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Maximising the benefits of NIR rapid analysis for sugarcane mill laboratories

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Abstract: Obtaining, training and retaining laboratory personnel for Australian sugarcane mills is a growing concern within the industry. The test duration for some primary laboratory methods is often too long to make the required process adjustments in time, such as altering the high-grade centrifugal settings. Once established, near-infrared (NIR) laboratory instruments using mature calibrations provide many advantages that address both issues, such as ease of use, speed of analysis, multiple constituent results generated in one scan for multiple mill products, and precision and accuracy of results. However, an initial "development" procedure is required to achieve mature calibrations, followed by an ongoing "operation" procedure. This paper demonstrates how the "development" and "operation" procedures were successfully applied to two sugarcane mills. A previously generated globalised calibration, created from various mill instruments and sample populations, was used to develop a mature localised calibration specific to the mill sample sets and their respective laboratory instruments. Stored raw sugar (pol. sugar, water), fresh raw sugar (pol. sugar, water), and molasses (dry substance, sucrose, and final molasses sucrose) were the targeted products considered for the two mills. The "development" methodology used as much data as was practical to rapidly represent the mill sample populations until the equation achieved stability, i.e. the standard error of prediction (SEP) was less than the error control limit (ECL) for each product's constituent equations. Once matured, the "operation" methodology was implemented, where only 10% of the total sample population scanned by the NIR instrument was required for validation to monitor and maintain prediction performance. Novel software tools were implemented to improve the efficiency of the validation process. Both mill instruments underwent the "development" procedure during the 2019 season. Multiple NIR calibration updates were applied to achieve SEPs that were within or converging to their respective ECLs. The "operation" procedure was implemented during the 2020 and 2021 seasons, where only a single-seasonal NIR calibration update was required for each mill for all product constituent equations to meet the required prediction performance criteria. Providing reliable NIR test results within such short time frames allowed near real-time decisions to be made by process operators with minimal training requirements. The two-stage NIR development/operation methodology can be employed with the appropriate data for similar products.

Keywords: near-infrared spectroscopy, laboratory analysis, mill application, process control

1 Introduction

Near-infrared (NIR) spectrophotometry is a secondary technique that leverages other primary laboratory techniques to provide more cost-effective and rapid analysis. This is achieved by using the NIR spectra from a population of samples with known primary laboratory values to develop a NIR prediction model using statistical methods such as principal component analysis or partial least squares regression. While the resultant prediction models are powerful tools for the rapid analysis of a variety of sample types, each model requires considerable initial work to develop and significant ongoing effort to maintain, typically requiring laboratory validation performed on at least 10% of the total samples scanned to ensure analytical performance remains within agreed acceptance criteria over time.

NIR has been used in online and at-line applications across many areas of the sugar industry for quality assurance, process control and mill payment purposes (O'Shea et al. 2010). Earlier work related to the viability of NIR methods in mill laboratory applications (O'Shea et al. 2011) resulted in an SRA-led research project' "Project 2014051 – Improving mill efficiency through rapid analysis methodologies" (Keeffe 2017). This project developed global calibration equations for multiple products enabling the near "turn-key" use of NIR instruments in mill laboratories. Global NIR prediction models are robust and contain data from multiple instruments, instrument types and/or mills but typically have higher errors than local calibrations. Local calibrations are models that comprise calibration data from a single instrument collected at a single mill. These typically provide the lowest errors but are specific to the mill for which they were developed and do not translate well to other mill situations.

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The global or "starter" calibration equations are used as valuable bridging calibrations until local models can be established (Keeffe 2017). Since the conclusion of the SRA-funded project, the global calibration equations have been implemented at multiple mills and have been localised using local sample data sets. This paper reviews the current implementation of starter calibrations and the operational procedures used to achieve and monitor mature localised calibrations at two mills.

2 Materials and methods

A Foss DA1650 NIR benchtop spectrophotometer was installed at Factory 1 in 2018 and Factory 2 in 2019. While many mill product equations are available for the DA1650 instrument (raw sugar, massecuite, juice/syrup, prepared cane, boiler water, mill mud and bagasse), the primary focus for this work was the use of the molasses, raw sugar and "fresh raw sugar" product equations.

Once installed, the instruments were connected to a remote SRA-hosted server where product calibrations, sample cup requirements and scan setups were downloaded to the instrument. The instruments were subsequently synchronised with the server, either continuously or at the discretion of the user, to upload data (scans, predictions, and reference values) and to download updates (changes to equations, settings or bias values). All data is stored on the server in a Structured Query Language (SQL) database that can be queried for information stored within its structure.

SRA provided operational training to key mill staff to pass on to their laboratory staff or process operators. The training was provided for the following tasks: sample cup loading, collecting a sample scan, entering sample information, synchronising the instrument and performing diagnostic tests. Physical training entailed basic sample preparation for the two cup types including the use of the gold reflector for clear liquid samples as well as effective cleaning methods for the various products. Once a product was selected, the instrument instructed the user on what sample cup to use, and a radio-frequency identification (RF ID) chip on the cup ensured that the instrument would not scan if the wrong cup was used. It was demonstrated that scan times varied between 30 seconds to 2 minutes, depending on the absorbance of the sample, and multiple scans of the same sample were carried out to indicate NIR analysis repeatability.

The samples for NIR analysis were collected using the same sampling locations and methods as those for primary laboratory methods (except for the fresh raw sugar product). Most samples were poured or spooned into the sample cup to at least 80% of the depth of the cup. Juice/syrup samples were only filled to about 10% of the sample cup depth before a gold reflector was inserted to ensure a fixed 2 mm sample depth was obtained. Sample cups were cleaned with hot water (50 °C) and paper towels, followed by drying with a paper towel or by air drying.

For molasses samples, at least 80% of the sample cup depth had to be filled to ensure that no light escaped from the top of the sample. Once this requirement was met, the number of Mahalanobis distance rejections for molasses samples were maintained within acceptable limits (Mahalanobis 1936).

The fresh raw-sugar calibration was developed because the raw-sugar reflectance rapidly changed in the first 20 minutes after the sugar drier and subsequently stabilised to a linear relationship after 2 hours (Keeffe 2017). This phenomenon was an issue because effective process control decisions, such as centrifuge spin and wash times, needed to be made within the time required to process a batch of massecuite (1 hour). During the calibration phase, fresh raw-sugar samples were collected directly off the belt exiting the drier, scanned as soon as possible, and then rescanned at regular intervals until 2 hours had passed. While this was time-consuming, once enough samples had been collected to de-sensitise the calibration to the changes in raw-sugar reflectance, the sampling regime for fresh raw sugar became identical to that of raw sugar. Sugar samples older than 2 hours were scanned using the stored raw-sugar calibration. If a mill had not captured the data necessary to de-sensitise the raw-sugar calibration, the stored raw-sugar product equation was used to scan fresh raw sugar with the risk of higher errors due to the known changes in sugar reflectance.

It was important to use quantitative methods to benchmark the performance of the instrument where possible. The standard error of calibration was used to calculate the error control limit (ECL) for each constituent equation of the respective product calibration suites. The ECL was then used as the primary performance indicator for the standard error of prediction (SEP). If the SEP exceeded the ECL, the calibration performance was considered outside the expected performance criteria. In these situations, the SEP was improved by increasing the representation of the instrument and the target sample population by incorporating local scan data with matching laboratory values into the calibration population. Similarly, bias (or systematic offset) could be accounted for by adjusting the predicted values based on the average difference between NIR and laboratory results over a given population. Initially, laboratory validation was conducted as often as practical to obtain a baseline representation of any instrument differences. These validation data were used to either apply an update to the bias offset (if indicated by the *t*-test bias significance method) or to update the calibration equation if required (if SEP > ECL or if the slope of the linear regression deviated too far from one). Other performance metrics were also used, such as linear regression slope and R2.. However, caution is required when considering these two metrics, as they are both strongly influenced by the range of constituent values captured by the validation population. Once a calibration attained stable performance within the desired limits, laboratory validation could be reduced to 10% of the samples scanned. Potentially, this equated to a 90% reduction in expensive and time-consuming laboratory analyses whilst increasing the number of samples that could be analysed using the cheaper and faster NIR technique.

The molasses equation originally derived by Keeffe (2017) (G14 Molasses) was used to predict all molasses types for the

mills. To improve initial prediction performance, separate C molasses (also known as final molasses or F molasses) and A and B molasses equations had to be set up. With enough data, the need to separate the two populations (A and B molasses from C molasses) was not required. Furthermore, the initial performance of the Keeffe molasses prediction equations for the Factory 1 system did not meet the expected performance criteria due to insufficient molasses sample depth in the NIR sample cups (allowing light to escape through the top of the molasses sample). After two calibration updates, the issue was corrected, and the performance stabilised to meet the target performance criteria. The learnings from Factory 1 in 2018 were applied to the Factory 2 setup in 2019.

The global equations for stored raw sugar developed by Keeffe (2017) performed only slightly above the target limits despite any representation of the new instrument and sample populations. After subsequent updates to include scans from the new instruments and mill sample types, the stored raw-sugar equations consistently met the required performance criteria.

Factory 1 collected several fresh raw-sugar samples to capture the rapid changes in raw-sugar reflectance over the first 2 hours after drying. Factory 2 did not capture similar data, so the stored raw-sugar equation was used with the caveat that this effect would not be accounted for and may cause increased scatter in predicted results, i.e. a higher SEP if scanning fresh raw sugar.

Once NIR prediction metrics for a product were stable and within the required performance limits, the calibrations were considered mature. Mature calibrations typically require a validation population of 10% of the total samples scanned, but more than this could be performed at the discretion of the mill. Unfortunately, the 2018 trial in Factory 1 had a low bias update frequency and missed some key bias movements throughout the season. Therefore, for the 2019 season, weekly data checks were undertaken to assess performance and take appropriate action (bias adjustment/calibration update). After confirming calibrations had attained stable prediction, only 2-weekly assessments were conducted.

While the remote server and database are effective data storage tools, they did not meet the reporting requirements needed to track NIR analytical performance. Given the frequency required for performance reporting and how time-consuming traditional spreadsheet tools were, SRA developed a software application external to the server that automated the reporting process. This proved to be a valuable tool to leverage the data collected and enable objective decisions to be made that were related to NIR prediction performance.

One of the key features of this application was providing a quantitative, statistically derived measure to determine bias significance relative to the standard error of the mean for the primary data population under test; this could be used as a guide or direct decision-making tool. SQL filters were used to define the bounds of the desired data set. Additionally, outlier sub-populations could easily be excluded or included to identify Mahalanobis distance outliers, outliers outside the constituent calibration range, or those greater than three standard deviations from the bias-adjusted mean. With this tool, any population filtering option could be used to create a NIR performance report tailored to the desired reporting requirements in a relatively short amount of time.

3 Results and discussion

The 2018 season only involved Factory 1, with the original G18v1 Molasses and G18v1 Stored Raw Sugar equations derived from Keeffe (2017) loaded onto the DA1650. The calibration naming convention follows that the "G" represents "Global", the next two digits represent the year, "v" represents "version", followed by the number of updates that had taken place for that product, e.g., "G18v1 Molasses" represents the first version of the global calibration for fresh raw sugar implemented in 2018. If no version is provided (G18), the associated data relates to all versions for that year.

These early 2018 prediction results for Factory 1 were published in SRA's *Milling Matters* publication (Staunton 2018). However, a large portion of the 2018 validation data was entered into the remote database retrospectively towards the end of the season. This offered limited opportunity to carry out any bias adjustments or calibration updates during the crushing season. The validation statistics for the Factory 1 2018 season data are given in Table 1. The number of scans with associated laboratory validation results is listed as "Nv" and the total number of predictions as "Np" in the table. One bias adjustment was applied soon after instrument commissioning was completed based on the limited data available. However, in hindsight, several potential bias changes were found to have been warranted once the retrospective validation data was considered. Despite this, Table 1 shows that the respective ECLs for raw sugar pol. sugar and water were met in 2018 (although the calibration error for pol. sugar did improve in subsequent calibration iterations). Molasses did not meet the required limits for dry substance in 2018 but did for Sucrose. These results indicated that further updates were required for molasses to meet the ECL requirements. Additionally, 31.9% of molasses samples were rejected during 2018, and further investigation indicated that the sample cup was not consistently being filled enough and, as a result, the light was escaping out of the top of the sample, distorting the spectral result. Consequently, the requirement for filling 80% of the sample cup depth was enacted. An end-of-season report was generated to assess the overall 2018 performance and to identify issues that needed to be addressed.

The 2019 season saw a more structured attempt to bring the calibration performance within the required metrics as rapidly as possible. The resultant method for reaching calibration maturity now forms the basis of SRA's current methods. Both Factory 1 and Factory 2 laboratories operated FOSS DA1650 instruments for the 2019 season. The instrument use rate was high enough to warrant weekly performance reports to determine bias and/or calibration changes. Changes were discussed and agreed to with mill personnel before any

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updates were applied via instrument synchronisation events. During this time, bias adjustment decisions were made using a *t*-test to relate bias significance to the standard error of the mean value for the corresponding laboratory data. Weekly performance reports were manually generated using spreadsheets throughout the 2019 season.

While an improvement on the 2018 equation suite, the initial G19v2 equations and accompanying libraries still rejected a large percentage of the scans collected for the three priority sample types. An early update to the G19v3 library/equation reduced this rejection rate to a more acceptable level. The 2019 validation statistics for both Factory 1 and Factory 2 are shown in Tables 2 and 3, respectively. SEPs for stored raw sugar for both mills were within the corresponding ECLs. The fresh raw sugar equation used in Factory 1 had a slightly higher error than the target limit for 2019, but the capture and incorporation of raw-sugar reflectance changes from additional fresh raw-sugar samples helped improve this performance for the 2020 season (Table 4). However, no fresh raw-sugar samples were performed for Factory 2, so no fresh calibration update was possible. All molasses equations met their target error limits by separating the A and B molasses populations from the C molasses at Factory 2 (Table 3).

For the 2019 season, 12 update events were applied to both Factory 1 and Factory 2 instruments, including 25 and 21 constituent bias adjustments, respectively. The number of bias events for each product/constituent is listed as "bias changes" in Tables 2 and 3. The number and broad range of bias adjustments during the 2019 season reflected the changes that occurred in an effort to improve calibration representation. Each time calibration was updated, all corresponding biases were reset to zero, and validation was performed to determine if any bias adjustment was required. Throughout the 2020 season, the manual reporting methodology was phased out in favour of an automated reporting method. This greatly reduced the time and effort required for data analysis and reporting. Towards the end of the 2020 season, the Factory 1 DA1650 underwent a mid-season service. This resulted in a bias shift that had to be accounted for. While this event was managed effectively, it is recommended that such service events should occur in the off-season to minimise unnecessary effects on analytical performance within the season.

The 2020 season validation results for the Factory 1 and Factory 2 instruments are given in Tables 4 and 5, respectively. Stored raw-sugar equations for both mills met prediction

Table 1: G18 validation statistics for Factory 1 (2018)

Product	Constituent	SEP	ECL	R^2	Slope	Bias	Range	N_v	Rejected	Bias changes	Bias range	N_p
Stored raw sugar	Pol. sugar	0.089	0.11	0.56	0.74	0	98.48 – 99.32	1837	0.90%	1	–0.10 – 0	5948
	Water	0.03	0.03	0.54	0.66	0	0.14 – 0.37	509	0.90%	0	NA	5948
Molasses	Dry substance	0.77*	0.46	0.91	0.87	0	63.85 – 84.53	292	31.90%	1	–0.24 – 0	811
	Sucrose	1.11	1.2	0.96	1.03	–0.05	31.20 – 54.74	292	31.90%	1	–2.1 – 0	811

* Outside acceptable performance (above ECL).

Table 2: G19 validation statistics for Factory 1 (2019)

Product	Constituent	SEP	ECL	R^2	Slope	Bias	Range	N_v	Rejected	Bias changes	Bias range	N_p
Stored raw sugar	Pol. sugar	0.088	0.09	0.62	1.099	0.002	98.4 – 99.3	1195	0.40%	4	0 – 0.11	1827
	Water	0.029	0.03	0.515	0.999	0.002	0.14 – 0.60	113	3.50%	3	0 – 0.021	1827
Fresh raw sugar	Pol_Final	0.097*	0.09	0.553	0.818	0	98.5 – 99.1	2401	3.70%	5	–0.072 – 0.58	2728
	Water_Final	0.033*	0.03	0.558	0.883	–0.006	0.11 – 0.50	2341	6.10%	3	0 – 1.39	2728
Molasses	Dry substance	0.56*	0.5	0.967	0.962	–0.06	63.5 – 92.3	676	3.90%	4	–0.5 – 0	931
Molasses A and B	Sucrose	1.4	1.485	0.97	0.978	–0.015	32.1 – 59.8	637	6.70%	4	–1.27 – 0	931
Molasses C	Sucrose_Fmol	0.59	1.485	0.765	1.133	0.007	32.1 – 40.2	304	4.60%	2	0 – 1.39	395

* Outside acceptable performance (above ECL).

Table 3: G19 validation statistics for Factory 2 (2019)

Product	Constituent	SEP	ECL	R^2	Slope	Bias	Range	N_v	Rejected	Bias changes	Bias range	N_p
Stored raw sugar	Pol. sugar	0.088	0.09	0.311	0.592	–0.001	98.2 – 99.3	1128	1.80%	4	–0.41 – 0.07	2159
	Water	0.03	0.03	0.455	0.763	0.002	0.14 – 0.60	1127	3.90%	4	–0.04 – 0.08	2159
Molasses	Dry substance	0.53*	0.5	0.908	1.027	0.027	58.0 – 78.2	768	2.80%	6	0 – 1.02	1393
Molasses A and B	Sucrose	1.2	1.485	0.835	0.862	–0.01	44.4 – 59.1	497	2.70%	4	0 – 2.67	1393
Molasses C	Sucrose_Fmol	0.65	1.485	0.763	0.947	–0.111	30.0 – 39.6	271	5.10%	3	NA	503

* Outside acceptable performance (above ECL).

Table 4: G20 validation statistics for Factory 1 (2020)

Product	Constituent	SEP	ECL	R^2	Slope	Bias	Range	N_v	Rejected	Bias changes	Bias range	N_p
Stored raw sugar	Pol. sugar	0.086	0.09	0.538	0.569	–0.009	98.3 – 99.3	1770	1.70%	10	–0.08 – 0.04	2018
	Water	0.026	0.03	0.557	0.616	0	0.11 – 0.60	209	1.40%	3	0 – 0.01	2018
Fresh raw sugar	Pol. sugar	0.092*	0.09	0.554	0.58	0.008	98.3 – 99.4	3456	3.00%	10	–0.002 – 0.089	4882
	Water	0.026	0.03	0.587	0.67	–0.005	0.12 – 0.41	3430	2.60%	11	–0.03 – 0.003	4882
Molasses	Dry substance	0.448	0.5	0.954	0.915	0.001	58.0 – 82.3	780	13.50%	9	–0.137 – 0.116	1010
	Sucrose	1.328	1.485	0.976	1	0.07	31.1 – 65.2	792	12.20%	7	0 – 0.62	1010

* Outside acceptable performance (above ECL).

Table 5: G20 validation statistics for Factory 2 (2020)

Product	Constituent	SEP	ECL	R^2	Slope	Bias	Range	N_v	Rejected	Bias changes	Bias range	N_p
Stored raw sugar	Pol. sugar	0.071	0.09	0.511	0.71	0.016	98.3 – 99.3	941	9.40%	3	–0.022 – 0.03	2271
	Water	0.027	0.03	0.623	0.708	0	0.11 – 0.60	853	11.80%	0	NA	2271
Molasses	Dry substance	0.618*	0.5	0.962	0.908	0.132	58.0 – 82.3	553	20.00%	3	0 – 0.48	1825
Molasses A and B	Sucrose	1.391	1.485	0.968	0.965	0.074	31.1 – 65.2	653	14.80%	6	0 – 1.31	1825
Molasses C	Sucrose_Fmol	0.923	1.485	0.81	0.74	0.103	29.0 – 39.6	361	11.90%	5	–0.15 – 0.66	932

* Outside acceptable performance (above ECL).

Table 6: G21 validation statistics for Factory 1 (2021)

Product	Constituent	SEP	ECL	R^2	Slope	Bias	Range	N_v	Rejected	Bias changes	Bias range	N_p
Stored raw sugar	Pol. sugar	0.086	0.09	0.491	0.624	–0.016	92.6 – 99.7	1552	1.10%	2	0 – 0.019	1664
	Water	0.027	0.03	0.51	0.787	0.021	0.20 – 0.35	83	6.70%	1	0 – 0.001	1664
Fresh raw sugar	Pol_Final	0.09	0.09	0.422	0.534	–0.013	98.3 – 99.5	3057	2.50%	1	0 – 0.068	4613
	Water_Final	0.028	0.03	0.559	0.595	0.008	0.12 – 0.41	3027	2.20%	3	–0.005 – 0.012	4613
Molasses	Dry substance	0.482	0.51	0.958	0.987	0.321	63.4 – 82.3	676	2.70%	1	–0.5 – 0	846
	Sucrose	1.36	1.37	0.975	0.982	–0.044	31.4 – 57.9	682	12.60%	1	0 – –0.452	846

Table 7: G21 validation statistics for Factory 2 (2021)

Product	Constituent	SEP	ECL	R^2	Slope	Bias	Range	N_v	Rejected	Bias changes	Bias range	N_p
Stored raw sugar	Pol. sugar	0.09	0.09	0.467	0.631	0.0004	98.3 – 99.2	901	7.30%	4	–0.029 – 0.028	2657
	Water	0.03	0.03	0.469	0.604	0.003	0.11–0.93	4538	1.00%	4	–0.012 – 0.01	2657
Molasses	Dry substance	0.466	0.51	0.954	1.014	–0.025	66.4 – 78.4	462	18.40%	2	0 – 0.158	1791
Molasses A and B	Sucrose	1.618*	1.37	0.967	1.027	–0.273	31.0 – 57.5	284	19.00%	1	0 – 1.477	1791
Molasses C	Sucrose_Fmol	0.596	0.66	0.755	0.806	–0.027	31.2 – 38.9	258	6.80%	1	0 – 0.257	902

* Outside acceptable performance (above ECL).

performance metrics in terms of ECL, but the linear regression slope and R^2 were adversely affected by the limited range of the constituent values. These results indicated that stable performance had been achieved. The fresh raw-sugar equation showed improvement with the increased representation from the 2019 season fresh raw-sugar samples off the belt. The molasses equation SEPs for both mills met their required limits in 2020, except for the dry substance in Factory 2. NIR library rejections for the Factory 2 molasses data set were also higher than expected. This was related to a sub-population of C molasses snap samples which were not well represented in the 2020 calibration. The appropriate adjustments were made to represent this sub-population in the 2021 season.

12 synchronisation events to the Factory 1 instrument and nine to the Factory 2 instrument were applied during the 2020 season. The number of bias events for each product/constituent is listed as "bias changes" in Tables 4 and 5. Factory 1 required more constituent bias adjustments (50) than Factory 2 (17) to manage the effects of a mid-season lamp change.

The 2021 season validation results for Factory 1 and Factory 2 are given in Tables 6 and 7, respectively. All equation SEPs are within the target ECLs, except A and B molasses sucrose for Factory 2. The Factory 2 molasses sucrose equation performed within the desired criteria until late in the 2021 season. Upon investigation, the increase in error corresponded

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with increased dextran levels in the molasses product. The NIR analytical performance for this product type may be improved through the representation of the molasses calibration population.

The required bias update synchronisation frequency was reduced to four for Factory 1 and six for Factory 2 during the 2021 season. The Factory 1 DA1650 required nine constituent bias adjustments, and 12 were needed for the Factory 2 instrument.

Table 8 gives the DA1650 calibration update history for the two systems at Factory 1 (2018–2021) and Factory 2 (2019–2021). IN 2019, it is highlighted that both mill instruments underwent rigorous calibration development and required multiple calibration updates (up to version 3 or 4 for certain products). Typically, annually updated calibrations are applied prior to the start of the next season, and fewer updates (versions) are required within the season as the calibrations mature. Table 8 clearly shows that calibration maturity was achieved for both mills within one season (2019) as no further calibration updates were required after loading the routine pre-season calibration updates for either 2020 or 2021.

4 Conclusions

The progression from global equations developed by Keeffe (2017) to mature localised equations has been demonstrated at two different factories. While the present study has focused on the molasses and raw-sugar constituent equations, the same methodology can be applied to other products with similar results. It was demonstrate that a focussed effort to obtain enough data to represent the new instrument and local sample types into the corresponding equations and libraries is required to reach calibration maturity. The 2019 season for Factory 1 and Factory 2 shows how this can be achieved. The 2020 and 2021 seasons for both mills demonstrate how the systems can be maintained once the calibrations have reached maturity with no mid-season calibration updates and minimal bias changes required.

While direct cost benefits are difficult to identify and quantify, the savings for a mill laboratory are apparent in using NIR technology to obtain results for multiple constituents with a single scan in less than 2 minutes. This capability is significant considering the time and cost required to carry out the wet chemistry tests to obtain equivalent results. Instrument operation requires minimal training, which is an advantage when there are laboratory staffing issues. Potential benefits to the mill can also be attributed to the potential to make decisions in a timeframe that traditional wet-chemistry methods cannot provide.

Using the procedures and tools developed during this work coupled with an appropriately resourced sampling campaign, mature localised calibrations for various mill products can be established within one season of implementing new benchtop NIR installations.

Abbreviations

ECL	Error control limit
NIR	Near-infrared
N_p	Total number of predictions
N_v	Number of scans with associated laboratory validation results
SEP	Standard error of prediction

Table 8: DA1650 calibration update history for Factory 1 (2018–2021) and Factory 2 (2019–2021)

Year	Factory 1		Factory 2	
	Date loaded	Calibration update version	Date loaded	Calibration update version
2018	18/07/2018	G18v1 Molasses G18v1 Stored Raw Sugar	na	na na
2019	20/03/2019	G19v1 Molasses G19v1 Stored Raw Sugar G19v1 Fresh Raw Sugar	12/02/2019	G19v1 Molasses G19v1 Stored Raw Sugar G19v1 Fresh Raw Sugar
	17/06/2019	G19v2 Molasses G19v2 Stored Raw Sugar G19v2 Fresh Raw Sugar	15/06/2019	G19v2 Molasses G19v2 Stored Raw Sugar G19v2 Fresh Raw Sugar
	29/08/2019	G19v3 Molasses G19v3 Stored Raw Sugar G19v4 Fresh Raw Sugar	29/08/2019	G19v3 Molasses
	09/10/2019	G19v3F Molasses (Fmol added)	9/10/2019	G19v3F Molasses (Fmol added)
2020	18/06/2020	G20v1 Molasses G20v1 Stored Raw Sugar G20v1 Fresh Raw Sugar	10/06/2020	G20v1 Molasses G20v1 Stored Raw Sugar G20v1 Fresh Raw Sugar
2021	17/06/2021	G21v1 Molasses G21v1 Stored Raw Sugar G21v1 Fresh Raw Sugar	3/06/2021	G21v1 Molasses G21v1 Stored Raw Sugar

na: not applicable (instrument was not installed)

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