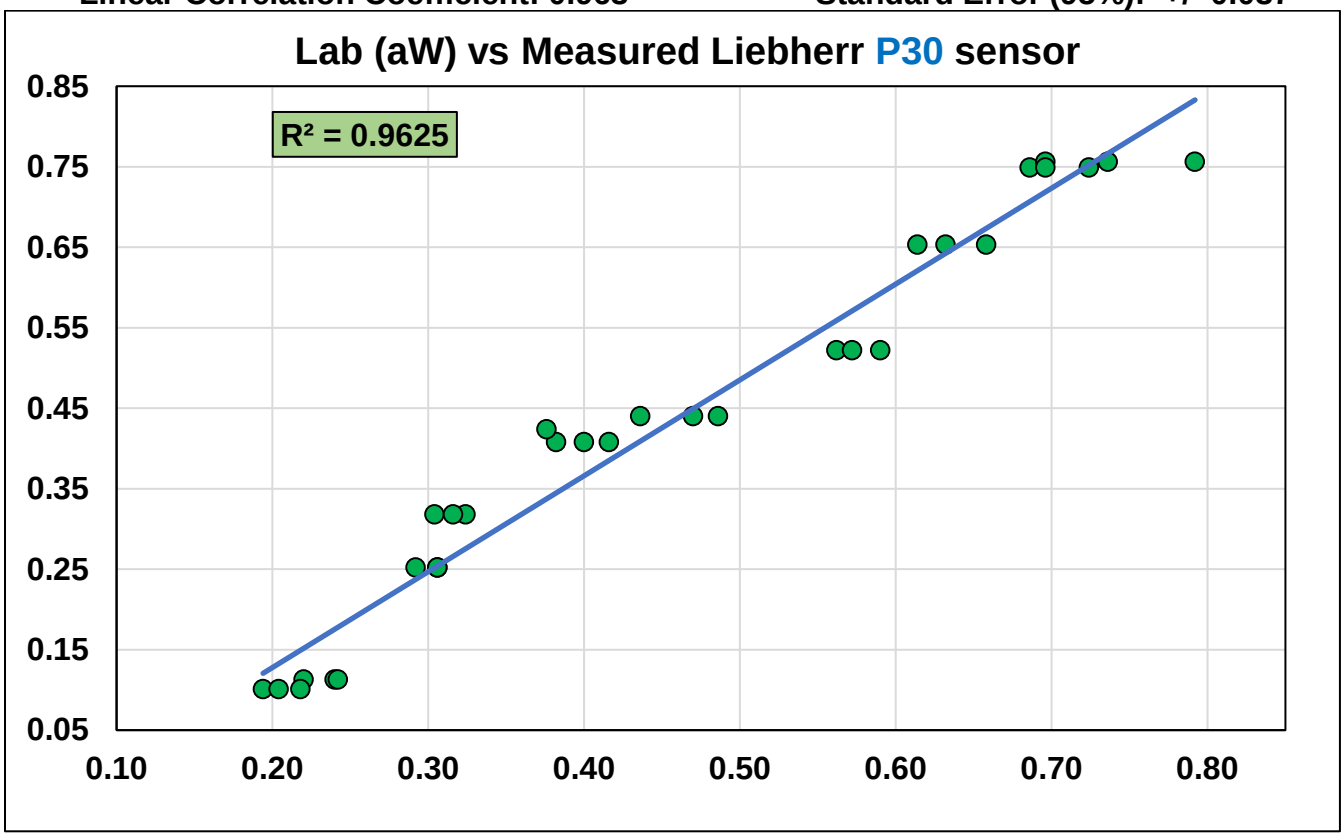
J1. Validation Water Activity Results, P78 sensor head

Linear Correlation Coefficient: **0.984** Standard Error (95%): +/- **0.025**



J2. Validation Water Activity Results, P30 sensor head

Linear Correlation Coefficient: 0.963 **Standard Error (95%): +/- 0.037**



K. Discussion, Definitions and Conclusion

The preceding information constitutes a preliminary report for review. The statistical values presented are based on data generated in our laboratory versus a limited quantity of material samples. Accuracy may differ somewhat from these results, depending on variations in material and actual process line conditions. In addition, poor sampling and lab analysis technique will add to instrument error. For these reasons, any study or statistical evaluation for any in-line measurement should be done with the sensor installed in the optimal production location.

Inconsistent voids or gaps in front of the sensor will cause variations in the instrument readings, however using various analyzer settings these false readings can be ignored. Consistent presentation of the product will improve the measurement for any in-line system and oftentimes have less error than the lab analyzer based on the large number of readings on all of the material under test, not just small portions.

R-Square (r2) description:

The correlation coefficient, r2 is a mathematical expression of the randomness of the data points around a linear regression line. It is a function of both the accuracy of the measurement and the range of moisture values in the calibration sample standards. A value of 1.0 is perfect (and not possible), however for many applications a value of >.80 is considered acceptable in most industries.

Standard Error description:

The standard error is the variability between samples that you would obtain if you took multiple samples in the same data set. It is also a function of the scatter of data points around the linear regression line and is an indication of how closely this line predicts the measured test values with a value of 0 being perfect. In theory, 68% of calibrated instrument readings will be within +/- one standard error of the real moisture value while approximately 95% of calibrated values fall between +/- two standard error values.

Please contact us with any questions regarding this report or Liebherr sensor availability and pricing.

Darrel Butler

dtb Technologies
Lakeway, Texas
darrel@dtbtech.net
www.dtbtech.net
Phone: +1, 512-426-8215