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BSI Standards Publication

Cereals — Determination of moisture and protein — Method using Near-Infrared-Spectroscopy in whole kernels

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National foreword

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The UK participation in its preparation was entrusted to Technical Committee AW/4, Cereals and pulses.

A list of organizations represented on this committee can be obtained on request to its secretary.

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Cereals - Determination of moisture and protein - Method using Near-Infrared-Spectroscopy in whole kernels

Céréales - Détermination de la teneur en eau et en protéines - Méthode utilisant la spectroscopie dans le proche infrarouge sur des grains entiers

Getreide - Bestimmung der Feuchte und des Proteins - Verfahren der Nahinfrarot-Spektroskopie bei ganzen Körnern

This European Standard was approved by CEN on 5 January 2015.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
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CEN-CENELEC Management Centre: Avenue Marnix 17, B-1000 Brussels

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Foreword

This document (EN 15948:2015) has been prepared by Technical Committee CEN/TC 338 “Cereal and cereal products”, the secretariat of which is held by AFNOR.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by October 2015, and conflicting national standards shall be withdrawn at the latest by October 2015.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

This document supersedes EN 15948:2012.

The following modifications were made in this new edition:

— Annexes have been enhanced with information on 2 other NIR instruments based on the results of the interlaboratory tests and the models of prediction.

According to the CEN-CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

1 Scope

This European Standard defines a routine method for the determination of moisture and protein in whole kernels of barley and wheat using a near-infrared spectrophotometer in the constituent ranges:

a) for wheat:

1) moisture content minimum range from 8 % to 22 %;

2) protein content minimum range from 7 % to 20 %.

b) for barley:

1) moisture content minimum range from 8 % to 22 %;

2) protein content minimum range from 7 % to 16 %.

This European Standard describes the modalities to be implemented by the supplier (5.3 and 5.4) and the user of the method.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN ISO 12099:2010, *Animal feeding stuffs, cereals and milled cereal products — Guidelines for the application of near infrared spectrometry (ISO 12099:2010)*

ISO 5725-2, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in EN ISO 12099:2010 apply.

4 Principle

The method is based on Near-Infrared (NIR) spectroscopy, an indirect, correlative technique to predict the concentration of various constituents in organic samples. Linear or non-linear regression modelling is used to relate NIR spectra to moisture or protein concentrations determined by officially approved standard methods (e.g. artificial neural network - ANN, Partial Least Square Regression - PLS).

5 Method of analysis

5.1 General

According to this document, the method of analysis is defined as the association between a NIR instrument and a model of prediction.

5.2 Near Infrared Instrument

Based on diffuse reflectance or transmittance measurement covering the wavelength region of 700 nm to 2 500 nm or segments of this or at selected wavelengths.

5.3 Prediction models

Each model for the prediction of protein and moisture contents in whole grain of wheat and barley is amongst others defined by:

— the number of samples used for the calibration development;

— the constituent ranges covered in the model for moisture and protein;

— the temperature range of the samples;

— the number and performance of involved reference labs;

— the stability of the model i.e. by number of harvests covered;

— the calibration file defined by its name and its IT name (for example CHECKSUM) insuring its integrity;

— the seasonal, geographic and genetic variations covered.

5.4 Initial validation of the model

5.4.1 General

Since NIR analysis is an indirect, correlative technique, the results shall be validated against chemical analysis reference methods. It is important that the reference methods used are officially approved such as the methods described in the standards previously cited (Clause 2). The purpose of validation is to determine the root mean square error of prediction which depends at the same time on the correlation, the bias and the slope.

The root mean square error between chemical analysis methods and predictions shall be compared to calibration performance specifications and/or historical performance.

5.4.2 Initial validation sample set

The initial validation of a calibration model shall be done in accordance with EN ISO 12099 using independent test sets of wheat and barley samples, originating from different countries and analyzed by the reference methods given in Clause 2.

Requirements for the validation sample set are:

— at least 200 samples coming from 10 countries (20 representative samples min/country) distributed homogeneously over the entire constituent range;

— the part of the range without any reference sample shall not exceed 0,3 %;

— different scans from one sample shall not be considered as different samples;

— seasonal effects over at least a three year period, temperature effects, instrument variation and the variability of reference data shall be included in the set.

5.4.3 Initial validation performances

The results of the initial validation shall at least fulfil the specifications given in Table 1.

Table 1 — NIR performances for the determination of moisture and protein (see also Annex B)

		Moisture Wheat and barley	Protein Wheat	Protein Barley
Overall accuracy expressed as SEP as constituent % w/w		0,24 %	0,27 %	0,27 %
Constituent concentration in the independent validation data set	Min	8 %	7 % d.m.	7 % d.m.
	Max	22 %	20 % d.m.	16 % d.m.

NOTE The minimum performance given in Table 1 includes the variation of reference data as documented by the number of reference labs involved, regional and genetic variations, the number of countries and crop species involved and the robustness over the last five years (see also Annex B).

5.5 Update of calibration model and validation of new model

The prediction model in accordance with this standard shall be updated by the one issuing the calibration model to ensure inclusion of new climatic crop conditions and new varieties introduced on the market. These updates shall be made by keeping the original database with addition of the new samples as needed.

The new prediction model shall be updated according to EN ISO 12099.

Validation shall be made according to the initial validation (5.4) and include at least 20 new samples.

6 Sampling

Sampling is not part of the method specified in this European Standard. A recommended sampling procedure is given in EN ISO 24333 [5].

It is important that the sample analyzed in routine is truly representative for the batch and has not been damaged or modified.

7 Procedure

7.1 Preparation of the test sample

No specific sample preparation is required.

7.2 Measurement

Follow the instructions of the instrument manufacturer.

7.3 Local validation of the method

Before use, the method shall be validated on an independent test set that is representative of the sample population to be analyzed. For the determination of bias, at least 10 samples are needed; for the determination of Standard Error of Prediction (SEP, see EN ISO 12099:2010, 6.5) at least 20 samples are

needed. Validation shall be carried out for each sample type, constituent/ parameter and temperature (see EN ISO 12099:2010, 5.4).

Bias or inherent systematic error, as described in EN ISO 12099:2010, Clause 6, is exhibited when the predicted results of a specific sample group or product show a mean offset value when compared to their reference values. This may occur with unique sample types.

The bias (i.e. mean difference between the chemical analysis results and the predicted results) may or may not be statistically significant. Based on the procedure described in EN ISO 12099, a bias confidence limit may be calculated.

When this limit is exceeded, a bias is implemented in the instrument software and the validation process repeated. Refer to the manufacturer instructions and to EN ISO 12099 for procedure.

7.4 Periodical adjustment of the instrument

To ensure its accuracy, each instrument shall be checked at least annually, against the reference method, either directly or through a master instrument.

The execution of this check shall be performed on samples covering a range as wide as possible, taking into account seasonal, geographical and genetic variations.

The number of samples for the adjustment should be sufficient for the statistics used to check the performance. For the determination of the bias, at least 10 samples are needed, for the determination of standard error of prediction (SEP) and for the slope adjustment, at least 20 samples are needed.

7.5 Checking instrument stability

See EN ISO 12099:2010, Clause 9.

7.6 Follow up of method performance

Performance of the method shall be checked at least annually, against reference methods to secure the constant adequacy of the model with the requirements of this standard (see 5.4.2).

This performance test shall be made on samples selected from the pool of analyzed samples. It may be necessary to resort to some sampling strategy to ensure a balanced sample distribution over the entire calibration range and to ensure that samples with a commercially important range are covered. At least 20 samples are needed (to expect a normal distribution of variance).

For instruments operated in a network and adjusted against a master instrument, it is sufficient to run the performance check of the method of this last one.

The adjustment (7.4) respecting the requirements of this clause may be used for the follow-up of the method performance.

It is recommended to participate in an internationally accepted proficiency testing scheme (PTS) that includes NIRS predicted results and results generated by following the standards specified in Clause 2.

8 Calculation and expression of results

The software of the instrument calculates the results for moisture and protein and displays them in % w/w (g/100 g) to two decimal places.

If multiple measurements are made on the same sample, calculate the arithmetic mean.

Express final results to two decimal places.

9 Accuracy and precision of the method

9.1 Accuracy

The accuracy of the prediction model is determined by validation in accordance with EN ISO 12099 and expressed by the Standard Error of Prediction (see EN ISO 12099:2010, Table 1). The Standard Error of Prediction (SEP) is an expression of the bias corrected average difference between predicted and reference values predicted by the model when applied to a set of samples not included in the derivation of the model. The values also include the uncertainty of reference results.

The predicted results will not in more than 5 % of cases deviate more than 1,96 x SEP (as determined in the paragraph above) from the best estimate of the true value.

NOTE As NIR is an indirect method, the typical standard deviation of reproducibility for the used reference methods are given here for comparison:

— moisture (EN ISO 712) = 0,16 %;

— protein (EN ISO 20483) = 0,20 %;

— protein (EN ISO 5983-2) = 0,20 %;

— protein (CEN ISO/TS 16634-2) = 0,21-0,26 %.

9.2 Precision

9.2.1 General

The precision of the prediction model shall be determined from an interlaboratory test organized according to ISO 5725-2 and at least fulfil the performance criteria of repeatability and reproducibility given below.

Details of an example of an interlaboratory test are summarized in Annex A. The precision data given below are derived from this example.

Figure A.1 and Figure A.2 show that the repeatability and the reproducibility are independent of the concentration. The figures in Annex B show that the dispersion is identical over the validated range (Figure B.1 and Figure B.2). Therefore the model can be used in the whole validated range, even though the interlaboratory trial covered a smaller range.

9.2.2 Repeatability

The absolute difference between two independent single test results, obtained using the same method on identical test material in the same laboratory by the same operator using the same equipment within a short interval of time will not in more than 5 % of cases be greater than the repeatability limit r ($r=s_r x 2,8$) with:

$r_{protein}$ = 0,42 %

$r_{moisture}$ = 0,15 %

9.2.3 Reproducibility

The absolute difference between two single test results, obtained using the same method on identical test material in different laboratories with different operators using different equipment, will not in more than 5 % of cases be greater than the reproducibility limit R ($R=s_R x 2,8$) with:

$R_{protein}$ = 0,45 %

$R_{moisture}$ = 0,25 %

9.2.4 Critical difference

9.2.4.1 General

When the difference between two averaged values obtained from two test results under repeatability or reproducibility conditions shall be assessed, the repeatability or reproducibility limit cannot be used, one shall use the Critical Difference (CD).

9.2.4.2 Comparison of two groups of measurements in one laboratory

The critical difference (CD) between two averaged values obtained from two test results under repeatability conditions is equal to:

$$CD = 2,8\, s_r \sqrt{\frac{1}{2n1} + \frac{1}{2n2}} = 2,77\, s_r \sqrt{\frac{1}{2}} = 1,98\, S_r$$

where

s_r is the standard deviation of repeatability;

$n1$ and $n2$ are the numbers of test results corresponding to each of the averaged values;

CD_r (protein) = 0,30;

CD_r (moisture) = 0,11.

9.2.4.3 Comparison of two groups of measurements in two laboratories

The critical difference (CD) between two averaged values obtained in two different laboratories from two test results under repeatability conditions is equal to:

$$CD = 2,8\sqrt{s_R^2 - s_r^2\left(1 - \frac{1}{2n_1} - \frac{1}{2n_2}\right)} = 2,8\sqrt{s_R^2 - 0,5 s_r^2}$$

where

s_r is the standard deviation of repeatability;

s_R is the standard deviation of reproducibility;

n_1 and n_2 are the number of test results corresponding to each of the averaged values;

CD_R (protein) = 0,32;

CD_R (moisture) = 0,23.

10 Test Report

The test report shall specify:

a) all information necessary for the complete identification of the sample;

b) the sampling method used (if known);

c) the application model and instrument used with reference to this European Standard;

d) all operating details not specified in this European Standard, or regarded as optional, together with details of any incidents which may have influenced the test result(s);

e) the test result(s) obtained.

Annex A
(informative)

Results of examples of interlaboratory test

A.1 FOSS interlaboratory test

An interlaboratory test, organized by the company FOSS Analytical AB (Sweden) in 2008, involving 20 participants from 12 countries was carried out on 6 wheat and 4 barley samples from the 2007 harvest, containing protein and moisture in various concentrations. The participants were the master labs of European grain networks. The grain networks did also assist in the collection of the samples (Table A.1).

The results obtained were subjected to statistical analysis in accordance with ISO 5725-1 and ISO 5725-2 to calculate the precision data shown in Table A.2 to Table A.5.

Table A.1 — Samples for the interlaboratory study

Sample	Description	Country of origin
B1	Spring barley (2-row, malting barley)	UK
B2	Spring barley (2-row, feed barley)	Denmark
B3	Spring barley (2-row, malting barley)	Denmark
B4	Winter barley (6-row, malting barley)	France
W1	Spring wheat (hard)	Germany
W2	Spring wheat (hard)	France
W3	Winter wheat (hard)	UK
W4	Spring wheat (soft)	Germany
W5	Winter wheat (hard)	Italy
W6	Durum wheat	Italy

Table A.2 — Results of statistical analysis for the determination of the protein content in wheat by the ANN model WB003034

Sample	WG 1	WG 2	WG 3	WG 4	WG 5	WG 6
Number of laboratories	20	20	20	20	20	20
Mean predicted protein content (% d.m.)	16,883	11,789	13,047	10,876	14,985	14,173
Repeatability standard deviation s_r (% P)	0,159[a]	0,113[a]	0,099	0,106	0,087	0,109[a]
Repeatability relative stand. dev. s_r %	0,943	0,958	0,76	0,979	0,583	0,771
Repeatability limit r [r = 2,8 x s_r], %	0,440	0,313	0,274	0,294	0,241	0,302
Reproducibility stand. dev. s_R (% P)	0,159	0,113	0,107	0,143	0,125	0,109
Reproducibility relative stand. dev. s_R %	0,943	0,958	0,819	1,317	0,834	0,771
Reproducibility limit R [R = 2,8 x s_R], %	0,440	0,313	0,296	0,396	0,346	0,302
Best estimate of true protein value (%)[b]	16,88	11,75	13,08	10,86	14,93	14,50
Critical difference (n=2), reference methods	0,31	0,22	0,22	0,34	0,30	0,21
Deviation predicted - true value (%)	0,01	0,04	0,03	0,01	0,06	0,33

a $s_r > s_R$, s_R set to be equal s_r.

b Average protein value (after elimination of outliers) generated by 17 Master labs of the European grain networks, using Kjeldahl (EN ISO 20483 or EN ISO 5983-2) and Dumas (CEN ISO/TS 16634-2) methods.

Table A.3 — Results of statistical analysis for the determination of the protein content in barley by the ANN model WB003034

Sample	B 1	B 2	B 3	B 4
Number of laboratories	20	20	20	20
Mean predicted protein content in %	10,964	13,05	11,15	12,772
Repeatability standard deviation s_r (% P)	0,198[a]	0,169[a]	0,123	0,343[a]
Repeatability relative stand. dev. s_r %	1,804	1,292	1,105	2,682
Repeatability limit r [r = 2,8 x s_r], %	0,548	0,468	0,341	0,95
Reproducibility stand. dev. s_R (% P)	0,198	0,169	0,151	0,343
Reproducibility relative stand. dev. s_R %	1,804	1,292	1,352	2,682
Reproducibility limit R [R = 2,8 x s_R], %	0,548	0,468	0,418	0,950
Best estimate of true protein value (%)[b]	10,66	13,11	11,27	12,97
Critical difference (n=2), reference methods	0,39	0,33	0,34	0,67
Deviation predicted - true value (%)	0,30	-0,06	-0,12	-0,20

a $s_r > s_R$, s_R set to be equal s_r.

b Average protein value (after elimination of outliers) generated by 17 Master labs of the European grain networks, using Kjeldahl (EN ISO 20483 or EN ISO 5983-2) and Dumas (CEN ISO/TS 16634-2) methods.

Table A.4 — Results of statistical analysis for the determination of the moisture content in wheat by ANN model WB003034

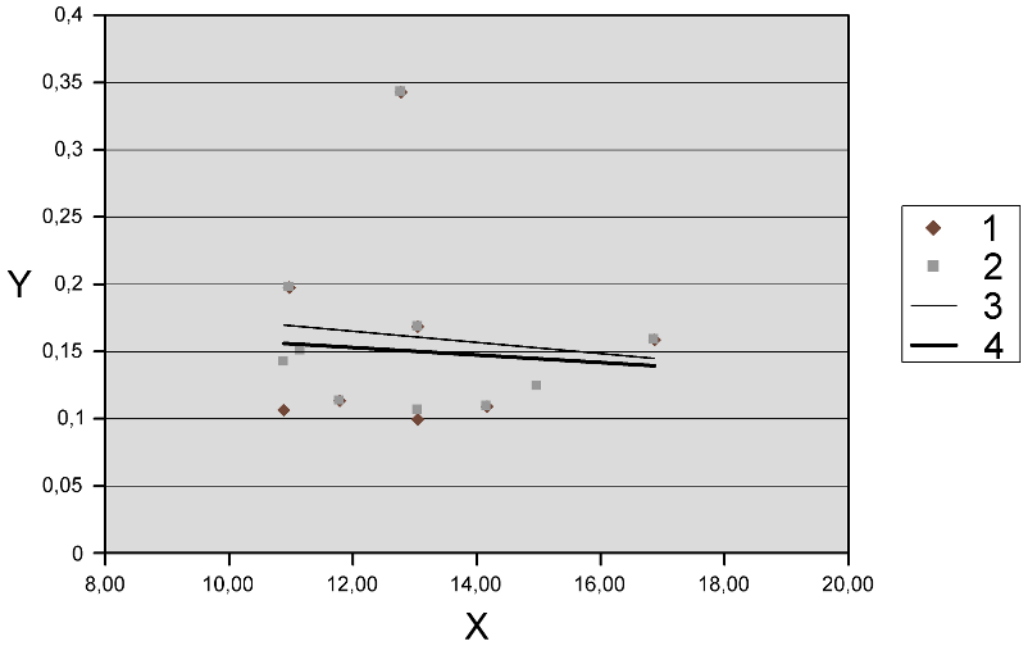
Sample	WG 1	WG 2	WG 3	WG 4	WG 5	WG 6
Number of laboratories	20	20	20	20	20	20
Mean predicted moisture content in %	14,125	13,415	13,978	13,764	11,102	11,074
Repeatability standard dev s_r (% H_2O)	0,024	0,025	0,030	0,016	0,015	0,019
Repeatability relative stand. dev. s_r %	0,170	0,187	0,215	0,116	0,138	0,167
Repeatability limit r [r = 2,8 x s_r], %	0,066	0,069	0,083	0,044	0,042	0,053
Reproducibility stand. dev. s_R (% H_2O)	0,077	0,07	0,090	0,079	0,045	0,047
Reproducibility relative stand. dev. s_R %	0,546	0,523	0,642	0,573	0,405	0,422
Reproducibility limit R [R = 2,8 x s_R], %	0,213	0,194	0,249	0,219	0,125	0,130
Best estimate of true moisture value (%)[a]	14,24	13,61	14,08	13,93	11,35	11,23
Critical difference (n=2), reference methods	0,20	0,28	0,26	0,34	0,27	0,56
Deviation predicted - true value (%)	-0,11	-0,20	-0,10	-0,17	-0,25	-0,15

[a] Average moisture value (after elimination of outliers) generated by 17 Master labs of the European grain networks, using EN ISO 712 method.

Table A.5 — Results of statistical analysis for the determination of the moisture content in barley by ANN model WB003034

Sample	B1	B2	B3	B4
Number of laboratories	20	20	20	20
Mean predicted moisture content in %	12,417	13,731	14,299	12,986
Repeatability standard dev s_r (% H_2O)	0,103	0,053	0,054	0,213
Repeatability relative stand. dev. s_r %	0,827	0,384	0,377	1,639
Repeatability limit r [r = 2,8 x s_r], %	0,285	0,147	0,150	0,590
Reproducibility stand. dev. s_R (% H_2O)	0,111	0,071	0,087	0,238
Reproducibility relative stand. dev. s_R %	0,896	0,517	0,611	1,836
Reproducibility limit R [R = 2,8 x s_R], %	0,307	0,197	0,241	0,659
Best estimate of true moisture value (%)[a]	12,51	13,79	14,37	12,83
Critical difference (n=2), reference methods	0,14	0,29	0,30	0,16
Deviation predicted - true value (%)	-0,09	-0,06	-0,07	0,16

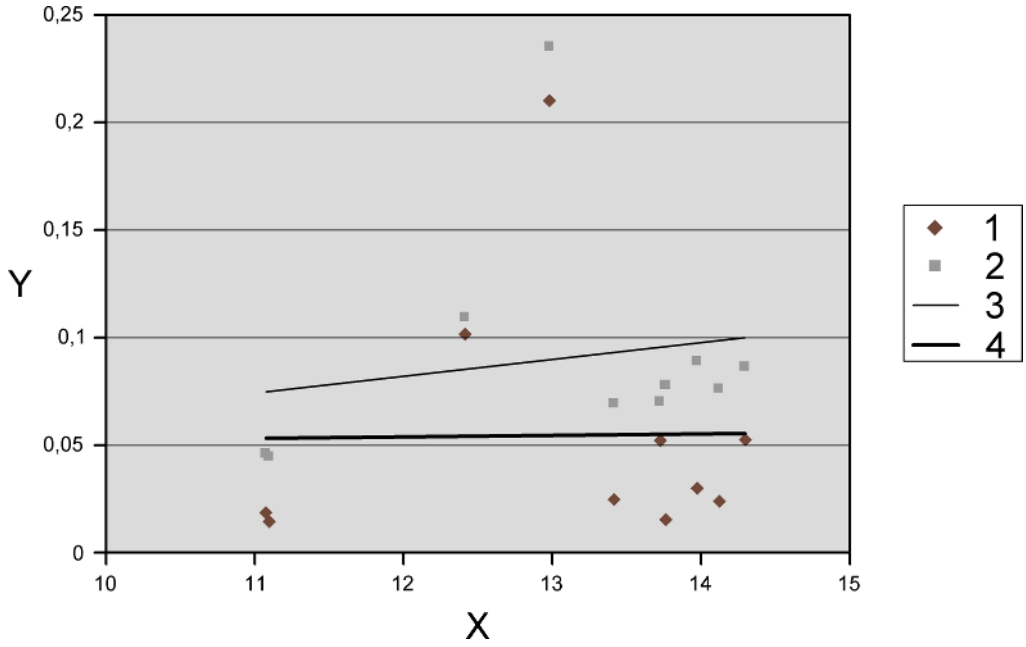
[a] Average moisture value (after elimination of outliers) generated by 17 Master labs of the European grain networks, using EN ISO 712 method.



Key

X	% protein (dm)
Y	s%
1	◆ s_r
2	□ S_R
3	Linear (s_R)
4	Linear (s_r)

Figure A.1 — Standard deviations for the repeatability, s_r, and reproducibility, s_R, as a function of the protein content



Key

X % moisture

Y s%

1 ◆ s_r

2 □ S_R

3 —— Linear (s_R)

4 ——— Linear (s_r)

Figure A.2 — Standard deviations for the repeatability, s_r, and reproducibility, s_R, as a function of the moisture content

A.2 PERTEN interlaboratory test

An interlaboratory test, organized by the company Perten Instruments AB (Sweden) in 2013, involving 8 participants from 6 countries was carried out on 6 wheat and 4 barley samples from the 2012 harvest, containing protein and moisture in various concentrations. The participants were the master or reference labs of European grain networks. The grain networks did also assist in the collection of the samples (Table A.6).

The results obtained were subjected to statistical analysis in accordance with ISO 5725-2 to calculate the precision data shown in Table A.7 to Table A.10.

Table A.6 — Samples for the interlaboratory study

Sample	Description	Country of origin
B1	Winter barley	France
B2	Spring barley (blind duplicate to B4)	Germany
B3	Spring barley	Sweden
B4	Spring barley (blind duplicate to B2)	Germany
W1	Winter wheat	Sweden
W2	Wheat	United Kingdom
W3	Winter wheat	Germany
W4	Winter wheat	Germany
D1	Durum wheat	France
D2	Durum wheat	Italy

Table A.7 — Results of statistical analysis for the determination of the protein content in wheat by Inframatic 9500 Wheat Calibrations Version 5, March 19, 2013

Sample	W 1	W 2	W 3	W 4	D 1	D 2
Number of laboratories	8	8	8	8	8	8
Mean predicted protein content (% d.m.)	11,17	11,70	14,88	14,88	13,12	17,13
Repeatability standard deviation s_r (% P)	0,141	0,138	0,138	0,139	0,090	0,175
Repeatability relative stand. dev. s_r %	1,266	1,182	0,931	0,936	0,684	1,019
Repeatability limit r [r = 2,8 x s_r], %	0,396	0,387	0,388	0,390	0,251	0,489
Reproducibility stand. dev. s_R (% P)	0,141	0,209	0,160	0,171	0,166	0,200
Reproducibility relative stand. dev. s_R %	1,266	1,789	1,078	1,149	1,269	1,168
Reproducibility limit R [R = 2,8 x s_R], %	0,396	0,586	0,449	0,479	0,466	0,560
Critical difference (n=2)	0,17	0,31	0,21	0,23	0,26	0,26
Best estimate of true protein value (%)[a]	11,35	12,10	14,64	14,90	13,46	16,78
Deviation predicted - true value (%)	-0,18	-0,40	0,23	-0,02	-0,34	0,36

[a] Average protein value generated by 3 reference labs of European grain networks, using Kjeldahl (ICC 105/2) or Dumas (ICC 167 or CEN ISO/TS 16634-2) methods.

Table A.8— Results of statistical analysis for the determination of the protein content in barley by Inframatic 9500 Barley Calibrations Version 6, March 19, 2013

Sample	B 1	B 2	B 3	B 4
Number of laboratories	8	8	8	8
Mean predicted protein content in %	10,15	9,22	11,39	9,24
Repeatability standard deviation s_r (% P)	0,152	0,107	0,166	0,095
Repeatability relative stand. dev. s_r %	1,502	1,162	1,457	1,032
Repeatability limit r [r = 2,8 x sr], %	0,427	0,300	0,465	0,267
Reproducibility stand. dev. s_R (% P)	0,171	0,119	0,187	0,095
Reproducibility relative stand. dev. s_R %	1,686	1,291	1,639	1,032
Reproducibility limit R [R = 2,8 x s_R], %	0,479	0,333	0,523	0,267
Critical difference (n=2)	0,22	0,15	0,24	0,11
Best estimate of true protein value (%)[a]	10,67	9,37	11,61	9,45
Deviation predicted - true value (%)	-0,52	-0,14	-0,22	-0,21

[a] Average protein value generated by 3 reference labs of the European grain networks, using Kjeldahl (ICC 105/2) and Dumas (ICC 167 or CEN ISO/TS 16634-2) methods.

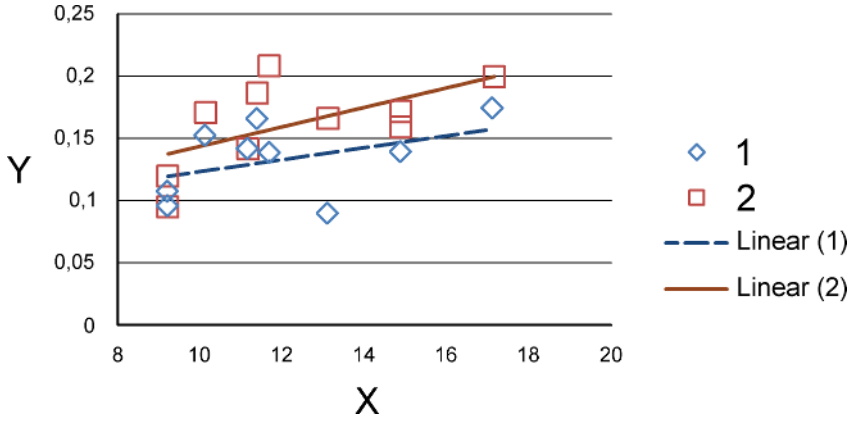
Table A.9 — Results of statistical analysis for the determination of the moisture content in wheat by Inframatic 9500 Wheat Calibrations Version 5, March 19, 2013

Sample	W 1	W 2	W 3	W 4	D 1	D 2
Number of laboratories	8	8	8	8	8	8
Mean predicted moisture content in %	13,94	9,53	14,18	14,16	12,95	9,76
Repeatability standard dev s_r (% H_2O)	0,021	0,027	0,025	0,020	0,034	0,014
Repeatability relative stand. dev. s_r %	0,148	0,285	0,176	0,142	0,265	0,145
Repeatability limit r [r = 2,8 x s_r], %	0,058	0,076	0,070	0,056	0,096	0,040
Reproducibility stand. dev. s_R (% H_2O)	0,038	0,082	0,057	0,060	0,045	0,064
Reproducibility relative stand. dev. s_R %	0,269	0,860	0,403	0,425	0,347	0,655
Reproducibility limit R [R = 2,8 x s_R], %	0,105	0,229	0,160	0,169	0,126	0,179
Critical difference (n=2)	0,06	0,13	0,09	0,10	0,06	0,10
Best estimate of true moisture value (%)[a]	13,75	9,63	13,96	14,14	12,85	9,68
Deviation predicted - true value (%)	0,19	-0,11	0,22	0,02	0,10	0,07

[a] Average moisture value generated by 3 reference labs of European grain networks, using EN ISO 712/ICC 110/1.

Table A.10 — Results of statistical analysis for the determination of the moisture content in barley by Inframatic 9500 Barley Calibrations Version 6, March 19, 2013

Sample	B1	B2	B3	B4
Number of laboratories	8	8	8	8
Mean predicted moisture content in %	12,53	13,10	13,92	13,09
Repeatability standard dev s_r (% H_2O)	0,036	0,021	0,029	0,017
Repeatability relative stand. dev. s_r %	0,286	0,157	0,209	0,132
Repeatability limit r [r = 2,8 x s_r], %	0,100	0,058	0,081	0,048
Reproducibility stand. dev. s_R (% H_2O)	0,069	0,033	0,078	0,036
Reproducibility relative stand. dev. s_R %	0,549	0,254	0,557	0,277
Reproducibility limit R [R = 2,8 x s_R], %	0,193	0,093	0,217	0,102
Critical difference (n=2)	0,11	0,05	0,12	0,06
Best estimate of true moisture value (%)[a]	12,45	12,94	13,91	12,96
Deviation predicted - true value (%)	0,08	0,16	0,01	0,12
a Average moisture value generated by 3 reference labs of the European grain networks, using EN ISO 712/ICC 110/1.				



Key

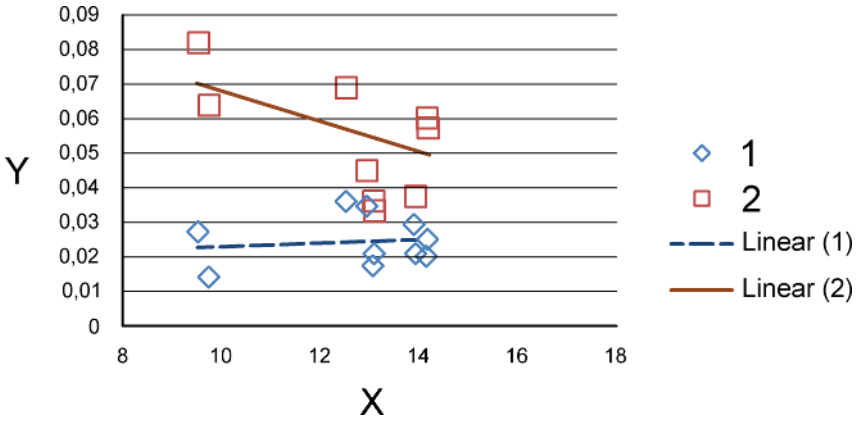
X % protein (dm)

Y standard deviation%

1 Sr

2 S_R

Figure A.3 — Standard deviations for the repeatability, s_r, and reproducibility, s_R, as a function of the protein content



Key

X moisture content%

Y standard deviation %

1 Sr

2 S_R

Figure A.4 — Standard deviations for the repeatability, s_r, and reproducibility, s_R, as a function of the moisture content

A.3 CHOPIN Technologies interlaboratory test

An interlaboratory test, organized by the company CHOPIN Technologies (France) in 2012, involving 13 participants from 7 countries was carried out on 16 wheat and 15 barley samples from the 2011 harvest, containing protein and moisture in various concentrations. The participants did assist in the collection of the samples (Table A.11 and Table A.12).

The results obtained were subjected to statistical analysis in accordance with ISO 5725-1 and ISO 5725-2 to calculate the precision data shown in Table A.13 to Table A.16 and Figures A.5 to A.8.

Table A.11 — Wheat Samples for the interlaboratory study

Sample	Description	Country of origin
W1	Common wheat	France
W2	Common wheat	France
W3	Common wheat	France
W4	Common wheat	France
W5	Common wheat	France
W6	Durum wheat	Italy
W7	Common wheat	Turkey
W8	Common wheat	France
W9	Common wheat	France
W10	Common wheat	France
W11	Durum wheat	France
W12	Durum wheat	France
W13	Common wheat	France
W14	Common wheat	France
W15	Common wheat	France
W16	Common wheat	Argentina

Table A.12 — Barley Samples for the interlaboratory study

Sample	Description	Country of origin
B1	Barley	France
B2	Spring barley	France
B3	Winter barley	France
B4	Barley	France
B5	Barley	France
B6	Barley	France
B7	Barley	France
B8	Barley	France
B9	Barley	France
B10	Winter Barley	France
B11	Spring barley	France
B12	Spring barley	France
B13	Barley	France
B14	Winter barley	Germany
B15	Spring barley	Argentina

Table A.13 a) — Results of statistical analysis for the determination of the protein content in wheat by Infraneo PLS model bl-pro03.cal

Sample	W1	W2	W3	W4	W5	W6	W7	W8
Number of laboratories	11	12	11	11	11	11	11	11
Mean predicted protein content (% d.m.)	10,97	15,45	11,27	12,01	16,55	14,18	10,97	10,99
Repeatability standard deviation s_r (% P)	0,064	0,106	0,055	0,079	0,088	0,063	0,091	0,125
Repeatability relative stand. dev. s_r %	0,555	0,685	0,492	0,660	0,532	0,443	0,828	1,139
Repeatability limit r [r = 2,8 x s_r], %	0,178	0,296	0,155	0,222	0,247	0,176	0,254	0,350
Reproducibility stand. dev. s_R (% P)	0,115	0,168	0,130	0,130	0,123	0,084	0,133	0,132
Reproducibility relative stand. dev. s_R %	1,005	1,086	1,150	1,083	0,742	0,592	1,209	1,203
Reproducibility limit R [R = 2,8 x s_R], %	0,323	0,470	0,363	0,364	0,344	0,235	0,371	0,370
True protein value (%)[a]	11,05	15,10	11,50	11,90	16,35	14,10	11,20	10,85
Deviation predicted - true value (%)	-0,08	0,35	-0,23	0,11	0,20	0,08	-0,23	0,14

[a] Protein value obtain by 1 Master labs of the European grain networks, using Kjeldahl (EN ISO 20483) method.

Table A.13 b) — Results of statistical analysis for the determination of the protein content in wheat by Infraneo PLS model bl-pro03.cal

Sample	W9	W10	W11	W12	W13	W14	W15	W16
Number of laboratories	11	11	11	11	11	11	11	11
Mean predicted protein content (% d.m.)	9,97	11,92	11,91	13,29	16,34	14,82	13,53	18,55
Repeatability standard deviation s_r (% P)	0,101	0,085	0,060	0,080	0,088	0,070	0,101	0,088
Repeatability relative stand. dev. s_r %	1,012	0,716	0,502	0,601	0,541	0,469	0,744	0,474
Repeatability limit r [r = 2,8 x s_r], %	0,283	0,239	0,168	0,223	0,247	0,195	0,282	0,246
Reproducibility stand. dev. s_R (% P)	0,136	0,131	0,113	0,119	0,166	0,127	0,118	0,152
Reproducibility relative stand. dev. s_R %	1,365	1,100	0,949	0,892	1,018	0,857	0,875	0,820
Reproducibility limit R [R = 2,8 x s_R], %	0,381	0,367	0,317	0,332	0,466	0,355	0,332	0,426
True protein value (%)[a]	9,95	12,00	12,20	13,45	16,45	15,20	13,45	18,30
Deviation predicted - true value (%)	0,02	-0,08	-0,29	-0,16	-0,11	-0,38	0,08	0,25

[a] Protein value obtain by 1 Master labs of the European grain networks, using Kjeldahl (EN ISO 20483) method.

Table A.14 a) — Results of statistical analysis for the determination of the protein content in barley by Infraneo PLS model or-pro03.cal

Sample	B1	B2	B3	B4	B5	B6	B7	B8
Number of laboratories	11	11	11	11	11	11	11	11
Mean predicted protein content (% d.m.)	10,93	11,80	12,08	10,41	11,74	11,85	10,09	12,52
Repeatability standard deviation s_r (% P)	0,096	0,090	0,104	0,086	0,134	0,106	0,119	0,141
Repeatability relative stand. dev. s_r %	0,874	0,762	0,859	0,830	1,139	0,897	1,175	1,127
Repeatability limit r [r = 2,8 x s_r], %	0,267	0,252	0,290	0,242	0,375	0,298	0,332	0,395
Reproducibility stand. dev. s_R (% P)	0,121	0,101	0,134	0,086	0,162	0,123	0,148	0,208
Reproducibility relative stand. dev. s_R %	1,104	0,859	1,110	0,830	1,383	1,035	1,469	1,661
Reproducibility limit R [R = 2,8 x s_R], %	0,338	0,221	0,375	0,242	0,455	0,343	0,415	0,582
True protein value (%)[a]	11,15	11,70	11,90	10,55	11,75	11,75	10,20	12,30
Deviation predicted - true value (%)	-0,22	0,10	0,18	-0,14	-0,01	0,10	-0,11	0,22

[a] Protein value obtain by 1 Master labs of the European grain networks, using Kjeldahl (EN ISO 20483) method.

Table A.14 b) — Results of statistical analysis for the determination of the protein content in barley by Infraneo PLS model or-pro03.cal

Sample	B9	B10	B11	B12	B13	B14	B15
Number of laboratories	11	11	11	11	11	11	11
Mean predicted protein content (% d.m.)	15,32	10,40	12,73	15,36	11,33	10,79	10,72
Repeatability standard deviation s_r (% P)	0,095	0,097	0,183	0,156	0,073	0,113	0,105
Repeatability relative stand. dev. s_r %	0,618	0,883	1,438	1,014	0,648	1,050	0,977
Repeatability limit r [r = 2,8 x s_r], %	0,265	0,257	0,513	0,513	0,206	0,317	0,293
Reproducibility stand. dev. s_R (% P)	0,256	0,119	0,183	0,183	0,155	0,190	0,147
Reproducibility relative stand. dev. s_R %	1,674	1,147	1,438	1,192	1,364	1,759	1,371
Reproducibility limit R [R = 2,8 x s_R], %	0,718	0,337	0,513	0,651	0,433	0,532	0,411
True protein value (%)[a]	15,05	10,40	12,65	15,50	11,65	10,95	10,70
Deviation predicted - true value (%)	0,27	-0,00	0,08	-0,14	-0,32	-0,16	0,02

[a] Protein value obtain by 1 Master labs of the European grain networks, using Kjeldahl (EN ISO 20483) method.

Table A.15 a) — Results of statistical analysis for the determination of the moisture content in wheat by Infraneo PLS model bl-hum05.cal

Sample	W1	W2	W3	W4	W5	W6	W7	W8
Number of laboratories	11	11	11	11	11	11	11	11
Mean predicted moisture content (%)	13,49	12,79	15,47	13,65	13,10	13,61	10,63	14,26
Repeatability standard deviation s_r (% H_2O)	0,059	0,033	0,030	0,037	0,035	0,046	0,051	0,060
Repeatability relative stand. dev. s_r %	0,434	0,258	0,195	0,270	0,269	0,341	0,478	0,423
Repeatability limit r [r = 2,8 x s_r], %	0,164	0,092	0,084	0,103	0,099	0,130	0,142	0,169
Reproducibility stand. dev. s_R (% H_2O)	0,096	0,103	0,099	0,075	0,106	0,092	0,085	0,091
Reproducibility relative stand. dev. s_R %	0,715	0,807	0,641	0,547	0,811	0,680	0,804	0,639
Reproducibility limit R [R = 2,8 x s_R], %	0,270	0,289	0,278	0,209	0,297	0,259	0,239	0,255
True moisture value (%)[a]	13,80	13,00	15,70	13,70	13,00	13,30	10,70	14,20
Deviation predicted - true value (%)	-0,31	-0,16	-0,23	0,00	0,15	0,31	-0,07	0,06

[a] Moisture value obtain by 1 Master labs of the European grain networks using EN ISO 712 method.

Table A.15 b) — Results of statistical analysis for the determination of the moisture content in wheat by Infraneo PLS model bl-hum05.cal

Sample	W9	W10	W11	W12	W13	W14	W15	W16
Number of laboratories	11	11	11	11	11	11	11	11
Mean predicted moisture content (%)	12,24	12,49	12,88	10,89	11,21	15,74	13,67	12,16
Repeatability standard deviation s_r (% H_2O)	0,056	0,036	0,047	0,050	0,018	0,035	0,039	0,036
Repeatability relative stand. dev. s_r %	0,456	0,287	0,367	0,456	0,161	0,221	0,285	0,300
Repeatability limit r [r = 2,8 x s_r], %	0,156	0,100	0,132	0,132	0,051	0,097	0,109	0,102
Reproducibility stand. dev. s_R (% H_2O)	0,087	0,092	0,082	0,088	0,118	0,108	0,092	0,120
Reproducibility relative stand. dev. s_R %	0,707	0,734	0,636	0,811	1,049	0,687	0,674	0,989
Reproducibility limit R [R = 2,8 x s_R], %	0,242	0,257	0,229	0,247	0,330	0,303	0,258	0,337
True moisture value (%)[a]	12,60	12,50	12,70	11,00	11,00	15,80	13,60	12,05
Deviation predicted - true value (%)	-0,31	-0,01	0,23	-0,06	0,26	-0,06	0,12	0,11

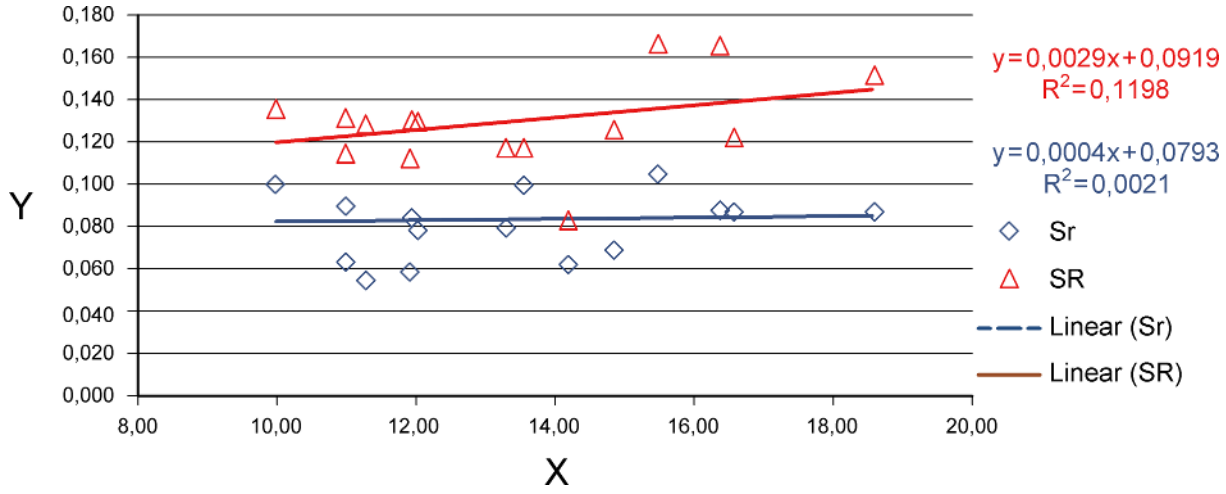
[a] Moisture value obtain by 1 Master labs of the European grain networks using EN ISO 712 method.

Table A.16 a) — Results of statistical analysis for the determination of the moisture content in barley by Infraneo PLS model or-hum05.cal

Sample	B1	B2	B3	B4	B5	B6	B7	B8
Number of laboratories	11	11	11	12	11	11	11	11
Mean predicted moisture content (%)	13,30	14,06	13,80	11,89	11,94	14,14	10,60	15,90
Repeatability standard deviation s_r (% H_2O)	0,042	0,041	0,046	0,050	0,050	0,041	0,058	0,046
Repeatability relative stand. dev. s_r %	0,312	0,289	0,335	0,422	0,419	0,288	0,547	0,290
Repeatability limit r [r = 2,8 x s_r], %	0,116	0,114	0,130	0,140	0,140	0,114	0,162	0,129
Reproducibility stand. dev. s_R (% H_2O)	0,099	0,079	0,061	0,088	0,064	0,067	0,140	0,127
Reproducibility relative stand. dev. s_R %	0,747	0,559	0,441	0,737	0,536	0,476	1,323	0,800
Reproducibility limit R [R = 2,8 x s_R], %	0,278	0,220	0,170	0,245	0,179	0,189	0,393	0,356
True moisture value (%)[a]	13,40	14,10	13,70	12,00	12,10	14,10	10,80	16,00
Deviation predicted - true value (%)	-0,10	0,01	0,10	-0,06	-0,16	0,09	-0,15	-0,05
[a] Moisture value obtain by 1 Master labs of the European grain networks using EN ISO 712 method.								

Table A.16 b) — Results of statistical analysis for the determination of the moisture content in barley by Infraneo PLS model or-hum05.cal

Sample	B9	B10	B11	B12	B13	B14	B15
Number of laboratories	11	12	11	11	11	11	11
Mean predicted moisture content (%)	12,50	12,72	13,89	15,70	13,49	11,68	11,13
Repeatability standard deviation s_r (% H_2O)	0,062	0,036	0,052	0,053	0,035	0,049	0,056
Repeatability relative stand. dev. s_r %	0,500	0,279	0,377	0,339	0,258	0,419	0,505
Repeatability limit r [r = 2,8 x s_r], %	0,175	0,099	0,146	0,146	0,098	0,137	0,157
Reproducibility stand. dev. s_R (% H_2O)	0,157	0,055	0,069	0,069	0,074	0,080	0,111
Reproducibility relative stand. dev. s_R %	1,258	0,433	0,499	0,442	0,549	0,688	0,996
Reproducibility limit R [R = 2,8 x s_R], %	0,440	0,154	0,194	0,384	0,207	0,225	0,310
True moisture value (%)[a]	12,50	12,70	13,60	15,80	13,30	11,80	11,20
Deviation predicted - true value (%)	0,00	0,07	0,29	-0,10	0,19	-0,12	-0,07
[a] Moisture value obtain by 1 Master labs of the European grain networks using EN ISO 712 method.							

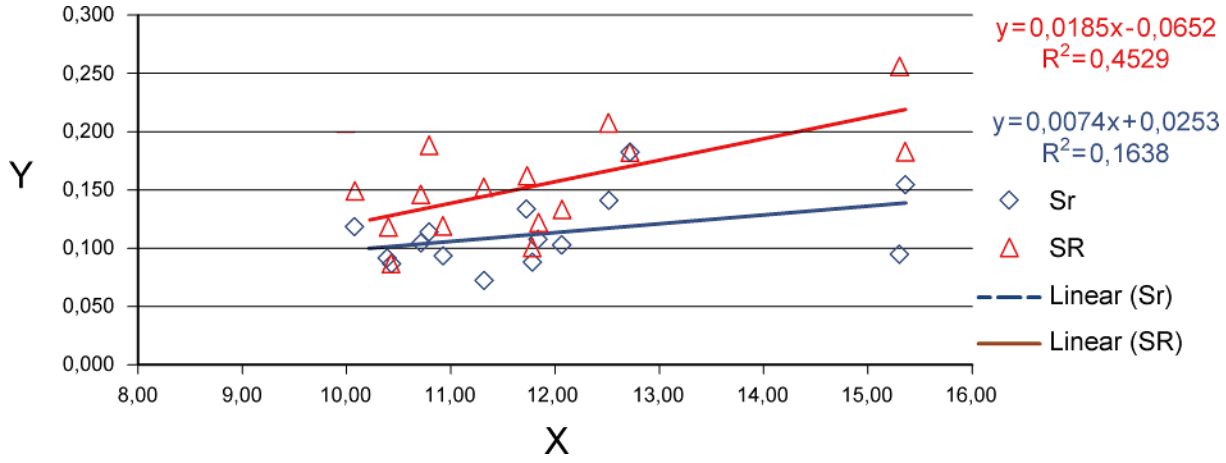


Key

X protein content %

Y standard deviation %

Figure A.5 — Standard deviations for the repeatability, s_r, and reproducibility, s_R, as a function of the protein content on wheat samples

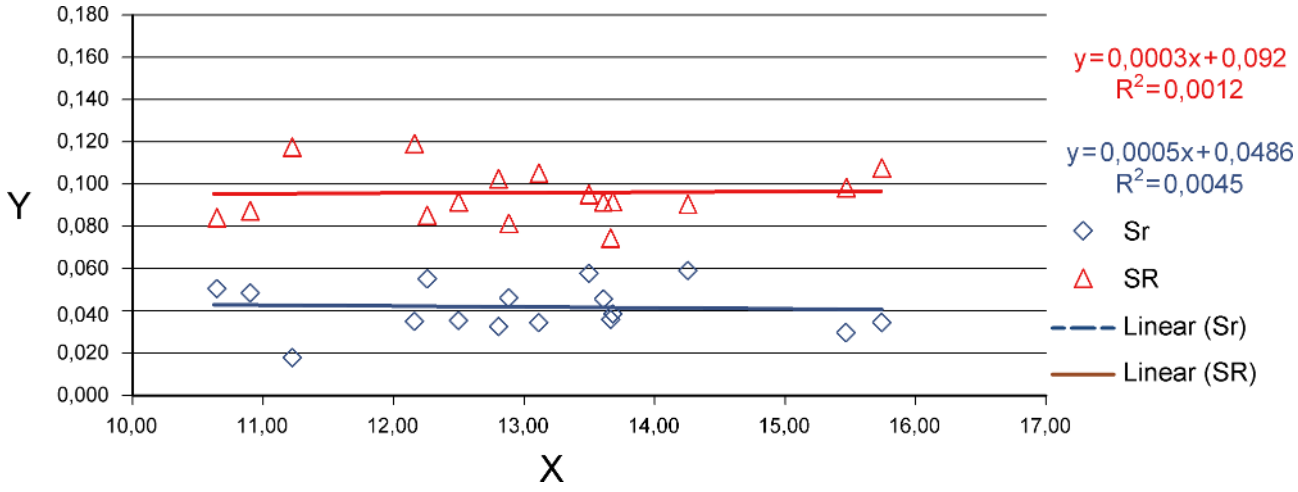


Key

X protein content %

Y standard deviation %

Figure A.6 — Standard deviations for the repeatability, s_r, and reproducibility, s_R, as a function of the protein content on barley samples

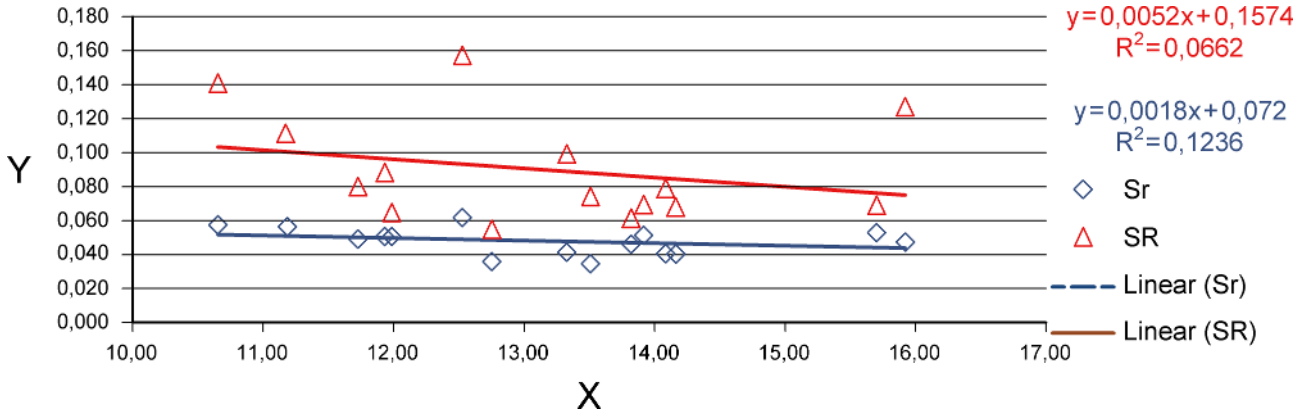


Key

X moisture content %

Y standard deviation %

Figure A.7 — Standard deviations for the repeatability, s_r, and reproducibility, s_R, as a function of the moisture content on wheat samples



Key

X moisture content %

Y standard deviation %

Figure A.8 — Standard deviations for the repeatability, s_r, and reproducibility, s_R, as a function of the moisture content on barley samples

Annex B
(informative)

Validation of ANN prediction model WB003034

B.1 ANN prediction model WB003034 with associated database

The ANN prediction model for the simultaneous prediction of protein and moisture contents in whole grain of wheat and barley is based on thousands of samples. See Table B.1 below.

Table B.1 — ANN prediction model WB003034

Component	Number of samples used in the model (N)	Constituent ranges covered in the model	
		Min	Max
Moisture, overall	10 572	6,2%	30,0%
Protein, overall	30 092	6,7 % d.m.	23,7 % d.m.

B.2 Results of validation according to EN ISO 12099

The application model WB003034 has been validated according to EN ISO 12099 by FOSS Analytical AB (Sweden) using independent test sets of wheat and barley samples, originating from different parts of the world (Table B.3) and analyzed by different laboratories using the reference methods given in Clause 2. The results are summarized in Table B.2.

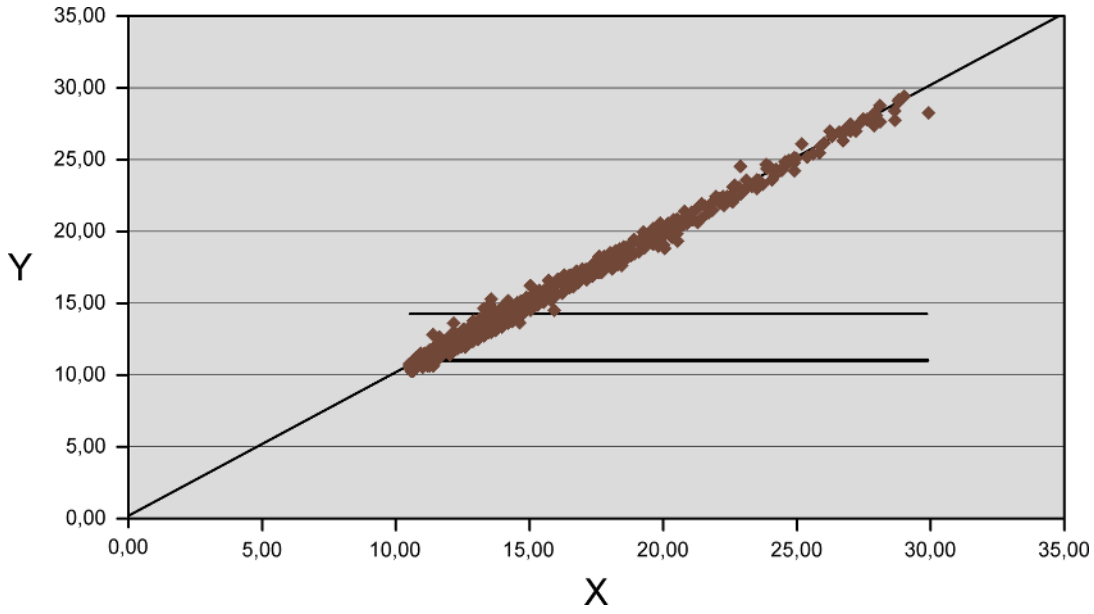
Table B.2 — ANN model WB003034 validation summary

	Moisture	**Protein**
Number (N) of samples in independent test set, total	4 600	11 822
N for barley	2 251	3 963
N for wheat	2 349	7 857
Accuracy expressed as SEP (constituent % w/w), total	0,24 %	0,27 %
SEP for barley	0,22 %	0,30 %
SEP for wheat	0,25 %	0,26 %
Constituent range in the independent validation set, total	7,8 % - 29,9 %	6,9 % - 24,0 % (d.m.)
Range for barley	8,6 % - 27,9 %	7,3 % - 17,7 %
Range for wheat	7,8 % - 29,9 %	6,9 % - 24,0 %
Linear correlation coefficient between predicted and reference results (R^2), total	0,99	0,99
R^2 for barley	0,99	0,97
R^2 for wheat	0,99	0,99

Table B.3 — Origin of samples used for the validation of the ANN model WB003034

Country
Australia
Austria
Canada
Denmark
France
Finland
Germany
Great Britain
Hungary
Italy
Japan
Sweden
Turkey
USA

Figure B.1 and Figure B.2 show the reference values vs. the predicted results.



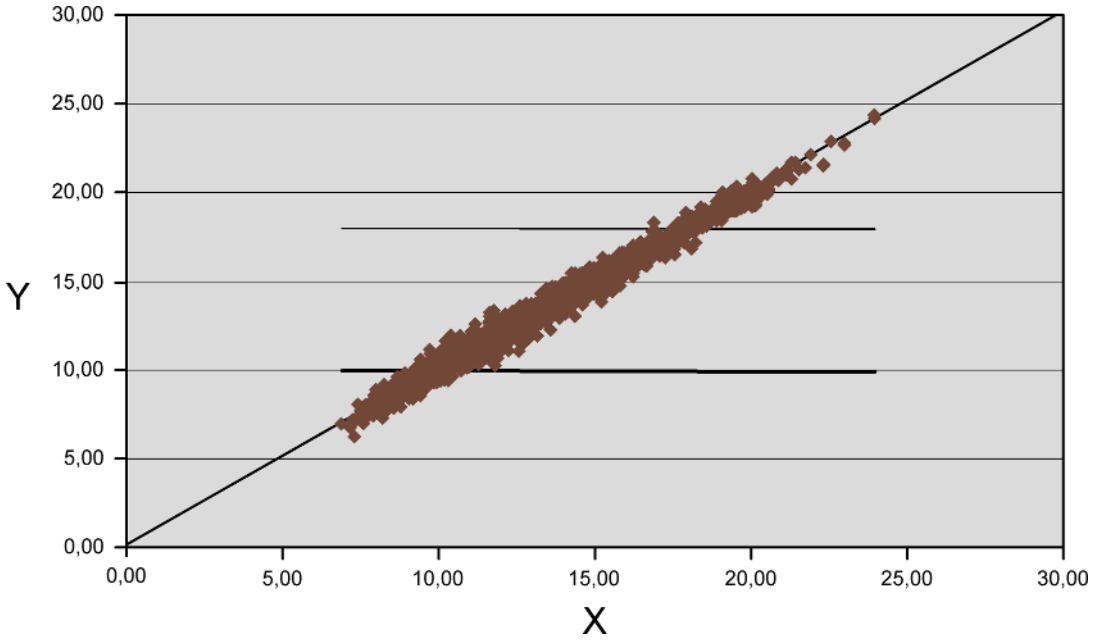
Key

X % predicted

Y % reference

NOTE Dark and light lines indicate range covered in interlaboratory test summarized in Annex A.

Figure B.1 — Plot of reference vs. predicted moisture results using the ANN model WB003034 (bias 0,01, slope 1,00)



Key

X % predicted

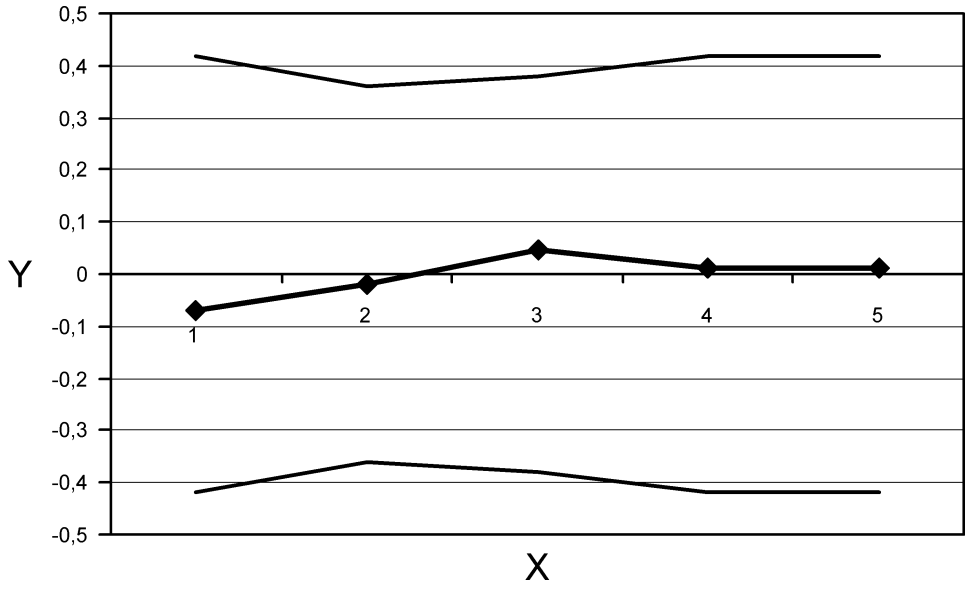
Y % reference

NOTE Dark and light lines indicate range covered by interlaboratory test summarized in Annex A.

Figure B.2 — Plot of reference vs. predicted protein results using the ANN model WB003034 (bias 0,01; slope 1,00)

B.3 Stability and robustness

The ANN model WB003034 has been validated each year since 1996 according to EN ISO 12099 involving the master labs of different networks in Europe and samples from the actual harvests from different countries (see Figure B.3 and Figure B.4). Results of these validations have been published as EGN reports (see Bibliography [10]).

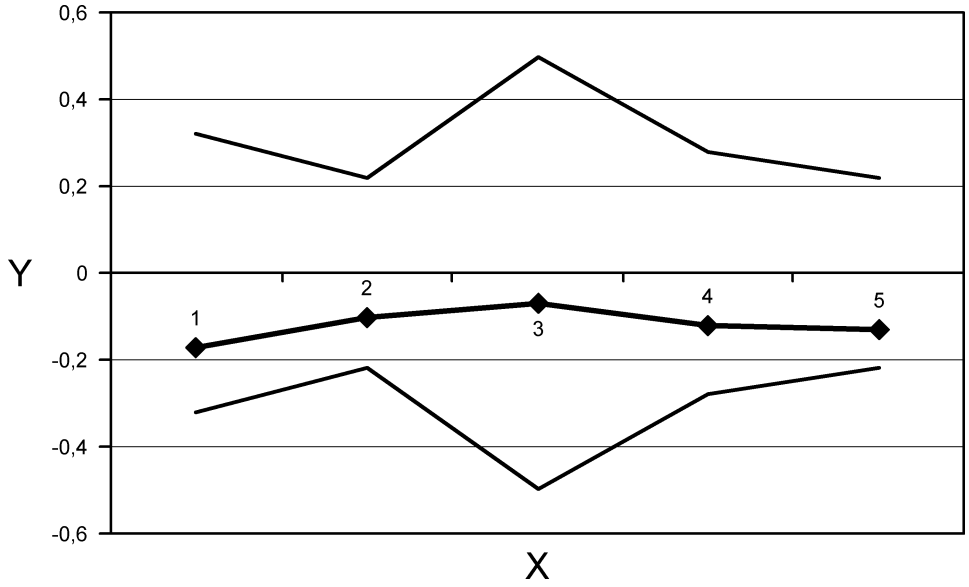


Key

X harvest year 2006-2010

Y dev% protein

Figure B.3 — Average deviation of protein values predicted by the model WB003034 from the best estimate of the true value as established by the mean value of the master labs of European networks (border lines : +/- 2 standard deviations of reproducibility of the reference method)



Key

X harvest year 2006-2010

Y dev% moisture

Figure B.4 — Average deviation of moisture values predicted by the model WB003034 from the best estimate of the true value as established by the mean value of the master labs of European networks (border lines : +/- 2 standard deviations of reproducibility of the reference method)

Annex C
(informative)

Validation of Inframatic 9500 prediction models

C.1 Perten Inframatic 9500 prediction models

A summary of the Inframatic 9500 prediction models for the determination of protein and moisture contents in whole grain of wheat and barley is given in the Tables C.1 and C.2 below.

Table C.1 — Inframatic 9500 Wheat Calibrations Version 5, March 19, 2013

Component	Number of samples used in the model (N)	Constituent ranges covered in the model	
		Min	Max
Moisture	➢ 4 700	7,1 %	25,7 %
Protein (factor 5,7)	➢ 4 700	6,5 % d.m.	21,7 % d.m.

The wheat calibrations are based on >4 700 samples covering the crop years 2009, 2010, 2011, 2012 and from Australia also 2012/2013 plus a smaller number of library samples from 2001 and forward.

Data have been collected at major grain elevators and analytical laboratories or institutes including wheat from Australia, China, Denmark, Finland, France, Germany, Italy, Sweden, Turkey, the UK and the USA.

Table C.2 — Inframatic 9500 Barley Calibrations Version 6, March 19, 2013

Component	Number of samples used in the model (N)	Constituent ranges covered in the model	
		Min	Max
Moisture	➢ 4 800	7,2 %	25,6 %
Protein (factor 6,25)	➢ 4 800	6,8 % d.m.	17,6 % d.m.

The barley calibrations are based on >4 800 samples covering crop years 2009, 2010, 2011 and 2012. Data have been collected at major grain elevators and major labs or institutes including wheat and barley from Australia, Canada, Denmark, France, Germany, Spain, Sweden, Turkey, and the UK.

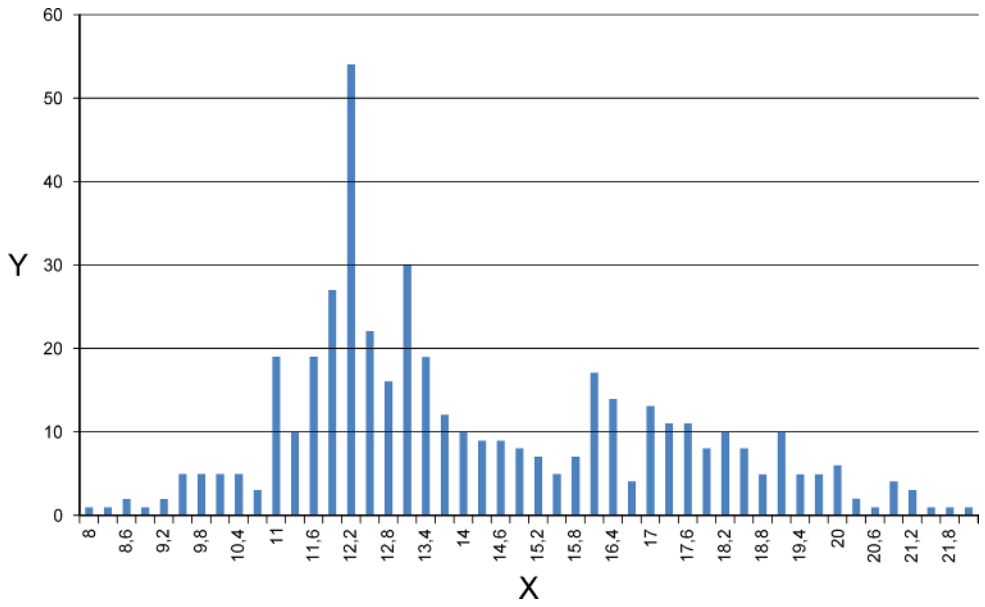
C.2 Results of validations according to EN ISO 12099

The prediction models described in Tables C.1 and C.2 have been validated according to EN ISO 12099 by Perten Instruments AB (Sweden) using independent test sets of wheat and barley samples, originating from different parts of the world, i.e. Australia, Denmark, France, Finland, Germany, Italy, Spain, Sweden, Turkey, United Kingdom, and analyzed by different laboratories using the reference methods given in Clause 2. The results are summarized in Table C.3.

Table C.3 — Validation summary for Perten Inframatic 9500 calibrations

	Moisture	Protein
Number (N) of samples in independent test set, barley	461	610
Number (N) of samples in independent test set, wheat	410	514
Accuracy expressed as SEP (constituent % w/w), barley	0,19 %	0,26 %
Accuracy expressed as SEP (constituent % w/w), wheat	0,17 %	0,21 %
Constituent range in the independent validation set, barley	8,0 % - 22,1 %	7,0 % - 16,0 % (d.m.)
Constituent range in the independent validation set, wheat	8,0 % - 22,1 %	7,0 % - 20,2 % (d.m.)
Linear correlation coefficient between predicted and reference results (R^2), barley	0,93	0,98
Linear correlation coefficient between predicted and reference results (R^2), wheat	0,99	0,99

The distribution of samples used for validation is depicted in the following histograms(Figures C.1 to C.4). The results of the validation of the instrument for the 2 parameters measured for wheat and barley are described in Figures C.5 to C.8.

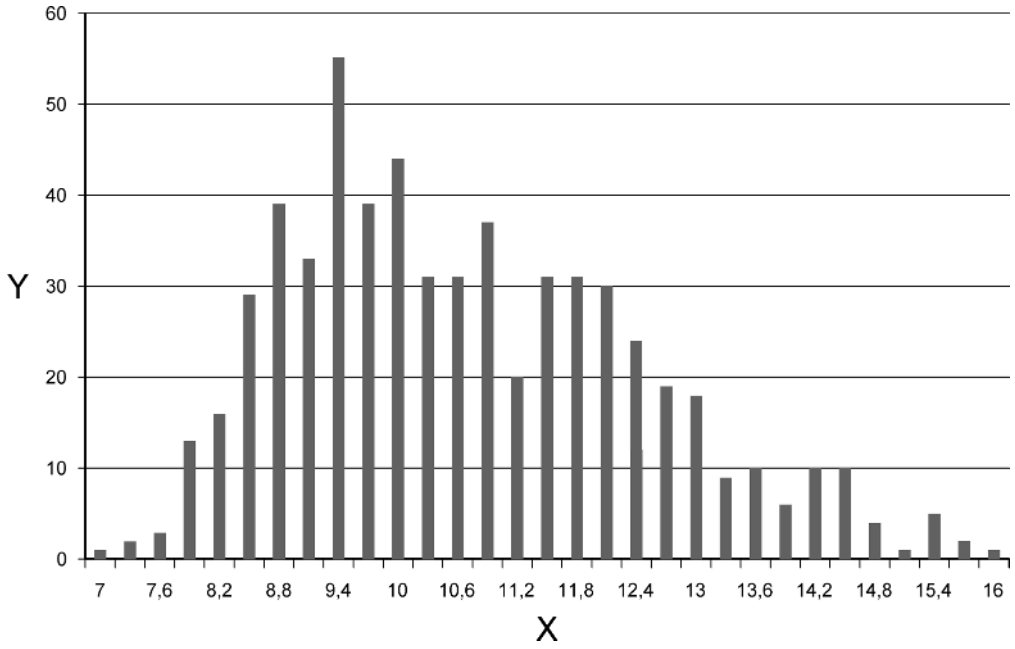


Key

X number of samples

Y moisture content %

Figure C.1 — Histogram of samples used for the validation of the prediction model - Inframatic 9500 Barley Calibrations Version 6, March 19, 2013 – for the moisture content in barley samples (no of samples vs. % moisture)

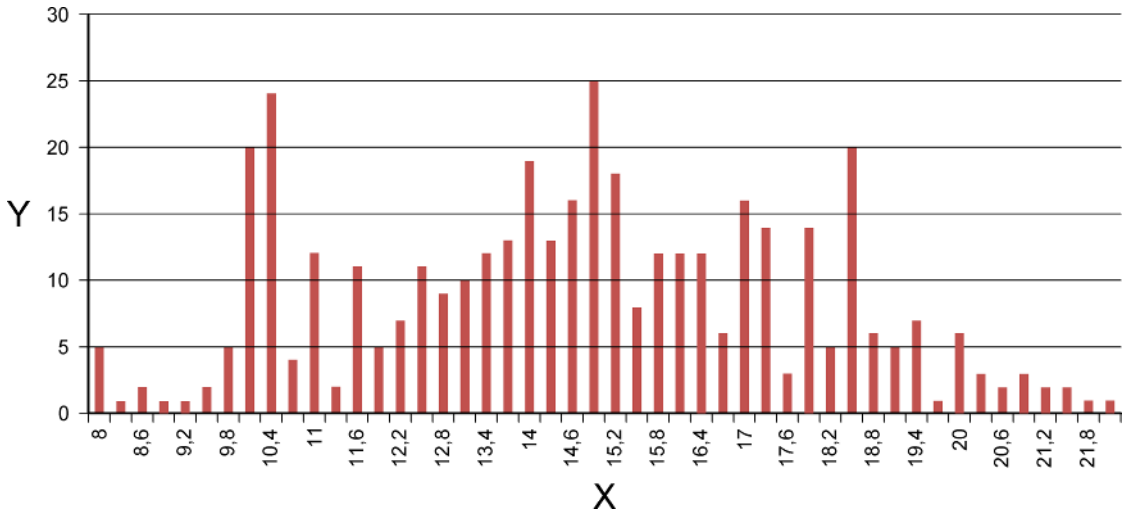


Key

X number of samples

Y protein content % (d.m)

Figure C.2 — Histogram of samples used for the validation of the prediction model - Inframatic 9500 Barley Calibrations Version 6, March 19, 2013 - for the protein content of barley samples (no of samples vs. % protein d.m.)

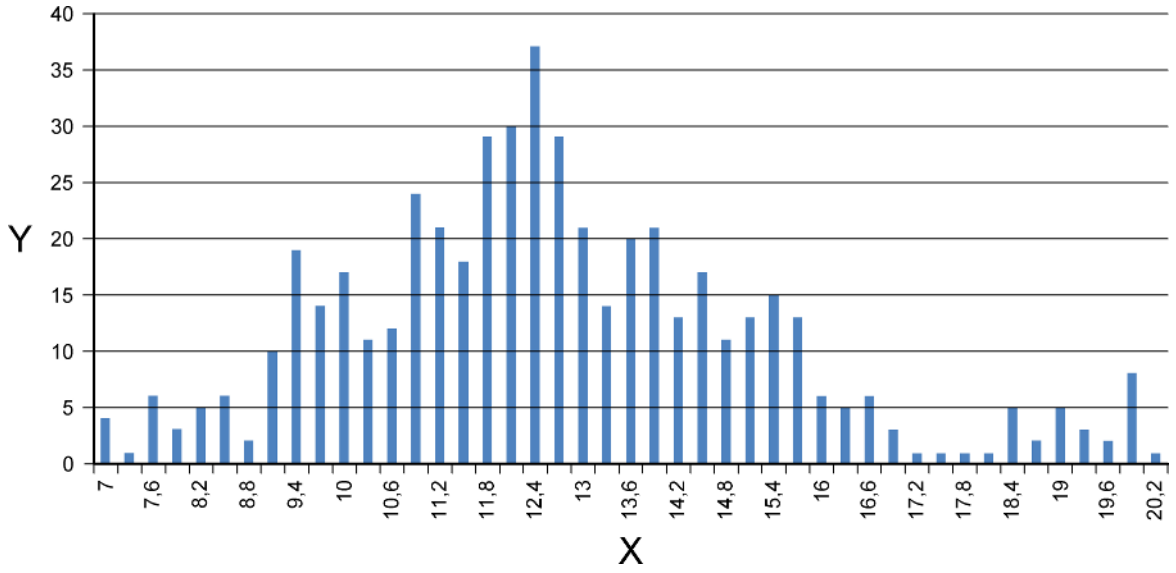


Key

X number of samples

Y moisture content %

Figure C.3 — Histogram of samples used for the validation of the prediction model - Inframatic 9500 Wheat Calibrations Version 5, March 19, 2013 – for the moisture content in wheat samples (no of samples vs. % moisture)

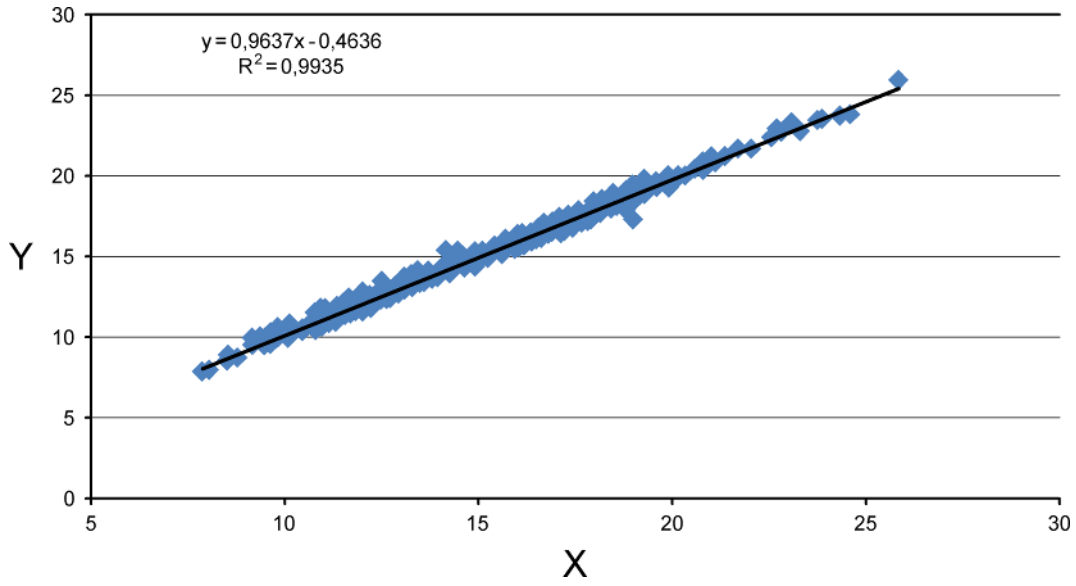


Key

X number of samples

Y protein content % (d.m)

Figure C.4 — Histogram of samples used for the validation of the prediction model - Inframatic 9500 Wheat Calibrations Version 5, March 19, 2013 - for the protein content of wheat samples (no of samples vs. % protein d.m.)

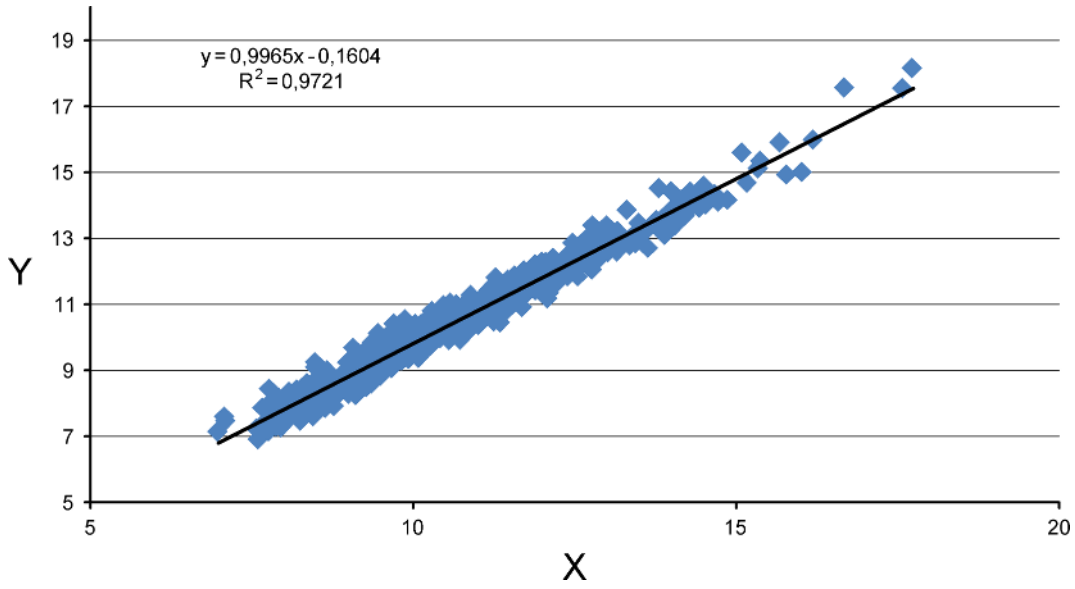


Key

X plot of reference

Y predicted moisture results

Figure C.5 — Validation of the Inframatic 9500 Barley Calibrations Version 6 from March 19, 2013: Plot of reference vs. predicted moisture results.

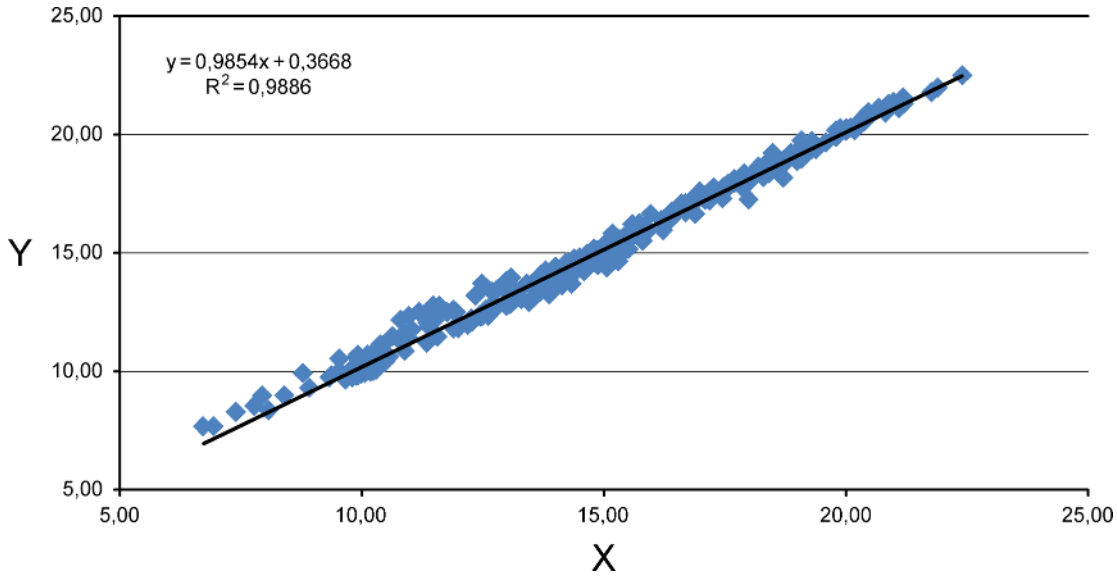


Key

X plot of reference

Y predicted protein results

Figure C.6 — Validation of the Inframatic 9500 Barley Calibrations Version 6 from March 19, 2013: Plot of reference vs. predicted protein results.

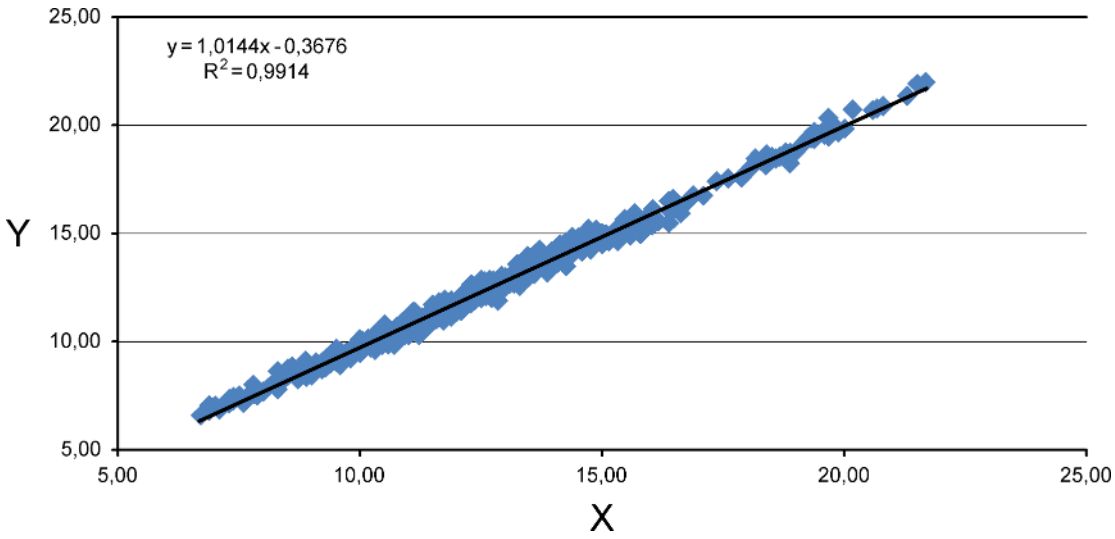


Key

X plot of reference

Y predicted moisture results

Figure C.7 — Validation of the Inframatic 9500 Wheat Calibrations Version 5 from March 19, 2013: Plot of reference vs. predicted moisture results.



Key

X plot of reference

Y predicted protein results

Figure C.8 — Validation of the Inframatic 9500 Wheat Calibrations Version 5 from March 19, 2013: Plot of reference vs. predicted protein results.

Annex D
(informative)

Validation of Infraneo prediction models

D.1 CHOPIN Technologies Infraneo prediction models

A summary of the Infraneo prediction models for the determination of protein and moisture contents on whole grain of wheat and barley is given in the Table D.1 below.

Table D.1 — Infraneo Calibration models

Product	Component	Number of samples used in the model (N)	Constituent ranges covered in the model	
			Min	Max
Wheat	Moisture	636	9,7 %	22,6 %
	Protein	1140	7,4 % d.m.	19,9 % d. m.
Barley	Moisture	444	9,7 %	21,0 %
	Protein	753	7,8 % d.m.	21,7 % d.m.

These Infraneo prediction models have been developed on samples worldwide collected and coming from crop year 2006 to 2011.

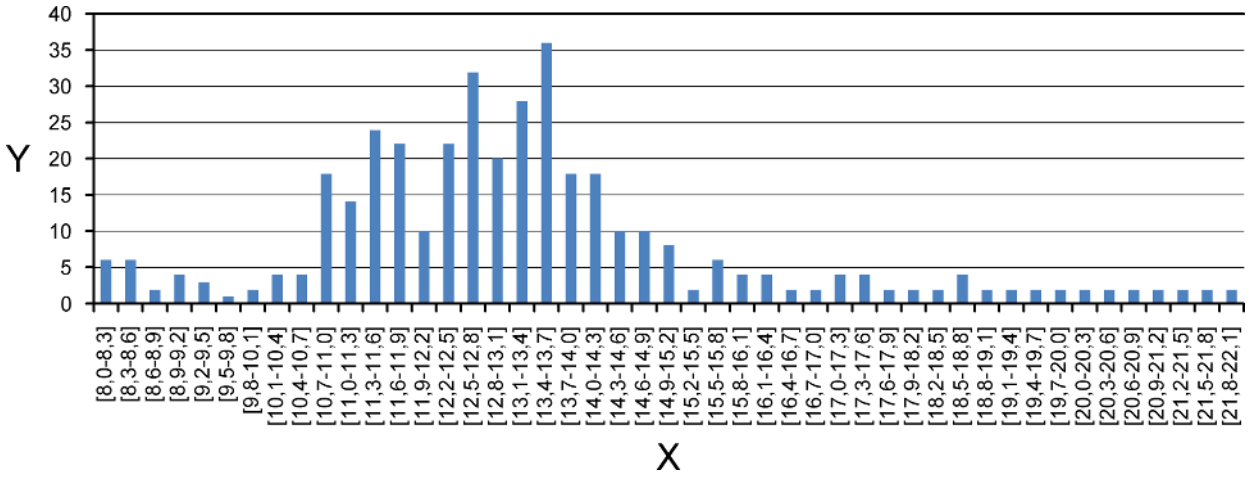
D.2 Results of validation according to EN ISO 12099

The prediction models described in Table D.1 have been validated according to EN ISO 12099 by CHOPIN Technologies (France) using independent test sets of wheat and barley samples, originating from different parts of the world, i.e. Argentina, Italy, France, Germany, Poland, Turkey, Russia, Spain, South Africa and United States, and analyzed on different Infraneo using the reference methods given in Clause 2. The results are summarized in Table D.2.

Table D.2 — Infraneo prediction models validation summary

	Moisture	Protein
Number (N) of samples in independent test set, **barley**	389	437
Number (N) of samples in independent test set, **wheat**	533	718
Accuracy expressed as SEP (constituent % w/w), **barley**	0,22 %	0,24 %
Accuracy expressed as SEP (constituent % w/w), **wheat**	0,19 %	0,22 %
Constituent range in the independent validation set, **barley**	8,0 % - 23,5 %	7,4 % - 16,2 % (d.m.)
Constituent range in the independent validation set, **wheat**	7,1 % - 22,8 %	7,0 % - 19,9 % (d.m.)
Linear correlation coefficient between predicted and reference results (R^2), **barley**	0,99	0,98
Linear correlation coefficient between predicted and reference results (R^2), **wheat**	0,99	0,99

The distribution of samples used for validation is presented in the following graphs of the following histogram (Figures D.1 to D.4). The results of the validation of the instrument for the 2 parameters measured for wheat and barley are described in Figures D.5 to D.8.

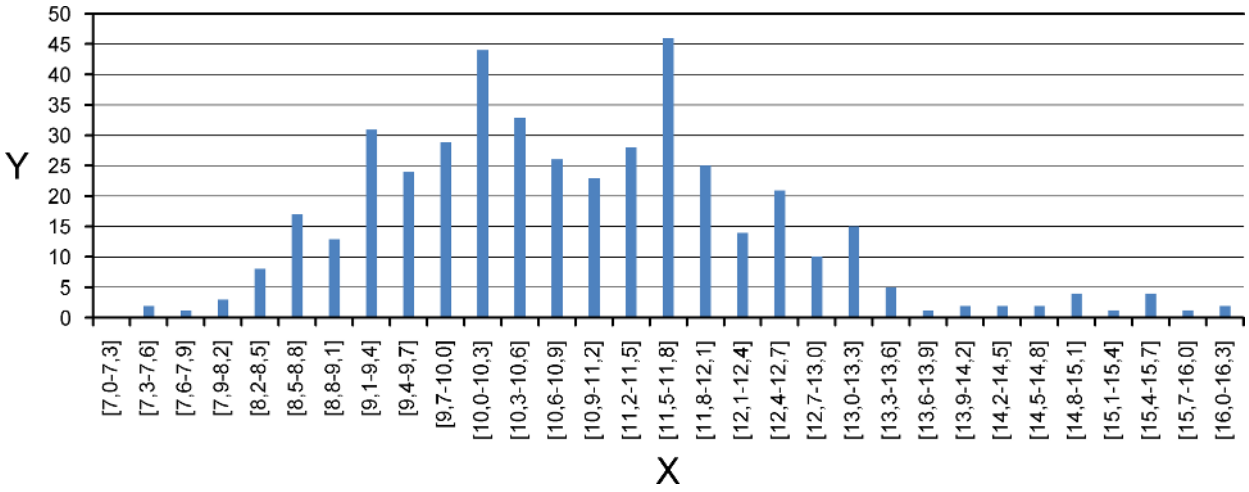


Key

X number of samples

Y moisture content

Figure D.1 — Distribution of samples used for the validation of the Infraneo prediction model or-hum05.cal for moisture content on barley.

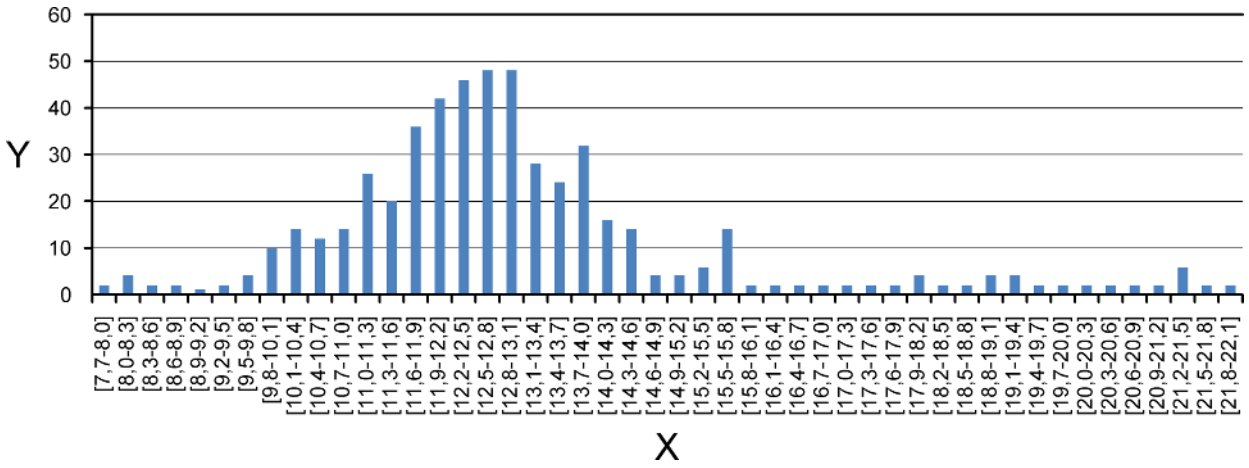


Key

X number of samples

Y protein content

Figure D.2 — Distribution of samples used for the validation of the Infraneo prediction model or-pro03.cal for protein content on barley.

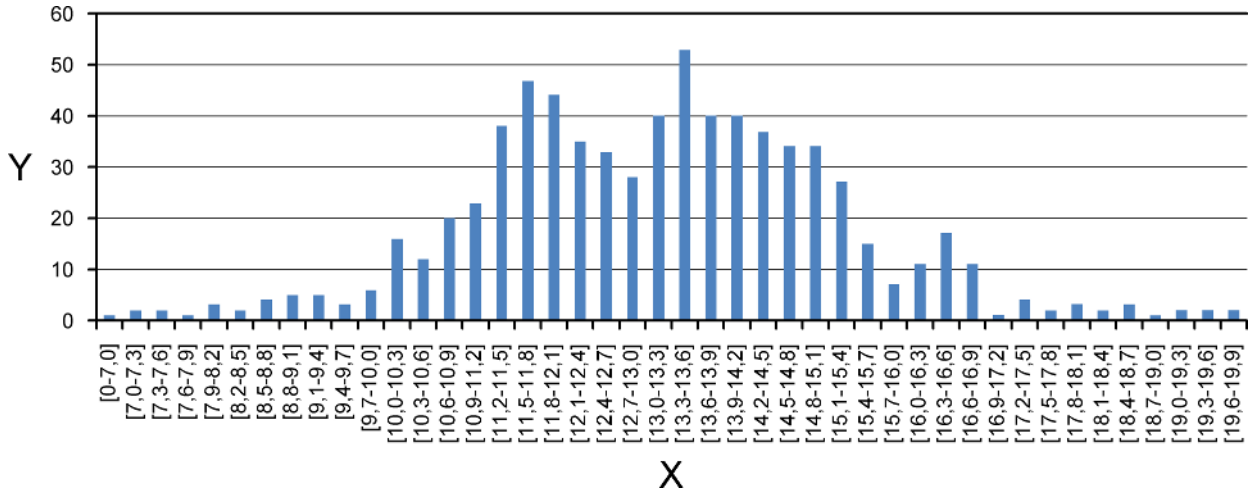


Key

X number of samples

Y moisture content

Figure D.3 — Distribution of samples used for the validation of the Infraneo prediction model bl-hum05.cal for moisture content on wheat.

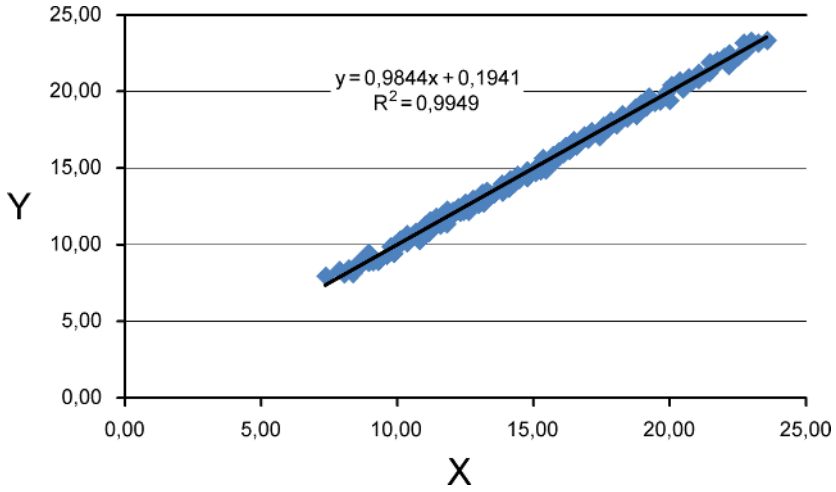


Key

X number of samples

Y protein content

Figure D.4 — Distribution of samples used for the validation of the Infraneo prediction model bl-pro03.cal for protein content on wheat.

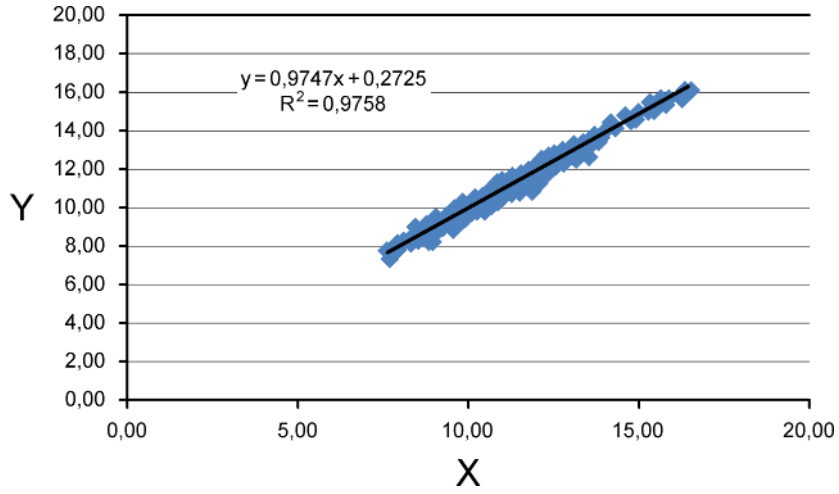


Key

X reference value

Y predicted results

Figure D.5 — Reference values vs. predicted results for moisture on barley

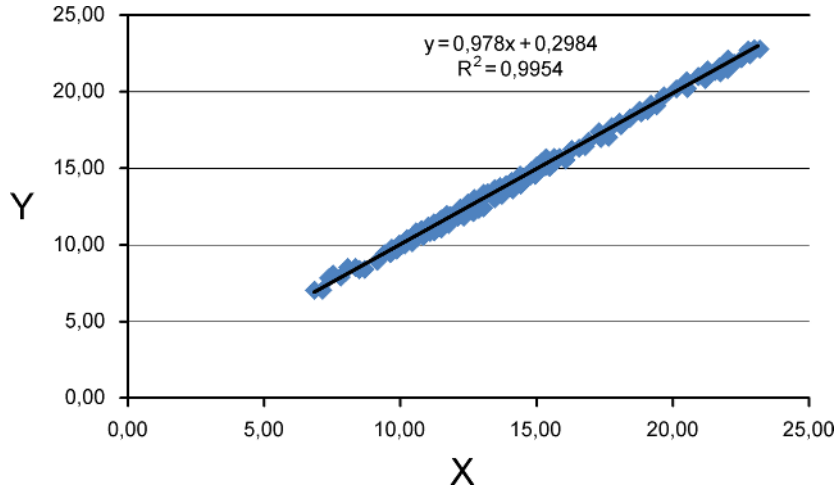


Key

X reference value

Y predicted results

Figure D.6 — Reference values vs. predicted results for protein on barley

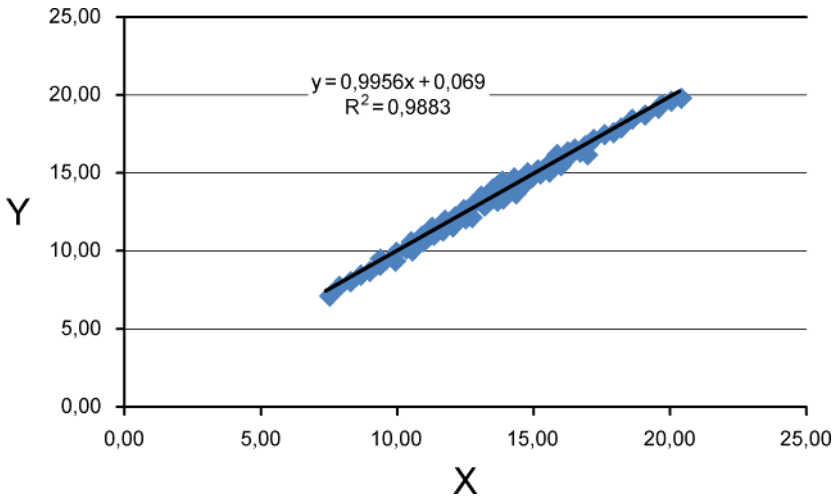


Key

X reference value

Y predicted results

Figure D.7 — Reference values vs. predicted results for moisture on wheat



Key

X reference value

Y predicted results

Figure D.8 — Reference values vs. predicted results for protein on wheat

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