Maple Syrup Digest 2015 54(4):11-15.

Comparison of Digital Refractometers for Use by Maple Producers

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For the contemporary sugar-maker, a refractometer can be an indispensable tool for measuring the sugar concentration of sap, concentrate, and syrup. Refractometers directly measure the index of refraction – the degree to which light is bent while passing through the sample. The index of refraction is linked to the dissolved sugar concentration (density) of the solution. Measurements of sap concentration can enable the identification of particularly sweet (or non-sweet) trees, verify the sugar concentration from reverse osmosis machines and test for sugar loss from membranes, and measurements of syrup provide a quick method of determining when syrup density is acceptable.

The Brix scale is commonly used in the maple industry to represent the concentration of sugar by mass in a solution of sugar and water. Since approximately 98% of the solids dissolved in maple syrup are sugars (North American Maple Syrup Producers Manual 2006) the Brix scale can be considered interchangeable with the percentage of sugar in syrup for all but the most precise analyses. The Brix scale is measured in degrees, where one ^{0} Brix is equal to one percent sugar by mass, so that a solution of 1 ^{0}Brix has a concentration of 1 g pure sugar per 100 g solution.

Many kinds of refractometer are available for purchase, including both digital and optical varieties. Optical refractometers have a prism onto which a few drops of the sample is placed and then distributed evenly with a top cover. The measurement in ^{0}Brix is obtained by looking through the eyepiece and finding the shadow line on the Brix scale of the prism. Digital refractometers have a sample dish or well into which the sap, concentrate, or syrup is added, and at the press of a button the measurement is displayed numerically on a screen. Most digital refractometers can be used to measure the sugar concentration across the range from sap and syrup, while the majority of optical refractometers have a more limited scale, restricting use to either sap and low levels of concentrate for some, or very high levels of concentrate and syrup for others.

The purpose of the present experiment was to test the precision of a variety of digital refractometers available to maple producers. Additionally, the effect of temperature on refractometer accuracy and precision was investigated, in order to assess the reliability of the automatic temperature compensation feature now present in the majority of refractometers. Such technology is necessary because the density of solutions is affected by changing temperature, and therefore measurements of refractive index are also altered.

Methods

Multiple brands of both optical and digital refractometers were used to measure the sugar concentration of

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sap, different levels of concentrate, and syrup, in ^{0}Brix.

- 5 Palm Abbe digital handheld refractometers: PA 202 (2), PA 202x, PA 203 (2)
- 4 Pocket-Pal digital handheld refractometers: Pocket-Pal (2), Pocket-Pal 3 (2)
- 1 Milwaukee MA871 digital refractometer
- Variety of optical refractometers

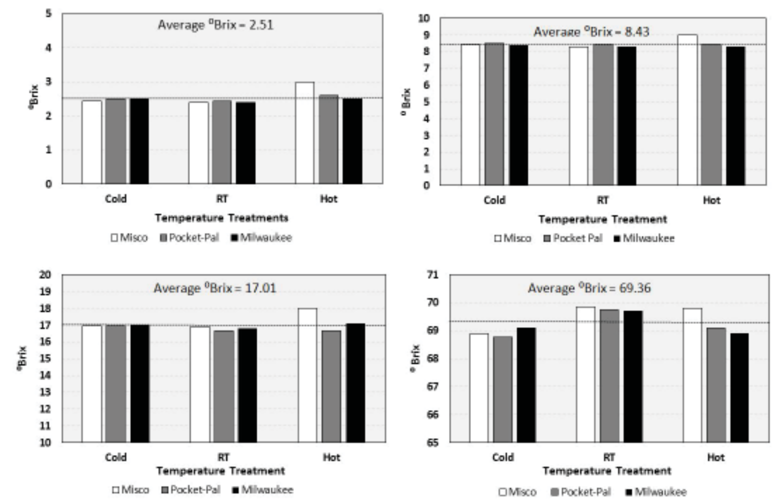
All refractometers were cleaned prior to use by lightly spraying distilled water onto the sample dish or prism surface and wiping dry with a clean absorbent cloth. Between each trial, all refractometers were cleaned by the same method. Distilled water was also used for calibration, in amounts specified by the instruction manual of each individual digital refractometer. The sample dish of digital refractometers is more difficult than the flat prism surface of optical refractometers to clean completely, and so all digital refractometers were tested with distilled water prior to each trial to ensure that no residual sugars were present in the sample dish. If a result over 0.0 ^{0} Brix was obtained, the sample dish was cleaned again and re-tested with distilled water. If distilled water measured under 0.0 ^{0} Brix, the refractometer was re-calibrated.

Sugar solutions representing various stages of concentrate and syrup produced at the PMRC were used as samples to measure refractometer precision. The sugar solutions were assembled from table sugar and sap frozen during the spring 2015 production season at PMRC and represented theoretical sugar concentrations of 2.5, 9, and 20 ^{0}Brix. Preliminary measurements with the digital refractometers placed the average sugar content of the sap at 1.56 ^{0} Brix, equivalent to 0.156 g sugar/10 g solution, or 0.468 g sugar/30 g solution. To determine how much sugar to add to the sap in order to create each concentrate solution, the theoretical sugar concentration in ^{0}Brix was converted into g sugar/30 g solution. The 0.48 g sugar/30 g solution already present in the sap was then subtracted from the total g of sugar present in the theoretical concentrate solution.

Digital and optical refractometers were used to measure the three concentrate solutions and the syrup at 0^{0}C, room temperature (approximately 20^{0}C), and 50 ^{0}C. Ice baths and hot plates were used to prepare the cold and hot trial samples respectively prior to measurement. During the room temperature trials, readings were taken within a few seconds after transferring the solution to the sample dish or prism. For the hot and cold trials, readings were taken after one minute to allow the temperature of the solution and the refractometer to equilibrate before taking the reading. All solutions were kept fully covered and sealed as much as possible between extracting samples for measurement in order to prevent evaporation.

Sample	Cold Trial SE	RT Trial SE	Hot Trial SE
2.5 °Brix	0.037	0.036	0.086
8.4 °Brix	0.075	0.086	0.130
17.0 °Brix	0.056	0.067	0.219
69.3 °Brix	0.060	0.043	0.178

Table 1: Comparison of the standard error for each sample dataset by temperature trial for all digital refractometers.



Figures 1-4

The results from the digital refractometers were analyzed primarily for precision and secondarily for accuracy, while the results from the optical refractometers were used to create a larger pool of data from which to establish a more accurate average sugar concentration for each concentrate solution and syrup.

To test the automatic temperature compensation capacity of the Misco refractometers during thermal equilibrium, additional interval trials were performed at hot and cold temperatures. For the equilibration processes of hot syrup cooling to room temperature and cold sap warming to room temperature, readings were taken with all Misco refractometers every 15 seconds for 2 minutes starting from the moment at which the solution was transferred to the sample dish.

Results and Discussion

Measurements of the concentrate solutions revealed the sugar concentration in ^{0}Brix to be slightly off from the theoretical sugar concentrations obtained by calculated addition of table sugar to the sap. Although the average experimental concentration of 2.51^0 Brix proved consistent with the theoretical sugar concentration, the solution mixed for an intended 9 ^{0}Brix had an average concentration of 8.43 ^{0}Brix. The solution with a theoretical sugar concentration of 20 ^{0}Brix had an average concentration of 17.01 ^{0}Brix. The discrepancy between the theoretical and experimental values likely occurred as a result of impurities present in the table sugar used to make the concentrate solutions. Such solid impurities would take up mass measured as pure sucrose, creating an experimental sugar concentration less than that of the theoretical. For the sample solutions representing higher levels of concentrate, more sucrose from table sugar than from sap contributed to the total sugar concentration. Therefore, the percent amount of associated impurities added to the solution along with the table sugar was likely higher for the sample solutions with higher sugar concentrations, providing a partial explanation for the increasing discrepancy between

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the theoretical and experimental sugar concentrations with increasing ^{0}Brix. This discrepancy is relevant to inquiries concerning refractometer accuracy, but is irrelevant to the analysis of refractometer precision, as the same solution was used for all refractometers.

Measurements of the same sample varied significantly based both on the temperature of the trial and on the refractometer. One interesting pattern concerning precision emerged based on the temperature of the solution. For all samples, a consistently larger standard error was observed for the hot trials than for the cold and room temperature trials (Table 1).

The relative imprecision observed for the hot trials is likely due in part to the measurements obtained from the Misco refractometers, as all are significantly over the average for each solution (Figures 1-4). These high results are produced in part by the automatic rounding which occurs when the setting on Misco refractometers is changed to measure samples with a temperature over 86 ^{0}F. Rather than reporting to a decimal place, the value in ^{0}Brix is rounded to a whole number due to the imprecision inherent within refractometer measurements of hot syrup (Misco Technical Bulletin). Additionally, this setting on the Misco refractometers was not properly set for the first round of hot trials, so it was necessary to redo these trials at the end of the experiment with the correct heat setting. Therefore, a probable source of error which could account for some of the above average readings taken by the Misco refractometers in the hot trials is the evaporation which inevitably occurred to some degree over the progression of the experiment.

The 15 second interval trial with the cold sap produced consistently high readings before stabilizing after approximately one minute. Starting with an average reading of 1.81 ^{0} Brix at 0 seconds, the readings quickly dropped within the first minute, so that after 60 seconds an average result of 1.62 ^{0}Brix was obtained. This stabilization of results indicates the point at which the solution and refractometer reached a state close to thermal equilibrium. This 0.18 ^{0}Brix decrease over a one minute period represents the error factor of the Misco automatic temperature compensation system.

Temperature	Sugar Concentration (^{0}Brix)	SE Misco	SE Other Digital
Cold	2.5	0.02	0.07
	8.4	0.04	0.15
	17.0	0.02	0.12
	69.3	0.03	0.12
RT	2.5	0.00	0.08
	8.4	0.03	0.18
	17.0	0.00	0.12
	69.3	0.02	0.08
Hot	2.5	0.00	0.11
	8.4	0.00	0.19
	17.0	0.00	0.15
	69.3	0.20	0.18

Table 2: The standard error for Misco refractometers versus the standard error of all other digital refractometers compared by temperature and sugar concentration.

The fifteen second interval trials with the hot syrup did not provide results useful towards understanding the temperature compensation capacity of the Misco refractometers, as the automatic rounding of results to whole numbers eliminated any measurable progression towards a stable reading in the realm of tenths of a ^{0}Brix.

Misco refractometers exhibited a considerably higher degree of repeatability for each trial compared to the other digital refractometers tested as indicated by the smaller standard error of the Misco dataset for each trial (Table 2).

The exception to the trend of greater precision for the Misco refractometers is evident when the results for all three temperature trials are analyzed as one dataset. Due to the automatic rounding which occurs when the Misco refractometers are set for temperatures above 86 ^{0}F, the rounded results are highly precise within a single trial, as the tenths of a ^{0}Brix values in the reading are eliminated (Table 2). However, when considered in combination with the other trials at the same ^{0} Brix value, these rounded results are consistently higher, thereby decreasing the precision of the Misco refractometers when considered overall. As discussed above, these high results may also be due in part to experimental error involving evaporation.

Conclusions and Recommendations:

- Using refractometers requires understanding how measurements should be made and following the recommendations of the instrument manufacturer in regards to calibration, care, and use. Some refractometers will work for hot syrup, others do not and may be irreparably damaged by such use.
- All refractometers produced measurements that were generally accurate; however there was some variation in the actual readings. Comparison of readings made with different instruments may produce varying results and lead to incorrect conclusions, and thus such comparisons should be eliminated or interpreted only with great care.
- While refractometers can provide some indication of the density of syrup, the actual legal definition of syrup may not include refractometry as an accepted method. Maple producers should be aware of the required procedure in their given jurisdiction.
- For more accurate and precise results, let hot or cold samples reach thermal equilibrium before measurement. For samples at freezing temperatures this can take at least a minute, as evidenced by the slight decrease in ^{0}Brix value obtained for the fifteen second interval trial over the first minute. For samples at hot temperatures at least a minute is advisable before measurement as well, as indicated by the larger standard error for all hot trials despite the minute delay to allow the sample and refractometer to reach thermal equilibrium. Care should be taken to reduce evaporation during this wait period.
- Misco refractometers are very precise, but precision is reduced for hot samples. This automatic rounding should be understood as a feature which eliminates inaccuracy in the results. More precise and accurate results can be obtained by testing the sample again when it has reached full thermal equilibrium with the refractometer.