



# FINAL REPORT 2017/003

## Evaporator Liquor Brix Sensor

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## ABSTRACT

A Pro-M-tec Ultrasonic Time of Flight (TOF) sensor was installed for trials at Victoria Mill in 2017. The TOF instrument calculates the brix of a liquid using the measurement of temperature and velocity of an ultrasonic signal through a liquid. The single sensor was located in a spool piece on the discharge line from final evaporator liquor pumps.

The prime objective was to examine the instrument's suitability for liquor brix measurement on mill evaporators. In addition, its performance with water, juice, pan products and caustic cleaning solutions would also be assessed.

Using the instrument with calibrations as supplied, tests were performed with water and sugar solutions over an extended range up to 77°Bx, and with low temperature caustic cleaning solutions. Under normal evaporator operation, stable and almost linear predictions for brix were found for liquor from 65°Bx to 74°Bx and temperatures between 55°C and 65°C. Below 50°Bx the original calibrated ultrasonic velocity response compared to a digital laboratory refractometer brix was non-linear (parabolic) and subject to large errors. Outside the 65°Bx to 74°Bx ranges for liquor, results diverged from the reference instrument, but did not experience some of the erratic responses experienced with microwave instruments. Caustic brix response was almost linear at low concentrations, but tended to be overestimated. Other solutions including aerated high grade molasses and magma appeared beyond the capability of the instrument.

Using experimental data, Wilmar developed multivariate calibration algorithms which enhanced the range and accuracy of the instrument for sugar solutions. The new equations enabled the instrument to be used to measure the brix of juice and liquor for control purposes, and provided an indicative brix to use with caustic solutions.

## EXECUTIVE SUMMARY

Evaporator brix measurement has historically been performed using a variety of in-line or at-line instruments. These included differential or static pressure brix towers, in-line conductivity, refractive index, microwave and coriolis instruments. Each one is prone to varying degrees of drift or failure due to fouling on sensors or within pipework which ultimately affects measurement and control.

Traditionally sites measured density at-line using the pressure of a liquid column of known height (brix towers which are partially open). Brix towers in particular are vulnerable to lag and interruptions in flow of product to the liquid column from evaporator vessels. They possess an inherently increased risk of spillage and exposure for operators to hazardous product, particularly during chemical cleans.

Australian sugar mills have sought alternatives or supplements to brix towers with varying degrees of success. Many of the instruments were vulnerable to fouling and unpredictable shifts in calibration and loss of accuracy. Each alternative instrument has required a level of intervention to manage calibration and inherent technical issues.

Initially, microwave instruments appeared to be a fairly economical and robust option whilst operating in “normal” operating ranges. However, over time they were found to cause erratic and sometimes catastrophic control outcomes. Wilmar had been conducting trials on the application of a microwave device for evaporator brix control, but encountered operational issues with this technology. Protracted investigation found that the instruments were subject to signals that misrepresented product (juice/liquor) density information, sometimes reversing the expected controller action and leading to loss of control.

This project assessed the suitability of a Pro-M-tec us-ICC1500 Time of Flight (TOF) ultrasonic instrument to measure brix. It was installed on a liquor product pump discharge line from one of the final effect evaporator vessels at Victoria Mill. The instrument uses velocity and temperature measurement information to calculate a brix value which can be used by the factory control system. A spool piece was installed allowing the TOF sensor to be inserted as it would be for normal in-line installations. It was fitted with sampling points on the top and bottom flange, and an extraction line which ran under vacuum back into the final evaporator vessel. This enabled liquids to be tested under both normal in-line operation for liquor between 66°Bx and 74°Bx and at-line for the addition of various “synthetic” solutions.

The performance of the TOF instrument was examined primarily in-line providing continuous measurement. Samples were taken from the spool piece for measurements with a digital laboratory refractometer in the field as a reference. Device responses were examined for water and a wider range of concentrations of liquor during normal operation. Other solutions such as diluted liquor and juice were manually introduced to the spool piece to allow temperature influences to be tested. Caustic-based cleaning solutions were tested on line and/or introduced manually to the spool piece. The influence of fouling, crystal and air, and the effectiveness of temperature compensation algorithms within the instrument were also examined.

Under normal evaporator operation and for brixes between 60°Bx and 77°Bx, the instrument as-supplied performed reliably for temperatures between 55°C and 65°C. Outside these ranges for liquor, results diverged from the reference instrument, but did not create erratic responses. The brix response for caustic solutions was almost linear, but offset from their true value. Other solutions

including aerated high grade molasses and solutions with high crystal content appeared beyond the capability of the instrument.

Observing that outputs from the supplied instrument had inherent limitations, Wilmar developed multivariate calibration algorithms which enhanced the range and accuracy for various sugar solution concentrations. The limitations due to air and crystal persisted but instrument outputs could now be used to more accurately measure the brix of water, juice and liquor, and to monitor caustic solutions during chemical cleans. The new algorithms could be applied to instrument outputs within a factory distributed control system (DCS) or PLC for liquor product and potentially intermediate streams through evaporators.

Whilst the new algorithm could reliably predict brix with a standard error of 0.59°Bx on the original velocity and temperature dataset, changes to the laboratory instruments and additional testing with hydrometers indicated that the calibration data needed to be updated and overwritten to improve the accuracy of predictions. With both the existing multivariate calibration coefficients and the addition of new data, good brix predictions and control could still be achieved.

With a firmware upgrade and Modbus communications, the instrument has the capability to provide several outputs for the purposes of measurement and control. Although it is still unknown how transferrable the calibrations would be, it is expected that since the raw measurements are for physical characteristics including temperature and the velocity of sound in a liquid, that the Australian Sugar Milling and refining industry would be able to apply or adapt the algorithms developed in this project to their own site applications.

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# 1. BACKGROUND

## 1.1. General Issues

Evaporator brix measurement has historically been performed using a variety of in-line or at-line instruments. These instruments included differential or static pressure brix towers, in-line conductivity, refractive index, microwave and coriolis. Each of the instruments are prone to varying degrees of drift or failure due to fouling on sensors or within pipework which ultimately affects measurement and control.

Traditionally sites measured density at-line using the pressure of a liquid column of known height (brix towers). Brix towers in particular are vulnerable to lag and interruptions in flow of product from evaporator vessels reaching the liquid column. This can occur at any time, but typically during or immediately after chemical cleaning cycles. Variations in sample product flow rate due to fouling or pressure variation in the long pipe runs to brix towers can also affect liquid column height. Product flow issues have frequently been reported to affect the accuracy of measurement and the response times for automatic controller actions. Brix towers are usually accompanied with lengthy pipe runs and are open to atmosphere allowing the ingress of air to vessels under vacuum. They possess an inherently increased risk of spillage and exposure for operators to hazardous product, particularly during chemical cleans. Many sites have sought alternatives or supplements to brix towers with varying degrees of success, with the cheapest and most common being conductivity. Each alternative instrument has required a level of intervention to manage calibration and inherent technical issues.

### 1.1.1. Concerns with Updated Microwave Technology

Initially, microwave instruments appeared to be a fairly robust solution to many of these issues, whilst operating in a “normal” operating range. However, over time they were found to cause erratic control outcomes, even within supposedly normal operating ranges, and particularly dramatic when operating near calibration limits. Wilmar had been conducting trials on the application of a microwave density measurement device for evaporator brix control, but encountered operational issues related to this technology. Protracted investigation found that the instruments were subject to phase shift and inversion which essentially generated signals that misrepresented product (juice/liquor) density information leading to loss of control.

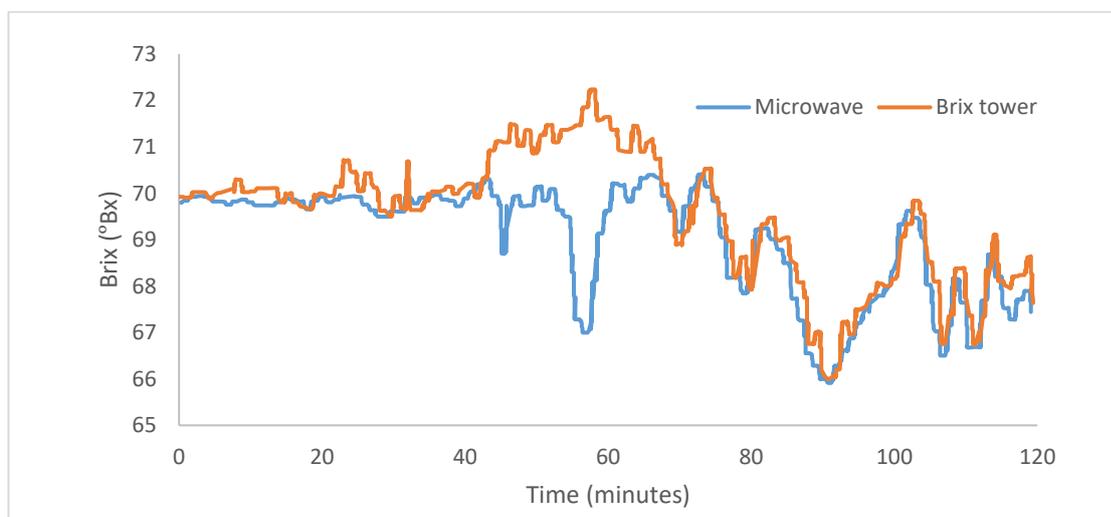
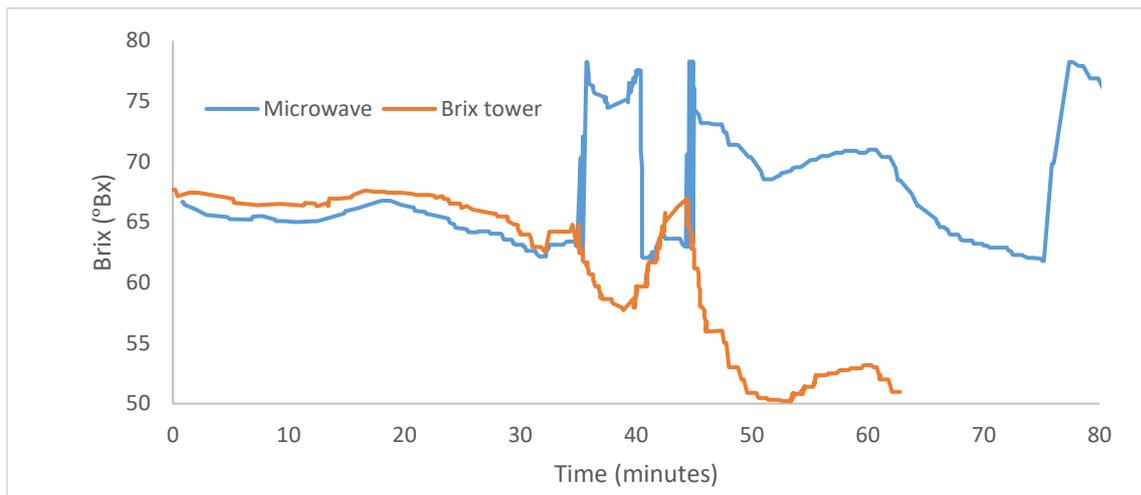


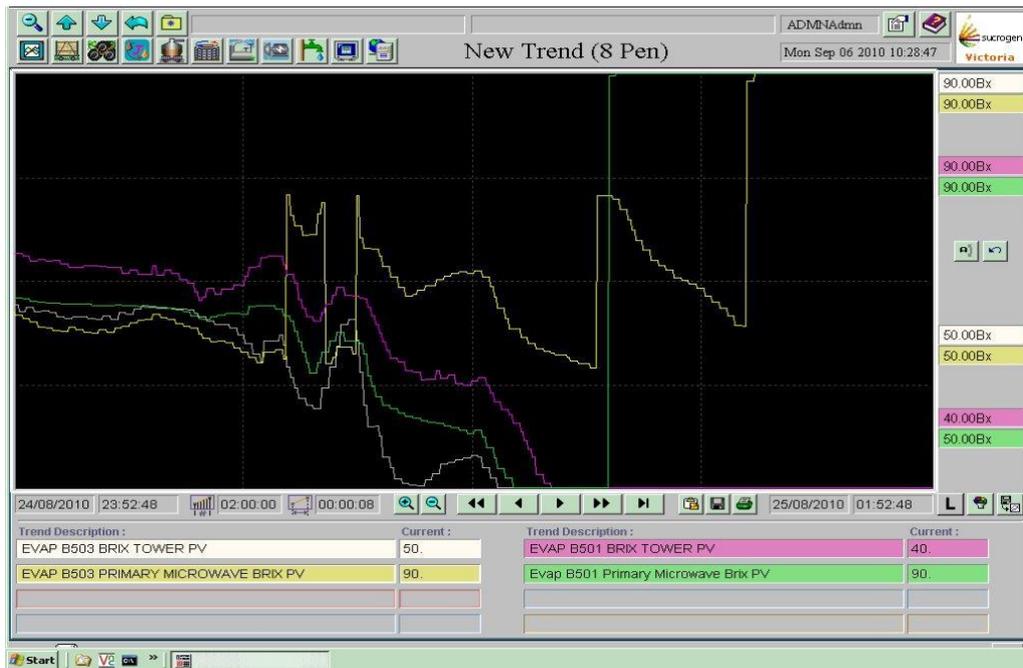
Figure 1 - Example of inversion phenomena at high brix with microwave sensor

The original microwave unit was installed in a DN100 spool with “frogs-eye” sensors approximately 75 mm apart and a calibrated range of 40 to 80°Bx. This hardware arrangement and signal evaluation produced a “phase inversion” phenomenon at approximately 69°Bx. Initially, this would create some divergence between readings from the brix tower and microwave shown in Fig. 1, until it transitioned to an inverted output. The brix signal would perfectly “mirror” the trend in the opposite direction to the actual brix rather than tracking it. The inverted output would then result in a control response completely opposite to that needed or expected to control density in a final evaporator vessel. Various remedies were sought to overcome this issue, including boosting signal amplification and adjusting the distance between sensors. In attempt to address all potential causes, it was thought even aeration may have potentially affected performance, so pipe work was rearranged and non-return valves repositioned but with no improvement. Insertion probes (used in Pans) were trialled which gave greater range and did not produce phase inversion but were found to be too susceptible to fouling.



**Figure 2 - Example of phase shift jumps with microwave sensor**

Following the installation of DN150 spools, the increased sensor distance halved the span, but additional hardware and signal amplification was required to maintain attenuation levels. Part of the inversion phenomenon at 69°Bx was overcome, but with the automatic phase correction switched “off”, phase jumping (Fig. 2) occurred more frequently over the shorter calibration range and proved difficult to mask. From previous work, this is dramatically illustrated from the Distributed Control System (DCS) in Fig. 3. Oscillations between the maximum and minimum brix span on either side of the target set-point would then occur continually with a corresponding response from the control system.



**Figure 3 - Example of phase shift jumps with microwave sensor on the DCS**

With automatic phase correction switched “on”, the evaluation unit would read beyond the period of a single sine wave and partially overcame the inversion issue. However if the spool ran empty then the unit could not find its way back to the original stand or phase cycle. The unit would continue to self-correct whilst outside the original range, requiring intervention by the instrument technician to restore readings to the correct part of the operating range once the evaporator was back on line.

### 1.1.2. Examining TOF as an Alternative Technology

The principles of the Time-Of-Flight (TOF) ultrasonic instrument offered an alternative and simpler approach. The performance of the instrument under similar conditions needed to be evaluated to ensure stable and repeatable results throughout the operating range. Apart from laboratory work by the instrument supplier in Germany, there had been no formal Australian trials performed on behalf of the industry.

Pro-M-tec manufacture a Time of Flight ultrasonic density sensor and have performed laboratory trials with their instrument and preliminary trials to measure juice concentration. In 2015 they loaned an instrument to Wilmar as an alternative to their microwave technology, and tests were conducted over a brief operating range. Further investigation in an industrial application was warranted. There was virtually no application data available that showed how these instruments would perform in cane sugar juice evaporators. Furthermore, measuring cane juice and liquor close to saturation and where the entrainment of air was possible posed operational risks for plant and process. The stability of such an instrument over a wide operational range and chemical cleans needed to be understood before the sugar industry could safely apply the technology to this application.

## 2. PROJECT OBJECTIVES

### 2.1. Broad Project Objectives

The mission was to:

Demonstrate that Ultrasonic TOF devices could operate successfully across the full range of (sugar) cane juice & liquor products, chemicals and water which are processed through cane sugar mill evaporators

Demonstrate that Brix could be reliably measured in the range 20 to 80°Bx (and establish lower and upper limits) through normal operation and staged calibration experiments including adverse conditions outside the normal operating envelope (down to 0°Bx)

Evaluate the impact of fouling on the instrument sensor between fortnightly evaporator cleaning cycles to identify if it introduced bias, drift or loss of sensitivity and to assess instrument recovery from these conditions.

#### 2.1.1. Key Project Objectives

The key objectives were to:

- a. Observe periods of normal operation to produce syrup at 66 to 74°Bx with TOF instrument as part of the control loop during the 2017 crushing season.
- b. Assess the impact of fouling during normal operating cycles between cleans and extended cycles where the instrument itself is not cleaned.
- c. Observe response during chemical clean cycles at fortnightly intervals throughout the 2017 season
- d. Confirm the true operating range of this device and relationship to physical properties (density) of caustic, acids (EDTA), liquor, water and juice
- e. Observe the impact of air inducted into the evaporator product, both on line and under laboratory conditions. Determine if a condition may exist while operating which may produce an output signal which could cause chaotic control responses
- f. Observe the impact of crystal present in the evaporator product, both on line and under laboratory conditions and test the device in a massecuite vacuum pan. Determine if a condition may exist while operating which may produce an output signal which could cause chaotic or undesired control responses.
- g. Confirm that temperature compensation is accurate and effective under each of the conditions being examined.
- h. Confirm that the device is sufficiently robust to withstand the range of mill operating conditions including temperature and vibration.

## 3. OUTPUTS, OUTCOMES AND IMPLICATIONS

### 3.1. Outputs

During the 2017 season, velocity and brix curves for sugar solutions and condensate were generated to record bare instrument responses covering 0 to 77°Bx for temperatures between 29 and 78°C. Plots for dilute cooled evaporator cleaning solutions of sodium hydroxide were also produced for a

typical usage range up to 12.1 w/w%. The calibration plots showed that the bare instrument relied on linear calibrations and temperature corrections which at best could only successfully predict brix for liquor in the range 60 to 74°Bx with temperatures between 55 and 65°C. Within these ranges, the error of predictions was generally within  $\pm 0.7^\circ\text{Bx}$ .

The bare instrument showed it had limitations for predicting the brix of sodium hydroxide (unless a constant factor was applied to brix results). Due to the simplicity of the native instrument calibrations, low temperature liquor and all diluted liquor and juice solutions displayed errors of prediction. The highest error was with condensate, where the instrument would indicate 29°Bx.

The temperature compensation in the native calibrations applied internally by the instrument to raw velocity figures enabled good prediction of liquor brix during normal evaporator operation, but magnified the errors associated with low brix and low temperature solutions.

In brief, initial testing demonstrated that the instrument was not suitable for predicting brix in massecuite, or heavily aerated liquors or syrups and high grade molasses due to the apparent loss of detection of a velocity signal. This made the instrument unsuitable for measuring brix of magma, massecuite in pans and syrup/molasses from fugal dilution tanks.

In 2018, after the initial tests, more sophisticated calibration equations were generated and reapplied to raw data to better predict the brixes of solutions already tested taking into account the “raw” time of flight velocity and temperature. The new calibration was a 3rd order surface equation that was tested off line and then programmed into the factory distributed control system to predict the brix of solutions “in-line”. The improved calibration successfully predicted the brix of liquor and diluted sugar solutions, including condensate with a standard deviation of 0.59°Bx. A separate equation was developed for sodium hydroxide, which was used to re-predict the original test solution brixes, but subsequently only tested at line on known cooled pure sodium hydroxide solutions.

Due to limitations with the native calibrations, there was insufficient data to quantify the impact of soft-scale fouling during normal operation, but testing showed no clear trends or greater bias between evaporator cleaning intermissions. Testing with the improved calibrations did reveal that immediately prior to cleaning intermissions when liquor was being washed from evaporator vessels, that the prediction for the brix of water was overestimated by approximately 2 units. After vessels were cleaned with caustic, the prediction for water brix decreased to 0.3°Bx.

The Pro-M-tec instrument and evaluation unit appeared to operate well in the field over the two seasons where it was subjected to ambient conditions up to 50°C and normal factory and evaporator operating conditions. The sensor cost is currently around \$18k and the separate evaluation unit which is required to operate with it sells for around \$10k (subject to Euro exchange rates). The stainless steel spool piece, electrical work and cabling are additional to this.

### 3.2. Outcomes and Implications

The instrument as tested only delivered a single analogue output which was “brix”. The evaluation unit display however showed brix, temperature, raw and corrected velocity. The updated calibration equations applied externally to the instrument, required a separate temperature input and used the “brix” output from the instrument. Updates to the Pro-M-tec instrument firmware and hardware reportedly now provide velocity, time of flight and other outputs which could be processed externally by a distributed control system or PLC using the form of the equations developed in this project to provide the more accurate brix predictions.

Note that due to limited testing after the new equations were loaded, that the surface equation coefficients still require some further updating before being used by other Australian Mills. At this

stage the instrument is only installed at Victoria Mill and provides sufficiently accurate results to be used for control purposes.

The form of the equations and current coefficients can be made available to Australian Mills from Wilmar Australia, and are not available from Pro-M-tec. There are two forms to the new equations where they are separately applied to either sugar solutions or caustic cleaning solutions. At the time of writing, the sugar equations were more robust and reliable than the initial caustic equations which remain purely indicative. A factory control system or PLC would still need to be programmed to identify and communicate a mode of operation to ensure the correct equations are being applied for their intended purpose. The instrument supplier has continued to develop the technology and sensor head for installations in Europe.

Results from this project suggest the following industry value benefits could realistically be achieved:

- a. Elimination of brix towers and associated flow and level measurement devices. Reduction in maintenance to fouled instruments or vessels or operating costs where loss of control had occurred. For a site this was estimated at \$10.5k p.a.
- b. Eliminate the need for the extensive at-line pipework and the incidence of induced air into return liquor streams under vacuum which periodically lead to loss of control and potentially sugar-up evaporators. A 1 in 20 year event for at least one mill leading to a 24 hour stop \$4.8k p.a.
- c. Reduced operating costs by increasing consistency of liquor brix and reducing LP steam requirements in pan stage operations through tighter liquor density control and potentially reducing pan boiling times with higher liquor brix.
- d. Improved operator safety by eliminating open elements (brix towers) which have been used for density measurement during chemical cleans. This also provides improved environmental risk by minimising the potential loss of product (caustic or sugar) from the process

## 4. INDUSTRY COMMUNICATION AND ENGAGEMENT

### 4.1. Industry engagement during course of project

Pro-M-tec had been working on updates to their instrument and developing Modbus firmware towards an upgrade. To facilitate an outcome to this project, the chief investigator decided to pursue updated calibrations in-house and to have them programmed into the distributed control system late in the 2018 crushing season.

As most of the testing was done exclusively at Victoria Mill for the development work, there was no communication with the remainder of the industry. There was only informal communication with production and technical management within Wilmar Australia sugar mills. A paper has been written for the 2019 ASSCT conference reporting the overall findings and generic nature of the updated calibration equations. Information on the equations in their current form would be available to Australian Sugar mills only from key contacts within Wilmar Sugar Australia. At the time of reporting, the form of the equations would potentially be accessible via the ASSCT paper, but the coefficients to-date would be of limited use in a commercial product. The paper will be presented in May 2019.

### 4.2. Industry communication messages

Under normal evaporator operation and for brixes of 60-77°Bx, the supplied instrument performed reliably and accurately at temperatures of 55-65°C. Outside these ranges for liquor, results progressively diverged from the reference instrument and did not create erratic responses as had been found with microwave devices. Responses for sodium hydroxide and EDTA were almost linear

but tended to overestimate brix. Other solutions including aerated high-grade molasses and magma appear beyond the capability of the instrument.

The multivariate calibration algorithms that enhanced the instrument range and accuracy provided predictions with a standard deviation of 0.59°Bx over the ranges tested. As the multivariate algorithms were unable to be loaded into the DCS until October 2018, subsequent tests on the multivariate equations were limited. During the course of testing towards the end of 2018, a difference between the refractometer readings when used in the field and readings conducted on cooled samples in the laboratory indicated that the reference methods were subject to an offset of about 1.2 units. This did not detract from the ability of the instrument to be used for monitoring and control purposes, but flagged an opportunity to improve the accuracy of predicted brix. Further testing would be required to hone the algorithm coefficients derived from the original data set.

The results from this fitting process showed that the polynomial resulting from the fitting process matched the measured data well across the entire range of the trial data. It also appeared to work well even when extrapolated into regions where there was no data available showing that the multivariate calibration algorithms were sufficiently robust and an improvement on the supplied instrument.

The time-of-flight ultrasonic technology using the multivariate calibration equations proved to be a viable alternative to other current technologies to use for measuring final evaporator brix and approximating caustic cleaning solution brix. The calibrations were shown to hold for water and sugar solutions up to 77°Bx containing minimal air and negligible crystal content. The calibrations would potentially make the technology suitable for evaporators further upstream, providing product was suitably degassed.

## 5. METHODOLOGY

### 5.1. Time of Flight Installation

A Time of Flight (TOF) ultrasonic instrument was installed on a branch line from one final effect evaporator vessel at Victoria Mill. The instrument uses velocity and temperature measurement information to calculate brix and generate a single analogue output. This output could then be used in the factory control system to monitor and control liquor brix. The branch line was assembled with a spool piece which allowed the sensor to be inserted as it would be for normal in-line installations. In addition, the spool piece was fitted with sampling points on both the top and bottom flanges and an extraction line which ran back to the final evaporator vessel. This enabled testing under both normal in-line operation for liquor between 66°Bx and 74°Bx and at-line for various “synthetic” solutions.

The performance of the TOF instrument was examined primarily “in-line” providing continuous measurement. Samples were taken from the spool piece for measurements in the field originally with an Atago hand held refractometer as a reference, and subsequently with a Bellingham Stanley OPTi digital laboratory refractometer, high temperature and standard brix spindle hydrometers. Device responses were examined for water and a wider range of concentrations of liquor during normal operation. Other solutions such as diluted liquor and juice were manually introduced to the spool piece to allow temperature influences to be tested. Caustic-based cleaning solutions were tested both in line and introduced manually to the spool piece. The influence of fouling, crystal and air, and the effectiveness of temperature compensation algorithms within the instrument were also examined.

### 5.1.1. Equipment

The ultrasonic sensor purchased for this trial (Fig. 4) was inserted into a modified spool piece which had previously been used for microwave probes. Wilmar's existing microwave evaluation unit was adapted for use with only time of flight velocities and temperature, as data processing no longer required phase shift or signal amplification corrections. The instrument would measure temperature and the velocity of an ultrasonic signal in the liquid between the sensor prongs which housed both the transmitter and receiver.

### 5.1.2. Sensor Probe



Figure 4 - Ultrasonic sensor probe

The ultrasonic Time-of-Flight sensor was installed in a spool-piece immediately downstream from the final liquor pump. Figure 4 shows the probe that was inserted through a 75 mm stub and sits in the main product stream which recycles back to the final effect evaporator vessel.

### 5.1.3. Spool Piece Configuration



Figure 5 - Instrument and in-line spool-piece

The spool-piece adapted from the previous microwave installation was installed with tapping points to enable direct measurement and sampling of synthetic and atypical process products for the purpose of this trial work. Material could be pumped into the base of the spool piece from the liquor pump for in-line measurement and control. Alternatively, the spool-piece in this installation could remain in position but taken off-line with the top flange removed to enable various products to be manually poured in, tested, drained and washed clear. Whilst the TOF unit already contained a temperature sensor to feed into its own internal temperature compensation algorithm, an additional temperature sensor (RTD) needed to be installed to provide an independent signal to the DCS. For the later trial work, this RTD occupied one sampling point on the top flange.

## 5.2. Testing Methodology

### 5.2.1. Testing Solutions with Calibrations as Supplied

To confirm the operating range of the device and relationship to the physical properties of liquor, juice, water, caustic and EDTA, tests were performed in the field primarily with the Atago portable digital refractometer as a reference instrument. Samples were extracted directly from the instrument spool-piece downstream from the final evaporator vessel liquor pump to compare with instrument readings taken direct from the evaluation unit and the DCS. Table 1 shows the seven testing frameworks that were used to examine the effects of normal and more extreme operating conditions for fluid density and temperature. Apart from liquor (operating on-line) and one of the caustic cleaning solutions, all other test solutions listed in Table 1 were prepared externally and manually introduced to the spool piece “at-line”.

To ensure thorough mixing of synthetic test solutions, they were cycled through the spool piece. The initial installation only allowed for brix to be transmitted to the DCS, so readings for temperature, brix, raw velocity and corrected velocities were taken directly from the evaluation unit display wired into an adjacent cabinet. Later in the project, the values were able to be extracted from the DCS. Measurements were taken for fixed concentration solutions and all readings were manually taken as the test solutions cooled towards ambient temperature. In some cases, test solutions remained in the spool piece overnight to capture the lower temperature series. The solutions were resampled for measurement on laboratory instruments when subsequently drained.

Sodium hydroxide and EDTA solutions were each taken from stock for testing. A 50% Sodium hydroxide solution was diluted by 50% and 75% and 87.5% to provide 3 litre test solutions. The test solutions were then checked on the laboratory refractometer to determine a concentration. The process of dilution did generate heat, so each test solution required a short time to stabilise after being introduced to the spool piece before taking readings. 39% EDTA was diluted and mixed and tested in a similar fashion.

For initial testing and calibration, working solutions of caustic were also taken. The brix and strength of each of the solution was established through laboratory analysis of brix by refractometer and by titration. The working solution was then progressively diluted to provide a series of test solutions for which TOF velocity and brix responses were recorded after being manually introduced to the spool piece.

To test the impact of fouling, sampling was conducted periodically over a 1 month period in the season starting the week before a scheduled (fortnightly) clean, between the cleans, and then for one week following the scheduled clean

**Table 1 - Operational and Synthetic Test Solutions**

Material	Brix °Bx	Temperature °C
1. Liquor (operating on line)	64 to 77	57 to 64
2. Liquor (including diluted samples)	26, 38, 40, 46, 51, 56, 60, 66, 72	33 to 72
3. Condensate	0	39 to 78
4. Evaporator Supply Juice	10	29 to 76
5. Magma and Massecuite	86 to 92% (dry substance)	56
6. High Grade Molasses	70 – 75% (dry substance)	66
7. Sodium Hydroxide	8 to 30 (2.3 to 12.1wt% working strength)	20 - 23

### 5.3. Determining the operating range and limitations of the instrument

Measurements were done with extended temperature profiles on water, ESJ and the different liquor dilutions cited in Table 1. ESJ was taken directly from the ESJ tank, and although fairly dilute and lower brix than normal, it was nonetheless suitable for testing. ESJ was poured into the spool piece, which was sealed and isolated. As the ESJ cooled, the instrument parameters were recorded manually. As changes were initially quite rapid, the instrument display was photographed to capture relevant figures. Once the ESJ had cooled to 39°C, the spool piece was drained and the solution tested on the laboratory refractometer. Clean condensate was then used to clean the internals of the spool piece, and then used as the next test solution. Instrument velocities, brix and temperatures were again monitored as it cooled. For initial testing, liquor was then diluted to produce a series of test solutions each of which differed nominally by 5°Bx. Each solution in turn was cooled and the instrument parameters recorded. Where possible, specific temperatures were targeted for each test solution to generate a series of isothermal curves. Isotherms for nominal 5 or 10°C ranges were generated. Equations and regression coefficients for each were determined and presented in Fig. 6 and Fig. 7.

#### 5.3.1. Liquor between 64 and 77°Bx – Normal on-line operation

Tests were performed over several days moving brix controller set-points through standard and extended operating ranges. During tests, evaporator boiling rates and vacuum varied, which led to differences in liquor temperature. Typical operating ranges between 64°Bx and 77°Bx were observed. Subsequently, the evaporator brix was increased beyond normal operating range, briefly to a range of target set-points up to 78°Bx. Given the long residence time in final vessels and relatively slow process response at this level, the set-point was lowered to avoid the possibility of establishing further crystal and “sugaring up” the final vessel.

#### 5.3.2. Liquor and Synthetic Juice between 26 and 72°Bx – At-line Operation

To examine lower brix solutions the spool piece configuration was taken off line to allow for all remaining test solutions to be assessed using the methods described above in section 5.3.

After initial testing and when the new algorithms had been loaded into the distributed control system, the methodology for subsequent testing used a new Bellingham Stanley refractometer and suitable brix spindles as a second point of laboratory reference. A high temperature (60°C) brix

spindle was used in the field for liquors between 60 and 80°Bx, but all other tests were performed after allowing samples to cool to laboratory temperatures before testing solution brixes with the laboratory digital refractometer.

### **5.3.3. Synthetic Solutions containing Crystal – At-line Operation**

To examine high brix solutions the spool piece configuration was taken off line and used “at-line” using the same technique as described in 5.2.1. Magma and massecuite having such high solids contents were simply poured into the spool piece and could not be repeatedly cycled through. Readings were taken shortly after the spool-piece had been filled with product.

### **5.3.4. Synthetic Solutions of aerated Molasses – At-line Operation**

B Molasses samples were taken from the fugal dilution tanks and transferred manually to the spool piece for testing. They were cycled once through the spool piece to establish a good mixture. The key characteristic of the molasses samples was that they were heavily aerated at the time of sampling and would retain much of that air during the tests.

## 6. RESULTS AND DISCUSSION

### 6.1. Determining the operating range of the instrument

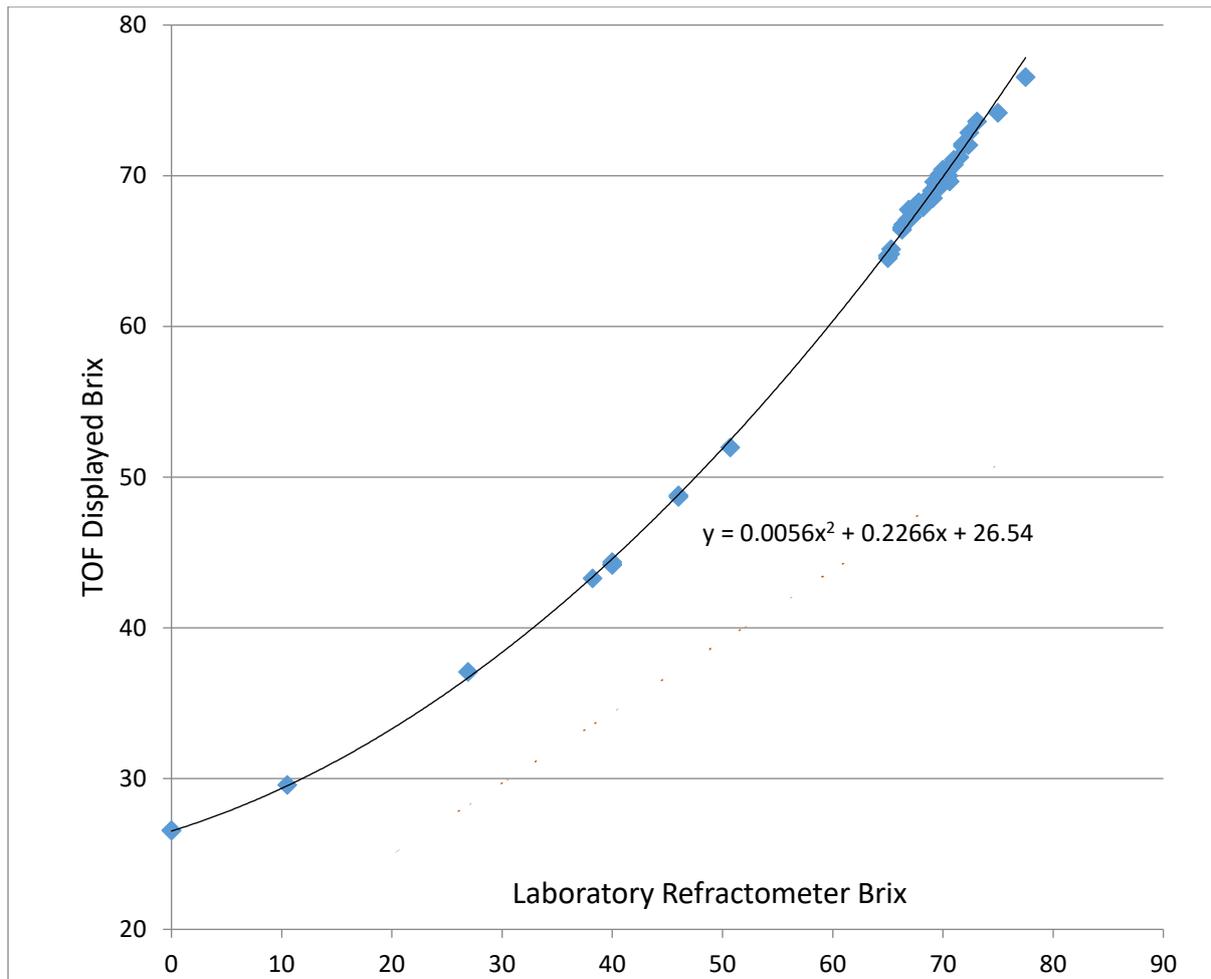
#### RESULTS

##### **Liquor and Diluted Sugar Solutions – Initial Calibration Profiles**

Measurements were done with extended temperature profiles on water, ESJ and the different liquor dilutions cited in Table 1. Where possible, specific temperatures were targeted for each test solution to generate a series of isothermal curves. Fig. 6 shows the native brix data for liquor and diluted sugar solutions with temperatures targeted at  $60^{\circ}\text{C} \pm 1^{\circ}\text{C}$ . The standard deviation of the errors for predicted brix versus the curve of best fit was  $0.42^{\circ}\text{Bx}$ . To help illustrate the limitations of the instrument at low brix and a broader range of temperatures, isotherms for nominal 5 or  $10^{\circ}\text{C}$  ranges were generated, and Fig. 7 and Fig. 8 show the response curves for velocity measurements. An attempt was then made to determine suitable equations to fit each of the isotherms.

##### **Liquor between 66 and $74^{\circ}\text{Bx}$ – Normal operation**

Despite the changes in temperature, stable signals and control within the process for typical operating ranges between  $64^{\circ}\text{Bx}$  and  $77^{\circ}\text{Bx}$  were observed. As evaporator brix was increased beyond normal operating range, instrument readings continued to track actual brix within  $1^{\circ}\text{Bx}$  at the top end of the range. At this higher brix however, liquor started to become turbid and highly viscous which indicated the presence of fine crystal, however operationally the system remained in control and no erratic responses were evident.



**Figure 6 - Native Calibration data for defined temperature range. TOF Brix at 60°C ±1°C with Initial Velocity Temperature Compensation Referenced to 65°C**

The simple internal calibration equations followed the following steps to calculate brix:

$$V_{comp} = V_{raw} + (T_{act} - T_{ref})$$

$$Brix_{disp} = A_0 + A_1 * V_{comp}$$

Where

$A_0$  and  $A_1$  are calibration constants

$T_{act}$  = Measured Temperature

$T_{ref}$  = Instrument Reference Temperature (constant) = 65°C

$V_{raw}$  = Measured Velocity (m/s)

$V_{comp}$  = Compensated Velocity (m/s)

The internal calibration constants can be updated when the instrument is installed based on sample data during normal operation if required. Given the simplicity of the temperature compensation, it is not surprising that errors were found to increase as temperatures decreased below the reference temperature. Hence there were native limitations to the operating range of the original instrument.

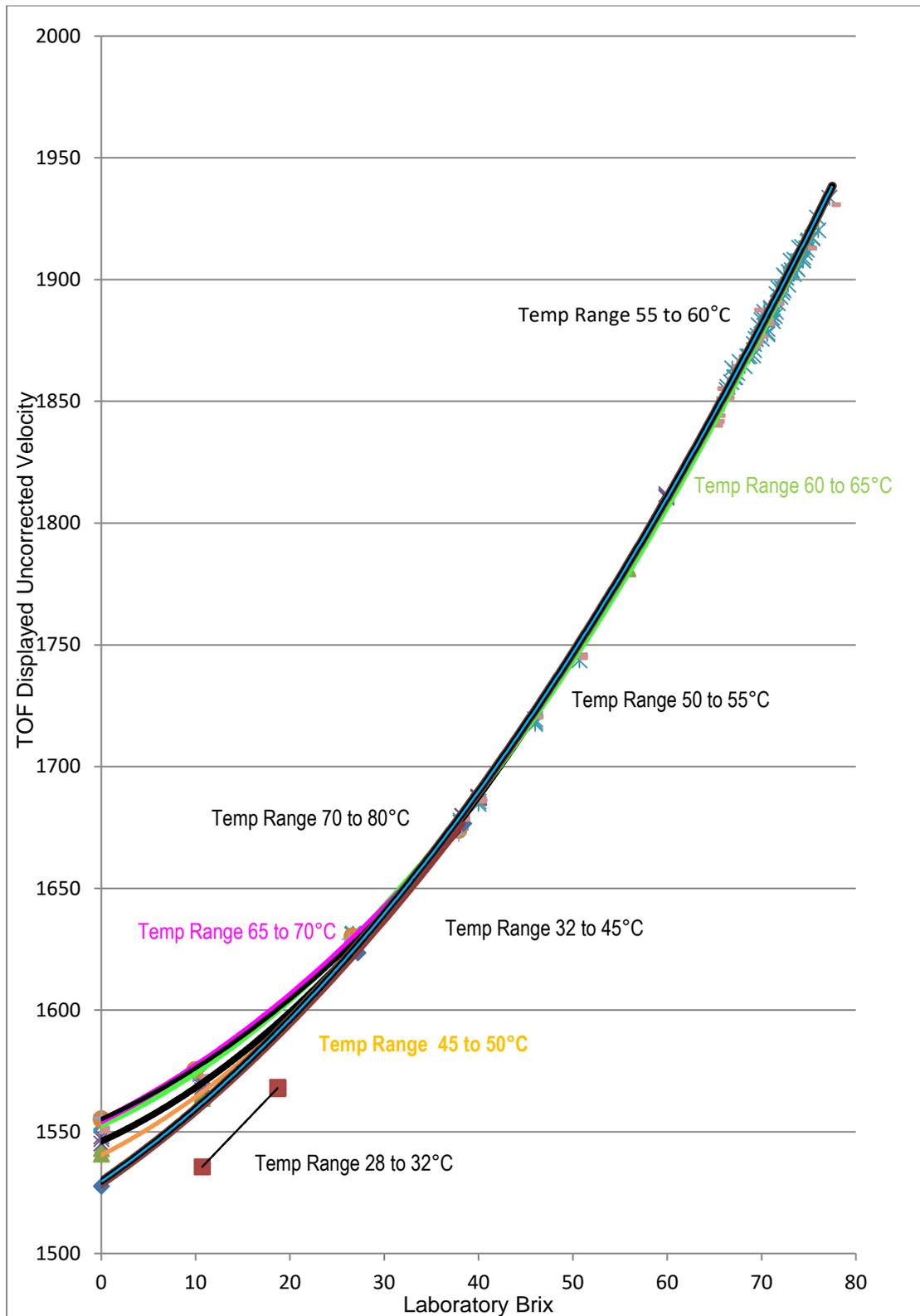


Figure 7 - Uncorrected velocity m/s (no temperature compensation) versus laboratory refractometer brix.

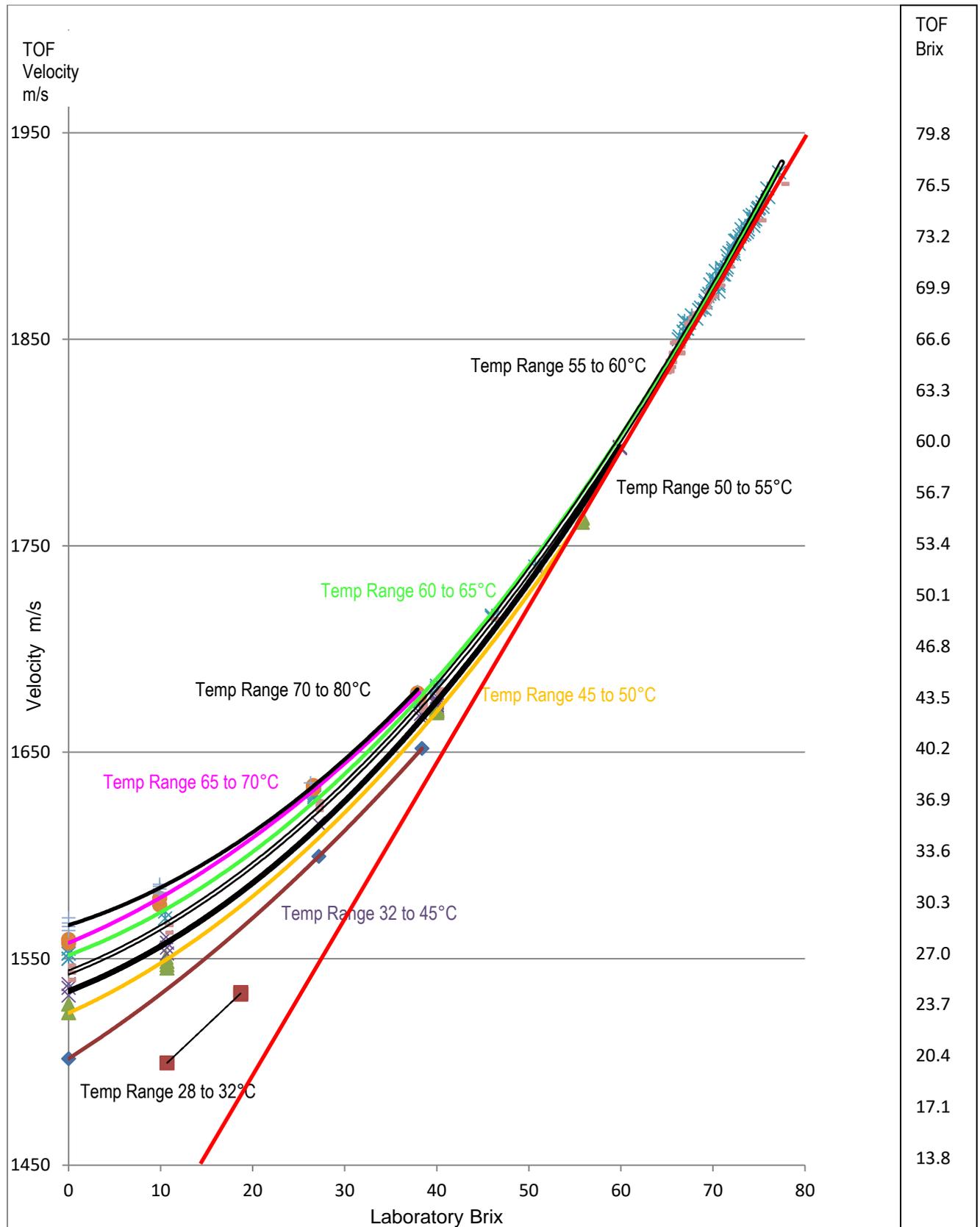


Figure 8 - Sugar solutions – TOF Brix and velocity based on a temperature-corrected velocity referenced to a temperature of 65°C.

The bare instrument provided good performance with liquor under normal operating conditions. However, with low brix solutions it was evident that errors were magnified where temperatures were significantly higher or lower than 65°C. Each sugar solution followed a parabolic velocity response profile which the instrument subsequently converted to brix using a linear calibration using a reference temperature of 65°C. Larger deviations were observed as the test solutions cooled and the internal temperature correction magnified the error in calculating velocity. This was less evident for higher brix solutions, but particularly apparent with low brix solutions. The instrument's simple temperature correction to velocity clearly required a more sophisticated approach, and an updated calibration to cope with these conditions.

Fig. 8 shows how these velocities were interpreted to corresponding brix and demonstrated the shortcomings of the instrument as liquors and juices were presented outside the normal operating range, particularly at lower brix. The error between calculated and measured brix was of the order of 10% around 40°Bx. The "y = x" line (in red) indicates the ideal simple linear relationship for TOF brix = refractometer brix to highlight the deviation if it were to apply for all temperatures.

## 6.2. Sensor Capability – Testing Technology Limitations

Having tested the performance of the bare instrument under normal and extended operating conditions, the project also targeted the situations which historically have compromised the performance of previous instruments or might pose a challenge for this technology.

### 6.2.1. Limitations with air

- (a) *Observe the impact of air inducted into the evaporator product, both on line and under laboratory conditions. Determine if a condition may exist while operating which may produce an output signal which could cause chaotic control responses.*

Qualitative tests were conducted with B molasses with a dry substance content of 74%. Initially it was thought that the samples could not be read, however since the molasses samples were taken from the fugal molasses dilution tanks, they were severely aerated and on testing took almost two hours for the entrained gases to be released sufficiently to give a velocity reading. The actual reading 437 m/s was well below the velocity range of 1500 to 1930 m/s used for water, juice and liquor. In-line measurement for this application would be problematic on a fugal station where there is considerable aeration and emulsification in the tank and pump. When liquor, juice and water samples were tested and cycled through the spool piece, velocity and brix readings appeared to be quite stable and the low velocity phenomenon was not observed to occur.

### 6.2.2. Limitations with crystal content

- (b) *Observe the impact of crystal present in the evaporator product, both on line and under laboratory conditions and test the device in a massecuite vacuum pan. Determine if a condition may exist while operating which may produce an output signal which could cause chaotic or undesired control responses.*

To simulate potential conditions in a vacuum pan, the device was tested to determine if a condition could be created while operating that may produce an output signal that could cause chaotic or undesired control responses. The initial test with liquor brix being steadily increased under controlled conditions led to supersaturation and spontaneous nucleation. The initial test with liquor which was undergoing spontaneous nucleation indicated that crystal *does* interfere with the measurement when the TOF probe becomes coated with crystal. The brix was measured at just below 78°Bx, and this was considered high enough for normal operation. The reading however did not suffer inversion or phase shift (as seen with microwave) and subsequent control action is not compromised. Controlled operation was maintained while the final vessels ran to purge heavy liquor

as they were diluted with lower brix feed. Given the lengthy purge time, however, it was not considered prudent to repeat the scenario as the liquor pumps were already operating at full capacity and there was a risk that the final vessels could fail to purge.

Diluted magma slurry was introduced to the spool piece, however the combination of aeration and crystal either prevented a reading being established, or delayed the response between 1 and 2 hours. Given that a slurry interfered with measurement, it was considered not appropriate to attempt to install the TOF probe into a pan but to continue with other test mixtures. High Grade Masecuite (A masecuite) was tested in the at-line spool piece located at the evaporator station and yielded no response. During normal operation this indicated that established crystal content does interfere with the measurement when the TOF probe becomes coated with or impeded by a large population of crystal. On this basis, and given the difficulty in cleaning the spool piece after each slurry, no further testing of crystal-laden product was conducted.

### 6.2.3. Temperature Compensation

- (c) *Confirm that temperature compensation is accurate and effective under each of the conditions being examined.*

As mentioned above, temperature compensation applied to velocity across a short range up to 10°C below the 65°C reference temperature was sufficient to give sound results. However temperature compensation in the native instrument amplified brix prediction errors where temperature was outside the reference temperature range by more than about 10°C. This caused an increase in the range of predicted brixes up to ±5 units relative to the regression curve purely due to temperature.

### 6.2.4. Operating Environment - Temperature and Vibration

- (d) *Confirm that the device is sufficiently robust to withstand the range of mill operating conditions including temperature and vibration.*

The instrument appears to function well after two full seasons, with no apparent loss of performance (other than inherent calibration) due to vibration during normal plant operation, ambient temperatures up to 50°C and test solution temperatures up to 78°C.

### 6.2.5. Fouling Performance between Cleans

- (e) *To test the impact of fouling, sampling over a 1 month period in the season starting one week before a scheduled (fortnightly) clean, then each day until the next clean, and then for one week following the scheduled clean.*

The impact from probe fouling was performed across two scenarios. One of these was during the course of normal testing where multiple samples were being taken across the calibration range, and the other leading into and out of two caustic cleans.

During the 2017 season, cleans were performed at intervals of 12 days, 8 days and 20 days. To test for the impact of fouling, readings across the brix range were taken and compared to the regression curve over the testing period to determine if there was an increase in the standard error of calculated results compared to the displayed readings. The standard error across all results in the 55 to 65°C temperature range was 0.44°Bx. Across the testing period which included 3 caustic cleans, the rolling 20 sample standard error varied between 0.48°Bx and 0.2°Bx but did not suggest there was a trend of increasing error due to fouling.

### 6.3. Improved Calibrations

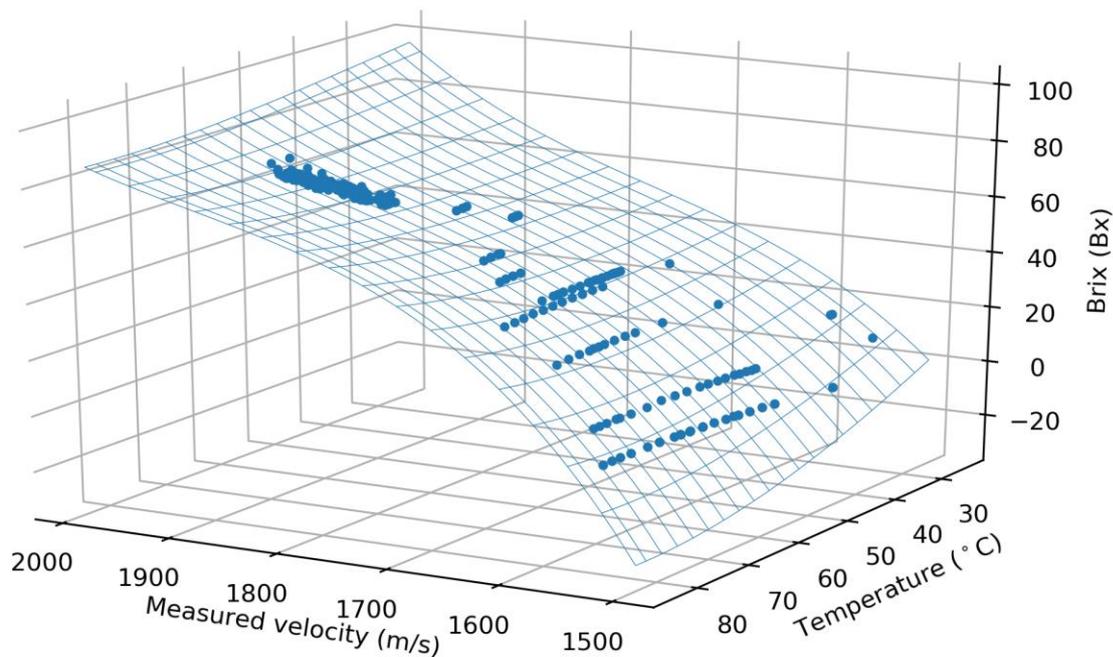
The calibration provided with the TOF probe did not cover the entire range of operating conditions required. Hence, an effort was undertaken to make use of the data obtained during the previous trials to develop an algorithm that would take the velocity and temperature measurements and calculate a brix over the entire operating range.

#### 6.3.1. New form of calibration equation

To perform this calibration, data from the 2017 trials for condensate, effluent supply juice, liquor (diluted samples), liquor (operating on-line) and liquor and water was collated. This data was fitted to a polynomial of the form shown in (1) where  $x$  is the measured velocity,  $y$  is the measured temperature and  $z$  is the brix of the solution. A third order polynomial was selected to use as this provided a good fit to the measured data while still being well behaved in regions where there was no experimental data available:

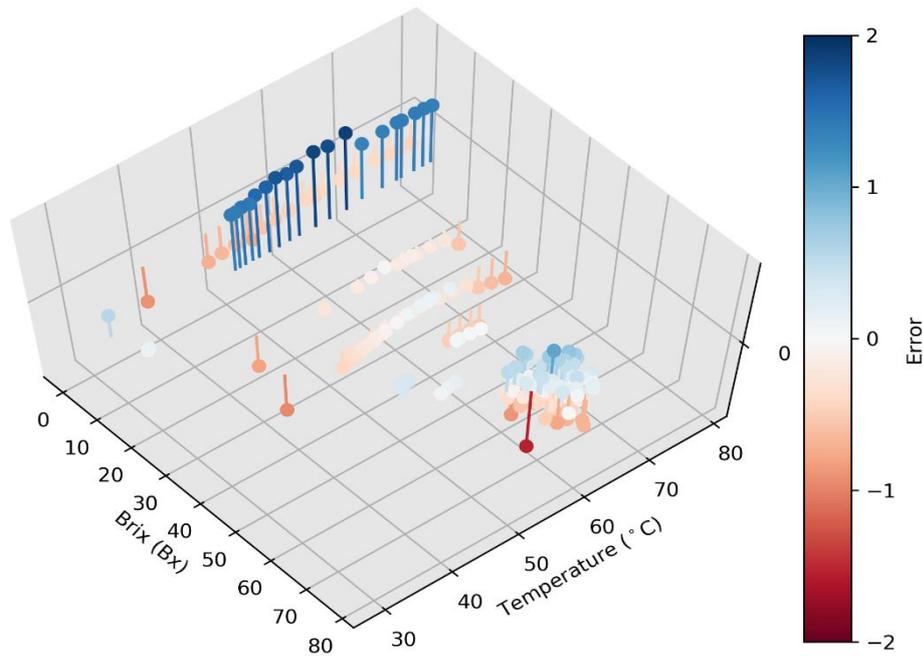
$$z = a_{00} + a_{10}x + a_{01}y + a_{20}x^2 + a_{11}xy + a_{02}y^2 + a_{30}x^3 + a_{21}x^2y + a_{12}xy^2 + a_{03}y^3 \quad (1)$$

The results from this fitting process are shown Fig. 9 and Fig. 10.



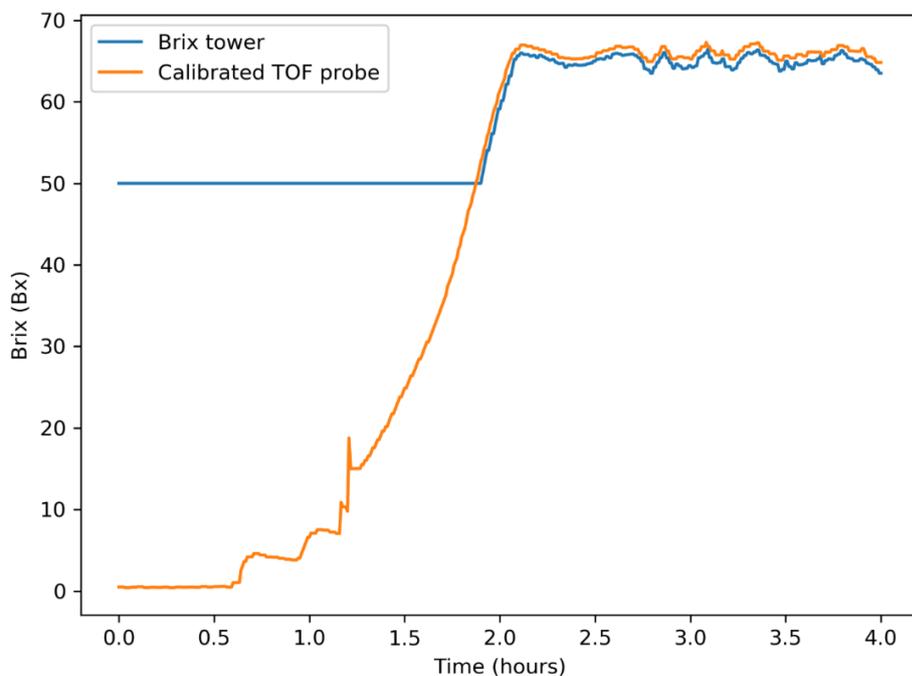
**Figure 9 - Surface of polynomial fitted to trial brix measurements**

Fig. 9 shows that the polynomial resulting from the fitting process matches the measured data well across the entire range of the trial data. It also appears to work well even when extrapolated into regions where there is no data available.



**Figure 10 - Differences between measured and fitted brix values**

Looking at Fig. 10, the difference between the calculated and measured brix is shown. For the majority of data points, the difference is within a  $\pm 1^\circ\text{Bx}$  band that lies fairly evenly around 0. Of particular note is that the differences were calculated from samples taken over a 6 weeks period and straddled three caustic cleaning intermissions, indicating that across the operating range that results were generally repeatable. The exception to this is the initial data set around  $10^\circ\text{Bx}$  that was obtained from testing ESJ. This may have been due to reference instrument errors associated with this set of trial data as a short series of tests subsequently provided more accurate predictions.



**Figure 11 - Comparison of brix measured by brix tower versus calibrated TOF probe on effert start-up.**

The resulting polynomial algorithm was implemented in the DCS system and the calculated figures were recorded alongside the existing brix tower measurement. Fig. 11 shows both of these sets of values from operation during the 2018 crushing season. In this particular instance, there is an obvious offset due to calibration applied to the brix tower measurement, however both values track very well over time. Also, it can be seen that the result calculated from the TOF probe measurements provides reasonable figures from almost straight water all the way through to normal liquor brix operating levels. The brix tower, in comparison, is only calibrated for concentrations over a shorter range above 50°Bx to maintain resolution and does not provide information at lower brix ranges.

Including the additional data from trials conducted in 2018, the overall standard deviation increased from 0.59°Bx to 0.77°Bx. While the new predictions still broadly tracked laboratory results, the laboratory results from 2018 were offset, on average, by approximately 1.2 units. The change in laboratory instruments appears to have contributed to the offset which increased the error of prediction. A revised set of coefficients was calculated incorporating data from both refractometers.

### 6.3.2. Fitted coefficients – Sugar Solutions

It was noted that the temperature difference between ESJ and the instrument during testing may have exceeded the capability of the laboratory digital refractometers, despite being within the specification limits which allowed for sample temperatures up to 60°C. This could have accounted, in part, for some of the larger differences up to 2 brix when incorporated into the multivariate model. This was quite apparent for the low brix juice samples and indicated an emerging trend for the higher brix liquor samples.

Comparing the resulting surfaces for sugar solutions, Fig. 12 shows a slight reshaping for high brix when outside the normal operating temperature range (55 to 65°C). The new season data was based on results from the improved laboratory techniques using newer refractometers with cooled samples, and further using suitable brix spindles as an additional point of reference. There was sound correlation between spindle and refractometer readings when samples were allowed to cool. Inclusion of additional samples would enable the coefficients to be refined and updated, but would not negate the utility of the coefficients established to date.

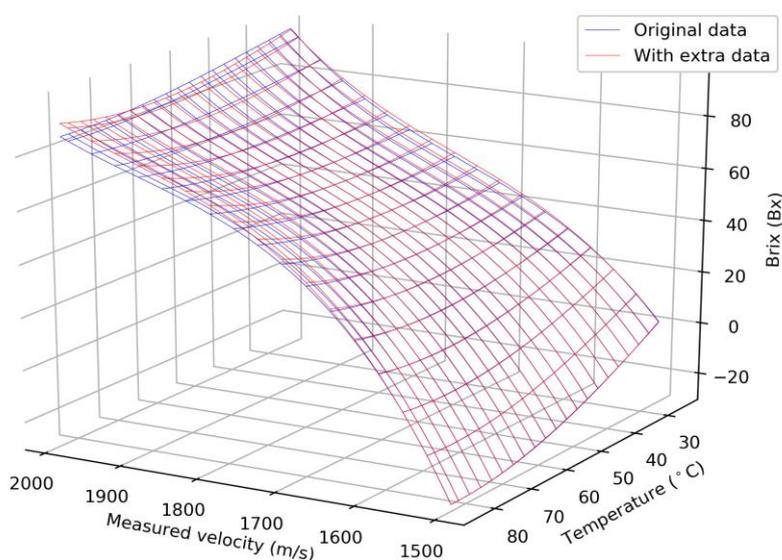
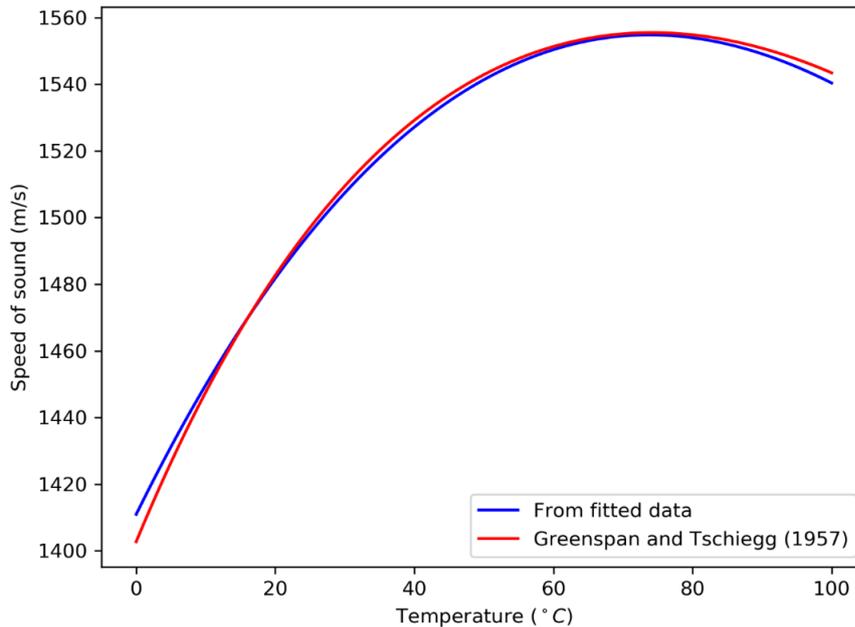


Figure 12 - Representation of Surface Equations with Original and New Season Data

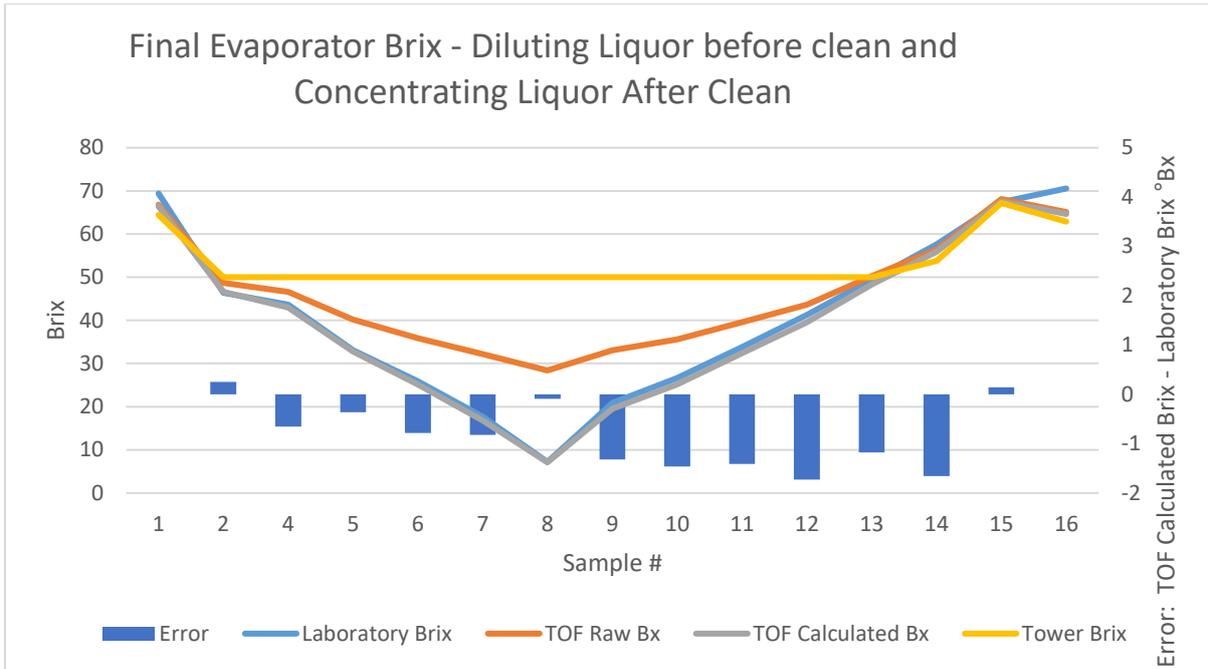
### 6.3.3. Zero Brix solutions



**Figure 13 - Comparison of expected speed of sound in clean water against values calculated using the fitted algorithm.**

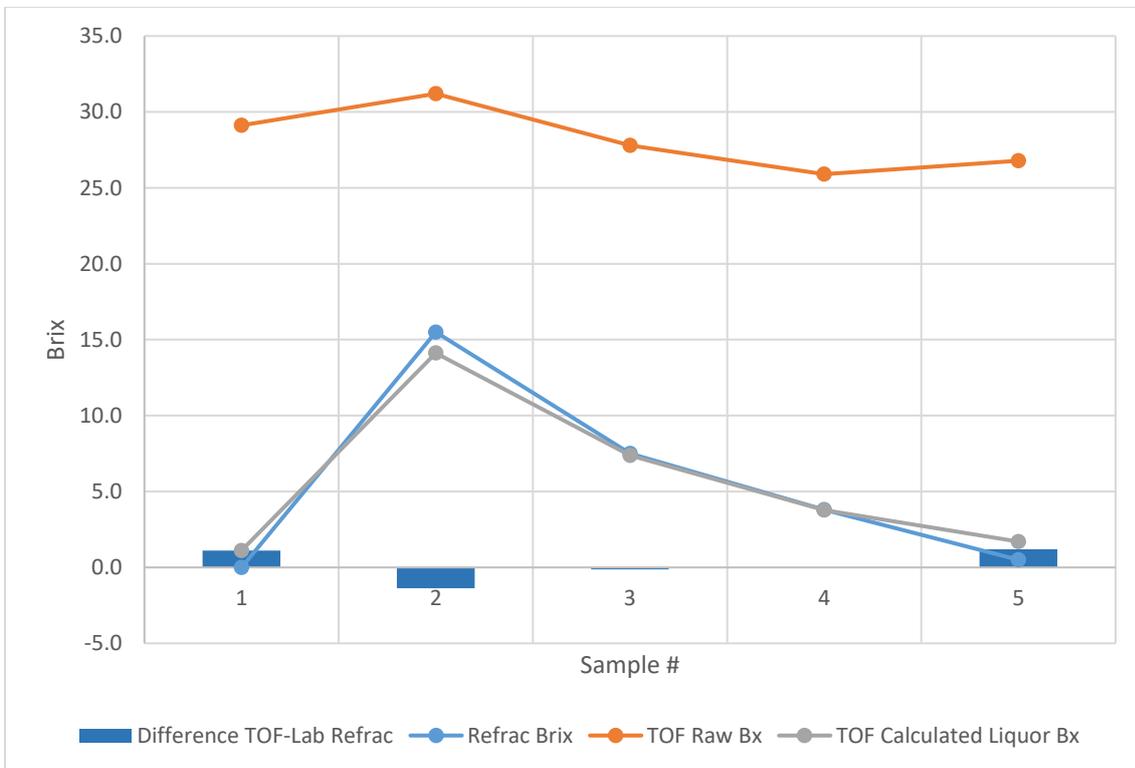
As a check on the validity of the overall fitting, the fitted algorithm was used to determine the speed of sound in clean water over a range of temperatures. Ideally this comparison would be done across a range of concentrations, but there is limited information available in the literature relating both temperature and concentration of a sucrose solution to its speed of sound. Greenspan and Tschiegg (1957) used experimental data to determine a separate algorithm for the speed of sound in water for the range 0 to 100°C. Fig. 13 shows a comparison of the expected speed of sound compared due to this algorithm against the results derived from fitting the TOF probe data. There is very good agreement across the entire range, with the difference typically being less than 5 m/s, except as the temperature approaches 0°C where the difference increases up to 10 m/s. As the data used in the TOF probe fitting process was only available for temperatures in the range 40 to 80°C, the close agreement outside of this range provides increased confidence for the other extrapolated values shown in Fig. 9.

To further test the instrument with the sensor remaining “on-line”, operational tests were repeated with particular focus on the scenarios leading into and out of caustic cleans. Samples were taken as the evaporator set purged of liquor and brix was decreasing, and again as juice was re-introduced to the set after an evaporator clean and brix was increasing. Under these conditions, the fall and rise of liquor brix was quite rapid, taking between 30 and 60 seconds to extract samples from the spool piece. During testing, a considerable quantity of solids, scale and fibre also required clearing from the liquor pump suction and causing the pump to cavitate. This resulted in temporary loss of liquor flow during sampling, and interruptions to the sampling routine as the pump had to be stopped and the strainer cleared. This was most likely to have introduced some additional variation and errors during sampling particularly at the higher brix levels which was not so apparent during normal operation.



**Figure 14 - Final Evaporator Brix - Diluting liquor before clean and concentrating liquor after clean**

As mentioned earlier, the calculated brix tended to be under-predicted, and the errors preceding the clean at 0.4°Bx were less than the errors after clean, from sample 9, which averaged around 1.2°Bx. The data however did show that the surface equation provided a substantially improved brix prediction as brix decreased below 50°Bx which neither the native TOF equations nor brix towers could satisfy. A specific set of tests was also performed on water and ESJ “at-line”. The error (Difference between TOF calculated brix and refractometer brix) for water and low brix juice during normal operation was around 1.1.



**Figure 15 - Final Evaporator Brix - Diluted ESJ and water one week after a scheduled clean**

#### 6.3.4. Zero Brix solutions and Fouling

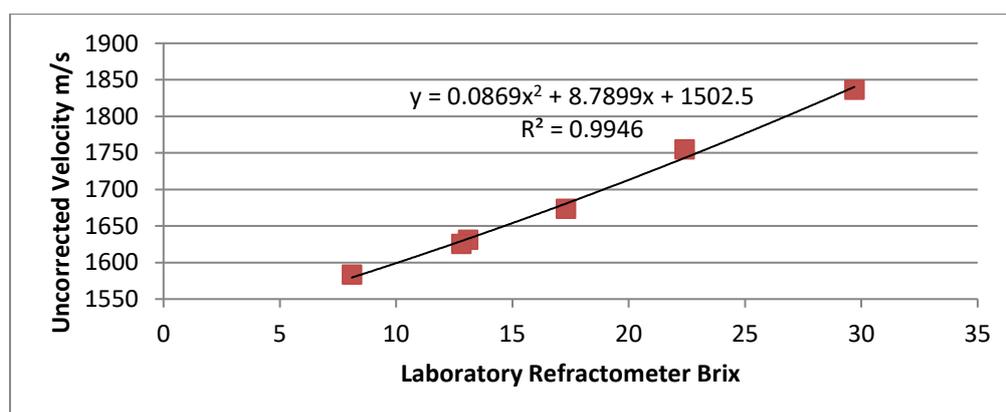
During the 2018 season after the new algorithms (covered in section 6.3.1) had been loaded, the fouling test was extended to examine the prediction of 0°Bx solutions – i.e. water. The tests looked at the brix prediction after the evaporators had been purged of liquor and washed with water prior to a caustic clean, and then again after the caustic clean had been completed and the vessels had again been washed with water.

For the first cleaning intermission, there was still sugar present between 2 and 4°Bx in the washed vessels which potentially masked any offset which may have occurred due to fouling. During the subsequent cleaning intermission monitored in Fig. 14 and Fig. 15, samples were again taken after liquor had been purged from the vessels. An offset of 2.5°Bx was observed on a single sample and a paste like residue was observed in the spool piece. The surface equation for sugar solutions predicted 2.5°Bx, but the caustic equation predicted zero brix. After the caustic clean, the caustic equation still predicted 0°Bx, and the sugar equation predicted 0.3°Bx. The results indicate that at this low brix, fouling may have affected brix predictions, but this was not evident at higher brix and was not sufficient to adversely affect control.

#### 6.3.5. Cleaning chemicals – sodium hydroxide

Cool sodium hydroxide (caustic) solutions (20–23°C) were used to test ultrasonic velocity response, and these data show a similar profile to liquor when plotted against refractometer brix (Fig. 16). Given the refractive index of caustic solutions relative to dissolved solids content is higher than liquor, and ultrasonic velocity response covers a similar span, the TOF device could only display an overestimated brix reading based on the simple liquor calibration.

Caustic solutions showed good responses at fairly low working strength concentrations ranging from 2.3% to 12.1% w/v which corresponded to refractometer brixes from 8 to 30°Bx. Initial observations would indicate that a correlation between brix and measurements using the TOF instrument are possible if final adjustment is made in the supervisory control system to allow for non-sugar solutions.



**Figure 16 - TOF uncorrected velocity versus laboratory caustic solution brix**

The calibration process used earlier for ESJ and liquor was repeated using a combination of the condensate and caustic results. This allowed an algorithm to be developed, based on a second order polynomial as shown in (2), to calculate the caustic working strength based on the TOF probe velocity and temperature measurements. Fig. 17 and Fig. 18 show the resulting surface and differences between measured and fitted values. In general, there appears to be very good agreement between the fitted and measured values and the resulting response surface profile is quite flat.

$$z = b_{00} + b_{10}x + b_{01}y + b_{20}x^2 + b_{11}xy + b_{02}y^2 \quad (2)$$

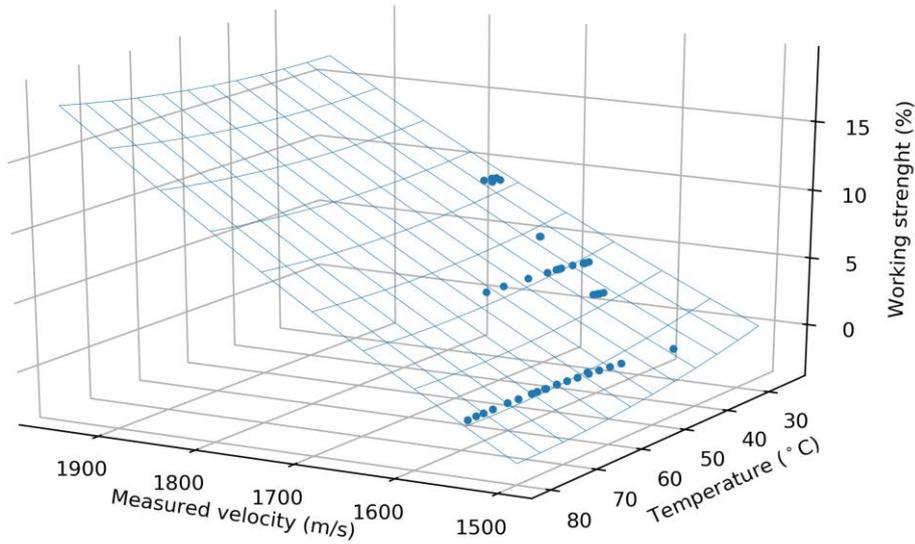


Figure 17 - Surface of Polynomial fitted to trial caustic measurements

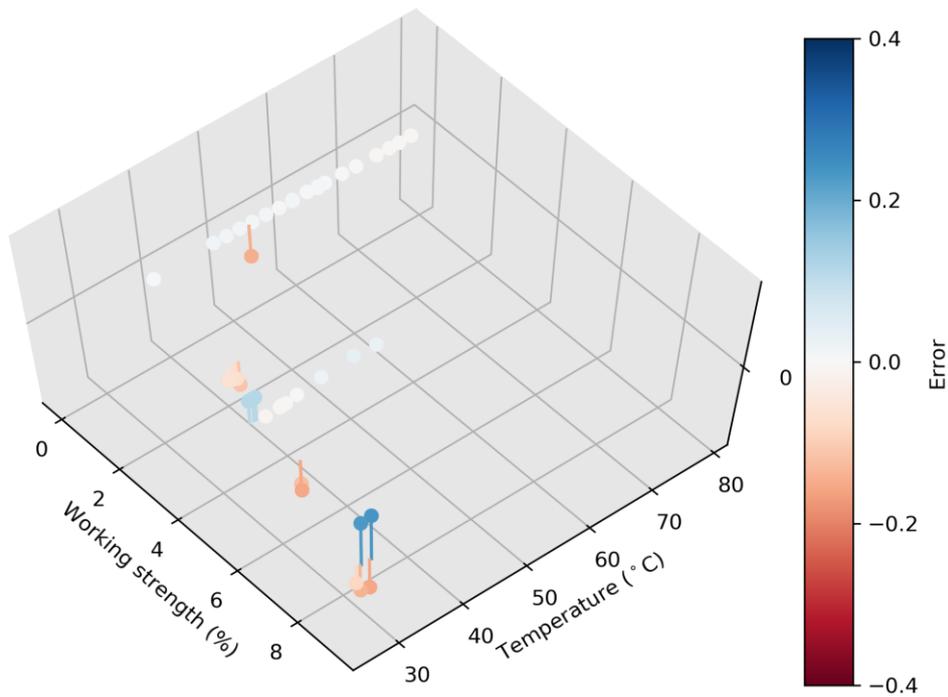


Figure 18 - Differences between measured and fitted caustic values.

**Caustic fitting from original data**

$b_{00}$	$b_{10}$	$b_{01}$	$b_{20}$	$b_{11}$	$b_{02}$
-0.181	-0.258	-0.406	0.638	0.333	0.048

The coefficients cited here apply to the original working solution of sodium hydroxide used when cleaning evaporators. As brix would change during any cleaning cycle as scales were either dissolved or brought into suspension, it would only approximate sodium hydroxide concentration during a clean. Due to resource constraints, samples were unable to be further analysed to determine the relative concentrations of caustic and non-caustic constituents. Whilst the information may be useful during a chemical clean, considerably more data would be required.

For operational purposes, the DCS was programmed to identify changes in mode of operation to select the appropriate algorithm in order to provide meaningful signal interpretation. Although not yet implemented, control could now be set based on “true brix” during a caustic clean. It should be noted that brix is not the same as working strength and is also subject to the accumulation of scale and salts in the mixture, so the calibration itself was not universal but nonetheless indicative. Caustic cleaning solutions showed good responses at fairly low working-strength concentrations ranging from 2.3% to 12.1% w/v that corresponded to refractometer brixes from 8 to 30°Bx. Over this range, the native TOF instrument calibration indicated a brix signal, or “liquor brix”, between 30 and 50°Bx. However with the algorithm loaded for caustic, the brix during the caustic boil could be monitored continuously.

### 6.3.6. Neat Solutions of Sodium Hydroxide and EDTA

Subsequent testing at higher brix indicated that a saturation point may have been reached. Both Fig. 19 and Fig. 20 show responses for predicted brix and TOF velocity tending to “saturate” around 1950m/s. A velocity limit of the instrument appeared to have been reached. Extrapolation would suggest this corresponds to a working solution laboratory refractometer brix around 40°Bx, but may be outside the range for pure sodium hydroxide solutions. Further tests would be warranted to find these limits, and the profile indicates this should be possible.

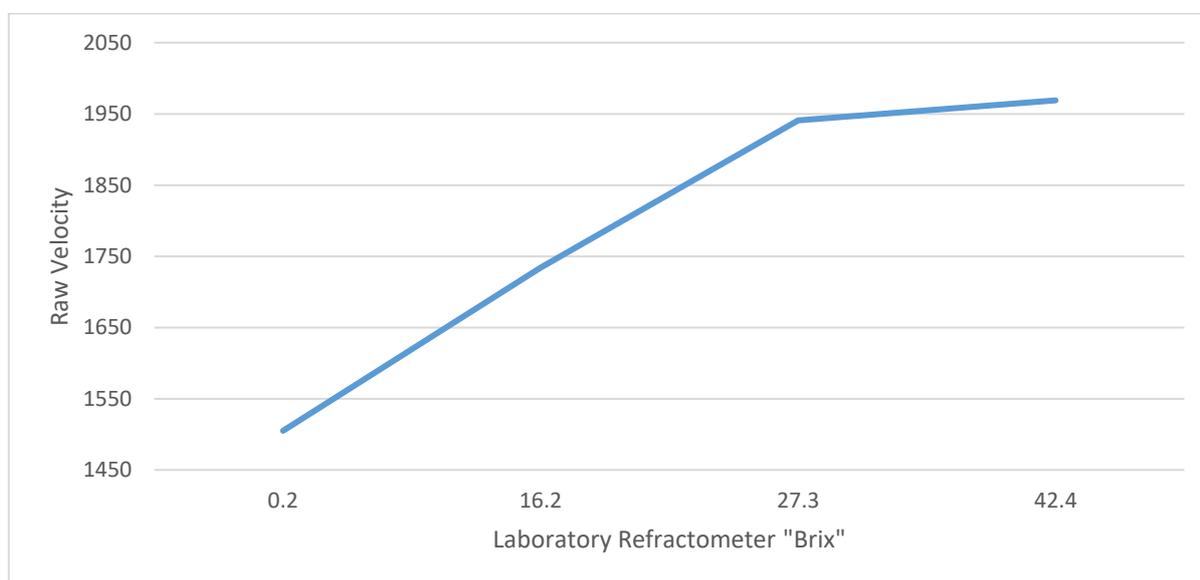
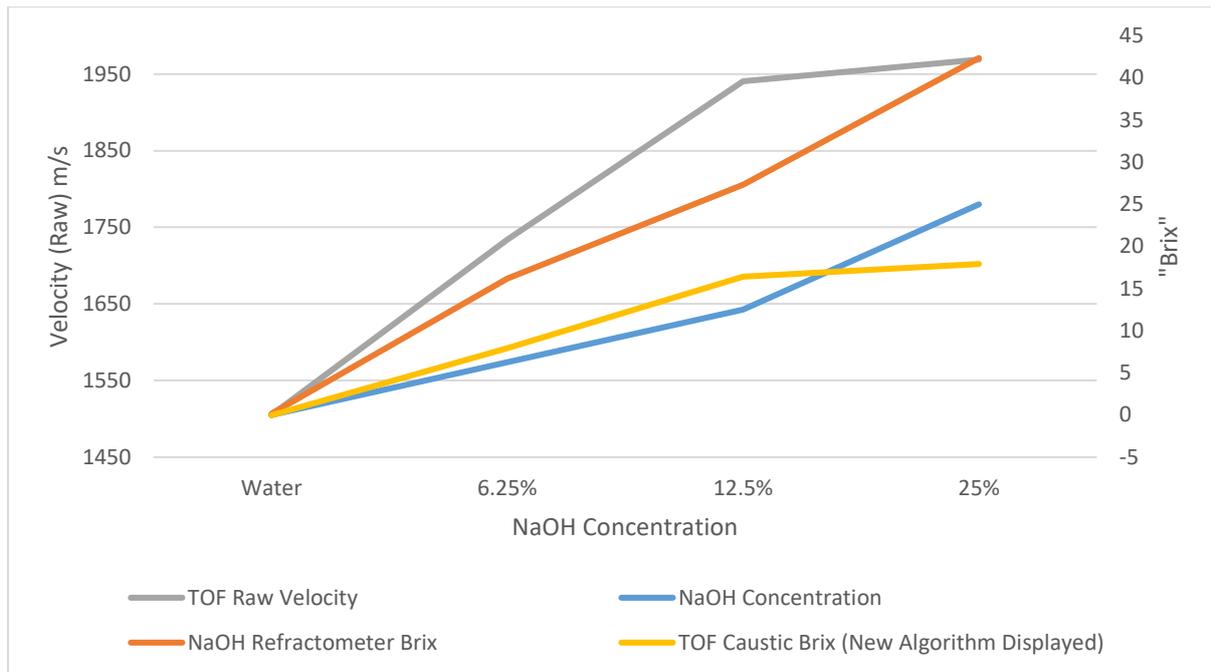
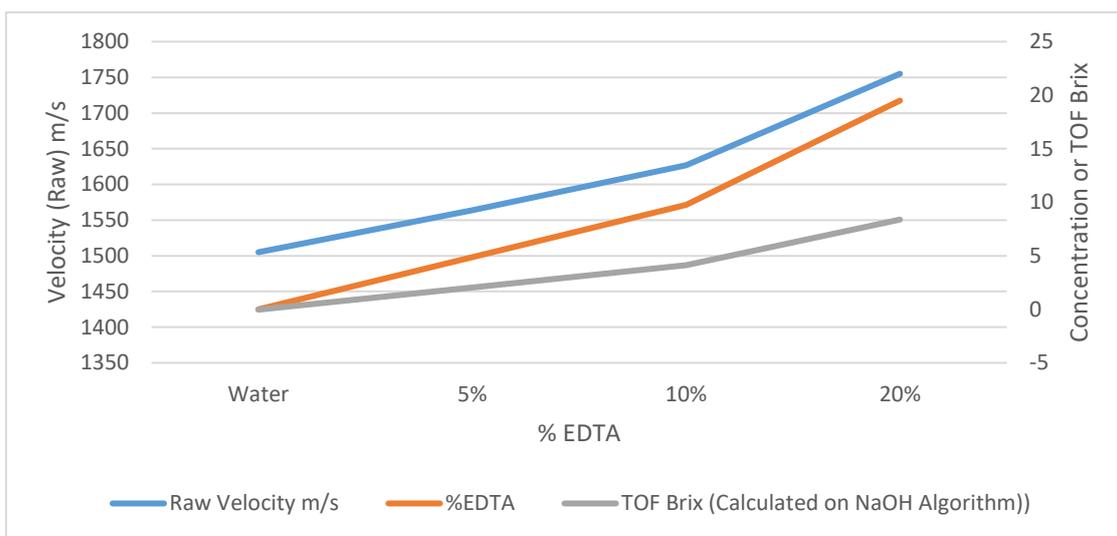


Figure 19 - Sodium Hydroxide Raw Velocity vs Refractometer "Brix"



**Figure 20 - TOF Velocity profile and TOF "Brix" for Sodium Hydroxide solutions**



**Figure 21 - TOF Velocity Response Profile for pure diluted EDTA solutions**

The final cleaning chemical tested was a solution of a sodium salt of EthyleneDiamineTetraaceticAcid (EDTA). Again this was performed on a neat solution of 39% EDTA diluted to three lower concentrations at 25°C for assessment. Typically for evaporator cleans, solutions are used from 2% to 5% depending on the temperature of the clean. In Fig. 21 the NaOH equation for TOF brix was plotted for comparison, and to show that the NaOH algorithm on its own would not predict EDTA concentration. An initial velocity response profile was plotted from which a surface equation could be generated, but this would be only for a single temperature. During a cleaning cycle, as EDTA chelates with evaporator scale the increase in brix due to impurities would also need to be examined further, but this was not possible with the existing trial installation and would be the basis for future work.

### 6.3.7. Experimental Error and Suggested Improvements

It was noted that the temperature difference between ESJ and the instrument during testing may have been beyond the capability of the refractometer at times, accounting for some of the larger differences up to 2°Bx when incorporated into the multivariate model. This was more apparent for low brix juice samples rather than high brix liquor samples.

The methodology for subsequent testing in 2018 with the multivariate equations used a new Bellingham Stanley OPTi digital refractometer and suitable brix spindles as a second point of laboratory reference. A high temperature (60°C) spindle was used in the field for liquors between 60 and 80°Bx, but all other tests were performed after allowing samples to cool to laboratory temperatures before testing with laboratory refractometer.

Initially the field tests in 2017 had been done with an Atago digital refractometer. The additional data incorporating samples taken in 2018 increased the standard deviation from 0.59 to 0.77°Bx and revealed an offset of approximately 1.2 units. A revised set of coefficients was calculated using data from both refractometers. The reference method revealed that both the instruments and technique did have an effect on the reliability of final results. The most reliable measurements were with a high temperature spindle and refractometer readings performed only with samples cooled to laboratory temperature (between 23 and 25°C).

The updated chemical algorithm would enable a mode of operation to monitor indicative caustic concentration during a chemical clean, however to improve accuracy and correlation with reliable metrics, the calibration dataset should perhaps be progressively overwritten to generate coefficients relating to % dry substance rather than dissolved solids (brix).

## 7. CONCLUSIONS

The shortcomings of microwave density measurement prompted this investigation into ultrasonic time-of-flight to control evaporator brix.

The Pro-M-tec TOF ultrasonic instrument used in this study showed the capability and limitations of its standard calibrations and the susceptibility of the instrument to air and larger crystal populations. The instrument as-supplied with on-board calibration equations showed stable and repeatable performance in measuring liquor from final evaporator vessels under normal continuous operating conditions. There was some susceptibility to fouling, but this did not compromise the performance of the instrument.

By enhancing calibration performance with downstream signal processing through the mill DCS, a more robust multivariate calibration was achieved which extended the instrument's capability to cover liquor, working solutions of caustic, sodium hydroxide, juice and water. This would make the instrument suitable for evaporator installations in the Australian Sugar Industry providing the downstream signal processing was also implemented.

The Pro-M-tec instrument as-tested is a well-engineered product and modifications to the product were being developed by the manufacturer during the course of this project, however it does not appear that there have been any improvements to their calibration equations. The instrument as tested provided good signal processing and was sufficiently stable to provide a basis for good process control. Greater involvement by the manufacturer could have led to improvements in the versatility of the instrument and more testing of the improved calibration equations.

## 8. RECOMMENDATIONS FOR FURTHER RD&A

Whilst the instrument supplier had shown passive interest in the work being conducted, it continued to develop the technology and sensor head for installations in Europe. Towards the end of the

project, the supplier indirectly made available a comparison curve for time of flight (rather than velocity) with refractometer brix. This appeared similar to previous “calibration curves” and did not address the limitations of the ultrasonic instrument.

Further work recommended to update surface equations based on new datasets referenced back to reliable laboratory metrics – either brix or % dry substance.

By their nature, cleaning chemicals used in relatively low concentrations dissolve scales affecting the brix of solutions. Further work with the instrument to monitor the change in brix with time may help monitor the effectiveness of a clean and the likely impact on chemical strength.

More recent versions of the instruments now generate actual time of flight as an output, and an updated sensor design with a lower profile. New applications would potentially need to take this into account if using a Pro-M-tec instrument. For information some images are presented in appendix 5, but the calibration curve following a 3<sup>rd</sup> order polynomial would still have the limitations reported in this project.

## 9. PUBLICATIONS

One paper is scheduled to be published and presented to the 2019 ASSCT conference:

Ultrasonic time of flight for evaporator brix measurement. RM Stobie, BJ Burke, W Robino, D Ferraris

## 10. ACKNOWLEDGEMENTS

I wish to acknowledge the support of several people associated with this project:

Firstly my co-investigator Wayne Robino, who spent many hours with me under extremely hot and humid conditions to conduct the field trials, and who had spent many frustrating years prior to this identifying the limitations of the microwave instruments.

Brendan Burke who, late in the project was of tremendous support in developing the multivariate calibration equations which were a key product of this project.

Danny Ferraris and Liam Castles who programmed the new calibrations through the Victoria Mill factory control system.

I also wish to thank Sugar Research Australia Limited and Wilmar for funding this project and permission to publish the general findings to the Australian Sugar industry through an ASSCT paper.

## 11. REFERENCES

Greenspan M, Tschiegg C (1957) Speed of sound in water by a direct method. *Journal of Research of the National Bureau of Standards* **59**: 249-254.

## 12. APPENDIX

### 12.1. Appendix 1 - Metadata Disclosure

**Table 2 - External Calibration Coefficients**

<b>Data</b>	Calibration coefficients for externally processing instrument outputs
<b>Stored Location</b>	Wilmar
<b>Access</b>	Restricted to Australian sugar mills and refineries
<b>Contact</b>	Robert Stobie, Brendan Burke

**Table 3 - Experimental and Field Data**

<b>Data</b>	Experimental and Field Data
<b>Stored Location</b>	Wilmar
<b>Access</b>	Restricted to Wilmar and Victoria Mill technologists
<b>Contact</b>	Robert Stobie

12.2. Appendix 2 – Regression Curve Data for Original On-Board Calibration at 60°C

Sample	Date	Time	TOF Display Brix (Instrument)	Velocity (m/s)		Temperature T (°C)	Laboratory Brix Values (y Axis)	Regression Curve for TOF Displayed Brix	Difference (Error) between Displayed Results and Curve of best Fit
			Raw		Compensa		y		
141	15/11/2017	13:28	26.55	1551.50	1546.50	60.00	0.00	26.5	-0.01
124	15/11/2017	10:53	26.57	1551.60	1546.70	60.00	0.00	26.5	-0.03
110	14/11/2017	14:10	29.59	1573.70	1569.50	60.70	10.50	29.5	-0.05
149	20/11/2017	11:12	37.08	1631.40	1626.30	60.00	26.90	36.7	-0.39
160	20/11/2017	14:00	43.29	1678.40	1673.40	60.00	38.20	43.4	0.08
188	23/11/2017	8:41	44.18	1686.00	1680.20	59.20	40.00	44.6	0.38
187	23/11/2017	8:40	44.22	1685.80	1680.50	59.70	40.00	44.6	0.34
186	23/11/2017	8:39	44.37	1685.60	1680.90	60.20	40.00	44.6	0.19
184	21/11/2017	9:29	48.70	1720.30	1714.50	59.20	46.00	48.8	0.11
183	21/11/2017	9:25	48.79	1719.40	1715.20	60.80	46.00	48.8	0.02
178	21/11/2017	9:05	51.98	1744.70	1739.40	59.70	50.70	52.4	0.44
1	7/12/2017	10:50	64.53	1840.10	1834.50	59.30	65.00	64.9	0.40
30	4/12/2017	13:37	64.71	1841.00	1835.80	59.80	65.00	64.9	0.22
2	7/12/2017	11:02	64.80	1841.80	1836.60	59.70	65.20	65.1	0.32
31	4/12/2017	13:42	65.13	1844.20	1839.00	59.90	65.30	65.2	0.09
3	7/12/2017	11:10	66.41	1853.80	1848.70	59.90	66.30	66.2	-0.23
28	4/12/2017	13:05	66.57	1854.60	1850.00	60.30	66.30	66.2	-0.39
29	4/12/2017	13:12	66.75	1855.90	1851.30	60.40	66.40	66.3	-0.47
64	8/12/2017	10:46	67.07	1857.90	1853.70	60.90	66.80	66.7	-0.40
26	4/12/2017	10:52	67.76	1863.50	1859.00	60.50	66.90	66.8	-1.00
65	8/12/2017	10:49	67.26	1859.80	1855.20	60.40	67.10	67.0	-0.30
4	7/12/2017	11:13	67.26	1860.00	1855.10	60.10	67.20	67.1	-0.20
27	4/12/2017	10:55	67.61	1862.20	1857.80	60.70	67.20	67.1	-0.55
36	5/12/2017	14:45	67.94	1866.30	1860.30	59.00	67.40	67.3	-0.69
25	4/12/2017	9:56	68.11	1866.10	1861.60	60.60	67.70	67.5	-0.56
37	5/12/2017	14:49	68.24	1868.40	1862.60	59.30	67.80	67.6	-0.59
5	7/12/2017	11:23	67.90	1864.40	1860.00	60.60	68.20	68.0	0.14
46	6/12/2017	9:01	68.28	1868.70	1862.90	59.20	68.30	68.1	-0.14
24	4/12/2017	9:52	68.45	1868.40	1864.20	60.80	68.50	68.3	-0.11
38	5/12/2017	14:56	69.01	1873.90	1868.50	59.60	69.00	68.8	-0.17
47	6/12/2017	9:12	68.96	1874.00	1868.00	59.10	69.10	68.9	-0.02
6	7/12/2017	11:25	68.51	1868.90	1864.70	60.80	69.10	68.9	0.43
20	4/12/2017	6:30	69.59	1878.40	1872.90	59.50	69.20	69.0	-0.55
49	6/12/2017	10:03	69.05	1873.60	1868.80	60.20	69.20	69.0	-0.01
19	1/12/2017	17:25	69.17	1874.70	1869.60	59.90	69.30	69.1	-0.03
48	6/12/2017	9:59	69.33	1875.60	1870.90	60.30	69.40	69.2	-0.09
39	5/12/2017	15:00	69.49	1876.90	1872.10	60.20	69.60	69.4	-0.05
21	4/12/2017	6:37	70.09	1881.30	1876.60	60.30	69.70	69.5	-0.55
84	13/12/2017	14:45	69.32	1876.60	1870.70	59.20	69.80	69.6	0.32
22	4/12/2017	8:40	70.41	1883.30	1879.00	60.70	70.00	69.8	-0.57
40	5/12/2017	15:13	70.11	1881.90	1876.70	59.80	70.10	69.9	-0.17
50	6/12/2017	10:13	70.38	1883.60	1878.80	60.20	70.30	70.1	-0.23
41	5/12/2017	15:16	70.32	1883.50	1878.40	59.80	70.40	70.2	-0.07
85	13/12/2017	14:47	70.00	1881.80	1876.00	59.20	70.50	70.3	0.35
97	19/12/2017	16:54	69.62	1877.60	1873.10	60.50	70.60	70.5	0.83
98	19/12/2017	16:57	70.74	1886.10	1881.50	60.40	71.00	70.9	0.12
51	6/12/2017	10:15	71.05	1888.30	1883.90	60.60	71.00	70.9	-0.19
88	13/12/2017	15:09	71.22	1890.20	1885.20	60.00	71.50	71.4	0.15
93	19/12/2017	16:24	71.97	1895.80	1890.80	60.00	71.80	71.7	-0.29
58	6/12/2017	14:31	72.10	1896.50	1891.90	60.40	71.80	71.7	-0.42
87	13/12/2017	15:00	72.03	1895.60	1891.30	60.80	72.30	72.2	0.17
54	6/12/2017	10:34	72.86	1901.70	1897.70	61.00	72.40	72.3	-0.56
59	6/12/2017	14:33	73.61	1907.70	1903.30	60.60	73.10	73.0	-0.58
100	19/12/2017	17:16	74.17	1912.90	1907.60	59.70	75.00	75.0	0.86
101	19/12/2017	17:18	76.54	1930.80	1925.30	59.40	77.50	77.7	1.20
<b>Std Deviation</b>								<b>0.42</b>	

## 12.3. Appendix 3 – Sample Data for Sodium Hydroxide Calibration

Sample	Date 20 Dec 2017 Caustic Clean Initiated	Brix Display Instrument	Velocity (m/s) Raw & Compensated		Temperature T (°C)	Laboratory Values (x Axis) Working Strength (by titration)
13	Condensate	25.60	1550.70	1539.00	53.40	0.00
25	Caustic	31.27	1611.20	1582.30	36.10	3.05
24	Caustic	31.50	1612.30	1584.10	36.80	3.05
23	Caustic	31.57	1612.60	1584.60	37.00	3.05
22	Caustic	31.70	1613.20	1585.50	37.30	3.05
21	Caustic	31.97	1614.80	1587.30	37.70	3.05
26	Caustic	35.03	1643.30	1610.80	32.40	4.57
27	Caustic	35.19	1644.10	1612.10	33.00	4.57
28	Caustic	35.26	1644.40	1612.60	33.20	4.57
29	Caustic	35.32	1644.60	1613.00	33.40	4.57
38	Caustic	36.16	1649.70	1619.40	34.70	4.57
37	Caustic	36.74	1652.00	1623.80	36.80	4.57
36	Caustic	36.80	1652.20	1624.20	37.00	4.57
35	Caustic	36.85	1652.40	1624.60	37.20	4.57
34	Caustic	36.98	1652.90	1625.60	37.70	4.57
33	Caustic	37.41	1654.40	1628.90	39.40	4.57
32	Caustic	38.36	1657.20	1635.30	43.10	4.57
31	Caustic	39.30	1660.10	1643.20	48.00	4.57
30	Caustic	40.00	1662.00	1648.40	51.40	4.57
20	Caustic	41.44	1691.60	1659.40	32.80	6.10
19	Caustic	41.58	1692.50	1660.30	32.80	6.10
14	Caustic	50.40	1765.50	1727.50	26.70	9.16
15	Caustic	50.70	1767.50	1729.80	27.30	9.16
16	Caustic	50.80	1758.00	1730.50	27.50	9.16
17	Caustic	51.11	1769.10	1732.80	28.70	9.16
18	Caustic	51.16	1759.20	1733.10	29.00	9.16

12.4. Appendix 4 – Recent Pro-M-tec Data and Latest Instrument

