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A WHITE PAPER FROM FOSS:

Annual comparison of the FOSS NIR global ANN calibration against reference methods: Global ring test study overview 2007 - 2017

ANALYTICS BEYOND MEASURE

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Introduction

In 2012, FOSS completed a five year ring test study conducted with European grain networks that demonstrated the outstanding accuracy and stability of the FOSS Infratec 1241 Grain Analyzer ANN calibration for wheat and barley. The study was explained in a whitepaper that outlined the 25 years of data collection behind the calibration. In particular, it looked at the importance of seasonal and geographic variations and the impact this had on the calibration. The NIR calibration is now a global standard for measuring protein and moisture in whole grain of wheat and barley and is arguably superior in performance to the reference methods.

Since 2012, FOSS has continued this ring test on an annual basis and has expanded its reach to include a number of additional participants across the grain growing world. In addition to the regular participants from Europe, new participants from Argentina, Australia, Canada, China, Malaysia, Russia, Slovakia, South Africa and Spain have further enhanced the value of this important work.

In 2009, a ringtest study for rapeseed was conducted and in 2013, rapeseed became a standard part of the study.
This updated 2018 version of the original white paper includes the latest study data.

The Scope of the Ring test

Annual collaborative studies for the World Grain Network (WGN) have been performed by FOSS for more than fifteen years. The original purpose was to harmonize the different local and regional grain networks operating throughout Europe, to investigate differences between master reference laboratories, to make any necessary calibration adjustments and to inform members on these matters. The interlaboratory study was strengthened in 2006 by improving the information flow to members and by performing a standardized annual validation of the ANN calibration according to EN ISO 12099. The validation uses samples from the actual harvest in different countries and applies the reference methods valid in the different countries. Over the past eleven years of annual ring tests, more than 80 organizations from 28 countries have participated in this successful validation. The results have been published in reports and presented at the annual FOSS hosted Grain Network meetings held annually in March and attended by more than 50 delegates representing some of the largest grain handling companies in the world and their grain quality laboratory service providers.

The ring test consists of about ten samples each of wheat, barley and rapeseed from different origins. Samples collected from different networks are cleaned, homogenized, divided and shipped to participants for analysis. The results from the local Infratec analysis and their reference method determination of protein and moisture for wheat and barley and oil and moisture for rapeseed are sent to FOSS for evaluation. Active participation in the study from a number of networks provides a solid foundation for results and the number of participating networks has been growing in recent years. The original study started with ten countries while for 2017, there were 22.

Table 1: Countries represented in the 2017 Ring Test

Argentina	Hungary
Australia	Italy
Austria	Latvia
Canada	Lithuania
China	Malaysia
Czech Republic	Netherlands
Denmark	Poland
Estonia	Russia
Finland	South Africa
France	Sweden
Germany	United Kingdom

To further enhance the ringtest, Rapeseed/Canola was added for the 2013 edition. This product now include six years of collected data and will therefore be part of this report.

Results

The results from the 2017 World Grain Network (WGN) ring test are summarized in Table 2 for wheat and barley. In particular, it is interesting to note that the reproducibility of the FOSS ANN method is better than the reference method for both protein and moisture. The results for rapeseed are summarized in Table 3. The same tendency of reproducibility being better for FOSS ANN method as compared to reference method is observed also for oil and moisture in rapeseed.

Table 2: Results from the 2017 WGN Ring test for wheat and barley.

WGN 2017 all samples (2016 harvest)	Ref. methods	local models	FOSS ANN
Protein, range		9.0 % - 14.8 %	
Mean (%)	11.90	11.97	11.93
deviation from mean		0.08	0.03
SD reproducibility	0.16	0.16	0.12
RSD reproducibility	1.4	1.4	1.0
Moisture, range		10.6 % - 15.3 %	
Mean (%)	13.17	13.15	13.10
deviation from mean		-0.02	-0.07
SD reproducibility	0.18	0.16	0.08
RSD reproducibility	1.3	1.2	0.6

Table 3: Results from the 2017 WGN Ring test for rapeseed.

WGN 2017 all samples (2016 harvest)	Ref. methods	local models	FOSS ANN
Oil, range		42.5 % - 52.7 %	
Mean (%)	48.73	48.78	48.80
deviation from mean		0.04	0.07
SD reproducibility	0.64	0.53	0.36
RSD reproducibility	1.3	1.1	0.7
Moisture, range		6.1 % - 8.6 %	
Mean (%)	6.96	6.98	6.96
deviation from mean		0.02	0.00
SD reproducibility	0.21	0.20	0.17
RSD reproducibility	3.0	2.8	2.5

From Table 2, it can be seen that the locally adjusted ANN prediction models deviate from reference methods about the same as for the global ANN prediction model based on the mean predicted values. This was not the case a few years back and it is clear that many of the national reference methods are now better aligned with the consequence that the locally adopted prediction models do not require major adjustment.

In some cases, the locally adopted ANN prediction model has been a different version or developed strictly on one grain type. The results of the global ANN prediction model strongly indicate that robustness and accuracy can be gained by not specializing in a certain grain type or adjusting to fit the national reference method. Thus, the essence of the ring test is to keep reference methods aligned and to indicate whether the local ANN prediction models really need adjustment, or if it is the reference method that requires adjustment.

The FOSS ANN calibration is based on a very large database covering a wide range in protein and moisture values, as well as broad variations in other aspects such as crop year, seasonal conditions, geography, grain type and variety and instrument and sample temperature. The inclusion of all these variables makes the ANN calibration extremely robust. The calibration set for WB003034 is outlined in Table 4.

Table 4: Number of samples (N) included and ranges covered in WB003034.

Parameter	N	Min	Max
Moisture	10 572	6.2 %	30.0 %
Protein (d.m.)	30 092	6.7 %	23.7 %

The calibration has been validated in accordance to ISO 12099 and EN15948:2012 using independent test sets of wheat and barley samples, originating from different parts of the world representing different classes, varieties and growing conditions. More than 50 independent validation sets originating from 14 countries were used to obtain the optimized ANN calibration. The goal was to be as strong as possible on overall accuracy, repeatability, transferability between instruments as well as the ability to handle grain temperature variations without showing any significant weaknesses in any of these areas. The overall performance is summarized in Table 5.

Table 5: Validation performance of WB003034 against a total test set.

Parameter	Grain type	N	Accuracy	Min	Max	RSQ
Moisture	All	4 600	0.24	7.8 %	29.9 %	0.99
Protein (d.m.)	All	11 822	0.27	6.9 %	24.0 %	0.99
Moisture	Barley	2251	0.22	8.6 %	27.9 %	0.99
Protein (d.m.)	Barley	3963	0.30	7.3 %	17.7 %	0.97
Moisture	Wheat	2349	0.25	7.8 %	29.9 %	0.99
Protein (d.m.)	Wheat	7857	0.27	6.8 %	24.0 %	0.99

N: Number of samples in the independent validation data set.
Accuracy: Overall accuracy expressed as SEP as constituent % w/w.
Min: Minimum value in the validation set.
Max: Maximum value in the validation set.
RSQ: Overall linear correlation coefficient between ANN predicted results and chemical reference analysis results.

Note that the values of accuracy and correlation given in Table 5 depend on the accuracy of the reference values. The predictions made with this model are without any bias correction.

In total, the independent test sets used consisted of 4,600 samples for moisture and 11,822 for protein. When such large data sets are used, it is inevitable that there is an influence from the reproducibility between laboratories due to uncontrolled variation between the different reference laboratories involved. Individual smaller independent test sets based on data from a single laboratory generally perform much better than the average of the total test set. In the example given in Table 6, all reference testing was done using a single laboratory.

Table 6: Validation performance of WB003034 against a single test set (Wheat, harvest 2007, one country, one reference laboratory).

Parameter	N	Accuracy	Min	Max	RSQ
Moisture	75	0.14	1.12%	23.70%	0.999
Protein (d.m.)	67	0.16	9.70%	16.30%	0.991

The combined Wheat and Barley calibration WB003034 is a combination of individual calibrations with typical labels WH003034 and BA003034, respectively. Other labels may exist with variants including other parameters. These options are provided for the convenience of being able to sort results by the different grain types.

The FOSS ANN calibration model RA002231 for the prediction of oil and moisture contents in whole rapeseeds is based on more than 5000 samples. A summary of the calibration set is given in Table 7.

Table 7: Number of samples (N) included and ranges covered in RA002231.

Parameter	N	Min	Max
Moisture (%)	5156	3.4 %	34.6 %
Oil (% d.m.)	5475	31.2 %	55.6 %

The rapeseed model and the wheat and barley model has been validated in accordance with EN 12099. A summary is given in Table 8.

Table 8: Validation performance of RA002231 against a total test set.

Parameter	N	Accuracy	Min	Max	RSQ
Moisture	2132	0.42	3.4%	25.8 %	0.97
Oil (d.m.)	2237	0.79	34.6 %	54.8 %	0.93

N: Number of samples in the independent validation data set.
Accuracy*: Overall accuracy expressed as SEP as constituent % w/w.
Min: Minimum value in the validation set.
Max: Maximum value in the validation set.
RSQ*: Overall linear correlation coefficient between ANN predicted results and chemical reference analysis results.

**NOTE: Depending on the accuracy of the reference values.*

The predictions made with this model are without any bias correction.

Discussion

The robustness of the ANN calibration is evident in its long term stability. In Figure 1, the stability of the protein prediction for wheat and barley during the past eleven years is illustrated. The stability plot for moisture prediction is shown in Figure 2.

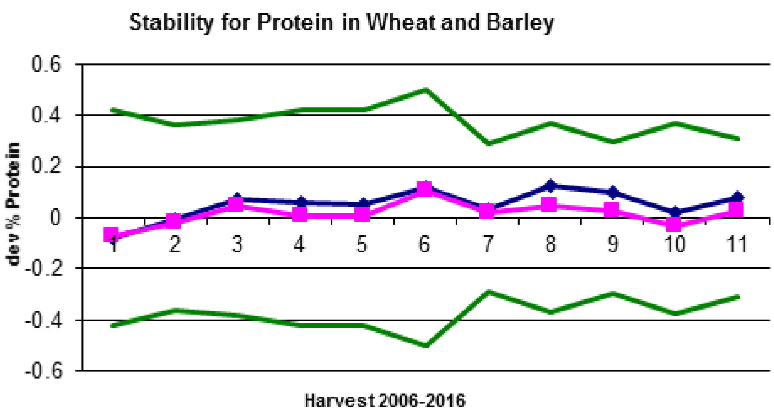

Figure 1: Average deviations of predicted protein results from the best estimate of the true value during the past ten years. Pink = global ANN and dark blue=local ANN.

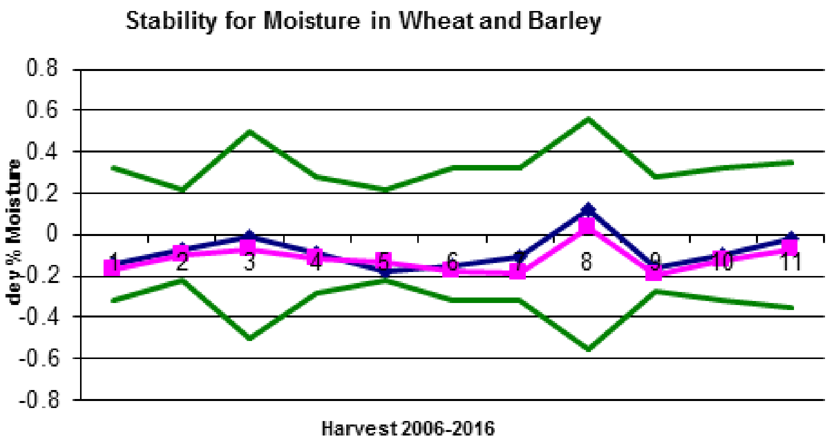

Figure 2: Average deviations of predicted moisture results from the best estimate of the true value during the past ten years. Pink =global ANN and dark blue=local ANN.

The key to the superior stability is two-fold: the extremely large calibration database covering the widest range of all possible variations and the ANN technology that captures all variations in an optimal way.

As mentioned in the previous section, the locally adjusted prediction models have, over the last couple of years, become better aligned with the reference values. This is the same tendency as already observed with the global ANN calibration for some time now. This is seen for both protein and moisture of wheat and barley in Figures 1 and 2, respectively. Adjustments to the prediction models should only be applied when the reference laboratory is aligned with respect to other approved laboratories.

The stability for oil and moisture in rapeseed for a period of six years is shown in Figures 3 and 4, respectively.

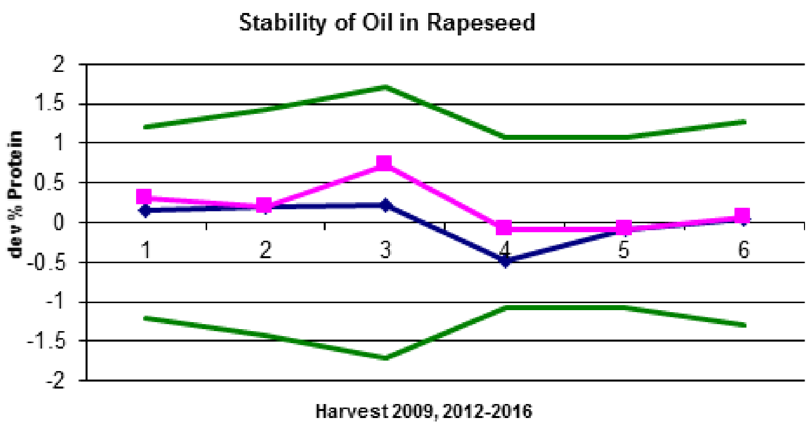

Figure 3: Average deviations of predicted oil results from the best estimate of the true value during the six years. Pink = global ANN and dark blue=local ANN.

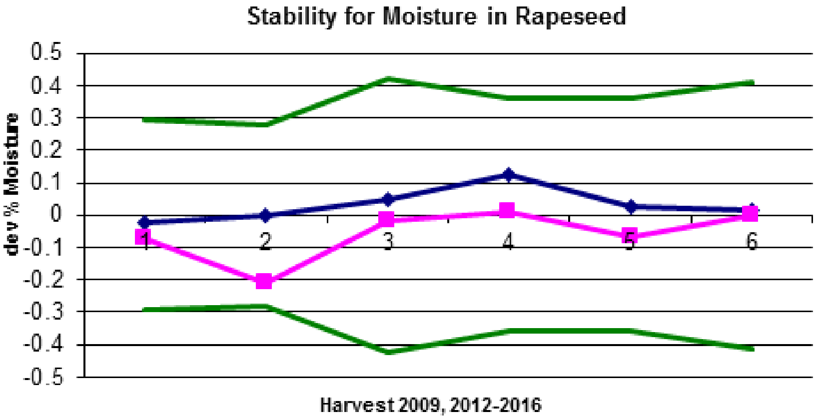

Figure 4: Average deviations of predicted moisture results from the best estimate of the true value during the six years. Pink = global ANN and dark blue=local ANN.

The same trends on stability as for protein and moisture in wheat and barley can also be seen for oil and moisture in rapeseed. Both the FOSS ANN and the locally adopted models have become better aligned with the reference method recently compared to when rapeseed was first included in the study.

Conclusion

The high performance of the ANN prediction model is further demonstrated by results from this updated study including global data when compared to those of the original report. As with the original study, this is particularly clear from the inter-laboratory study in 2017 for determination of protein and moisture in whole kernels, where ten wheat and ten barley samples from the 2016 harvest were used. The FOSS ANN model WB003034 was used for simultaneous determination of protein and moisture in wheat and barley. The results given in Table 2 clearly show that the FOSS ANN calibration can be used without loss of accuracy and performance.

The FOSS ANN prediction model WB003034 is approved as a European standard for the simultaneous prediction of protein and moisture content in whole grain of wheat and barley. This is described in more detail in EN 15948:2012.

The dedicated FOSS solution with the Infratec grain analyzer combined with the ANN prediction model WB003034 remains the optimal choice for prediction of protein and moisture content in whole grain of wheat and barley. The stability of the model also holds great promise as a platform for effective grain analysis in the future.

The inclusion of rapeseed in the study also shows great accuracy and stability for the FOSS ANN calibration of oil and moisture. This emphasizes the importance of the ring test to facilitate the alignment between reference methods across the globe and the means to assess whether adjustments of the FOSS ANN models are required.

References

1. EN ISO 12099:2010, *Animal feeding stuffs, cereals and milled cereal products – Guidelines for the application of near infrared spectrometry*
2. EN 15948:2012, *Cereals – Determination of moisture and protein – Method using Near-Infrared Spectroscopy in whole kernels*

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