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Žito (pšenica in ječmen) - Tehnično poročilo medlaboratorijskih študij o ugotavljanju vlage in proteinov v celih zrnih z uporabo bližnje infrardeče spektroskopije

Cereals (wheat and barley) - Technical Report of the interlaboratory studies for the determination of moisture and protein in whole kernels by near infrared spectroscopy

Getreide - Technischer Bericht des Ringtestversuchs zur Bestimmung des Feuchte- und Proteingehalts von ganzen Körnern

Céréales (blé et orge) - Rapport technique des essais interlaboratoires pour la détermination de la teneur en eau et en protéines par spectroscopie proche infrarouge sur des grains entiers

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ICS:

67.060	Žita, stročnice in proizvodi iz njih	Cereals, pulses and derived products
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Cereals (wheat and barley) - Technical Report of the
interlaboratory studies for the determination of moisture
and protein in whole kernels by near infrared
spectroscopy

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zur Bestimmung des Feuchte- und Proteingehalts von
ganzen Körnern mittels Nahinfrarotspektroskopie

This Technical Report was approved by CEN on 2 March 2020. It has been drawn up by the Technical Committee CEN/TC 338.

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European foreword

This document (CEN/TR 17474:2020) has been prepared by Technical Committee CEN/TC 338 “Cereal and cereal products”, the secretariat of which is held by AFNOR.

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CEN/TR 17474:2020 (E)**Introduction**

The determination of moisture and protein content in whole kernels of barley and wheat can be done using a near infrared spectrometer.

This document presents the results of 3 interlaboratory tests implementing this routine method and their overall statistical treatment.

The outcomes have been used to fix the precision (repeatability and reproducibility) of EN 15948, "Cereals — Determination of moisture and protein - Method using near infrared spectroscopy in whole kernels".

The 3 interlaboratory tests have been managed by FOSS Analytical AB (Sweden) in 2008, by CHOPIN Technologies (France) in 2012 and by PERTEN Instruments AB (Sweden), a PerkinElmer company, in 2013 in accordance with ISO 5725-2.

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1 Scope

This document defines the repeatability and the reproducibility of a method using near infrared spectroscopy in whole kernels for the determination of moisture and protein on wheat and barley. The performance of the method (accuracy) is found in EN 15948.

The values derived from the report are applicable to the following concentration ranges:

- for wheat:
 - moisture content range from 9,5 % – 15,7 %;
 - protein content range from 10,0 % DM to 18,6 % DM;
- for barley:
 - moisture content range from 10,6 % – 15,9 %;
 - protein content range from 9,2 % DM – 15,4 % DM.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN 15948, *Cereals - Determination of moisture and protein - Method using Near-Infrared Spectroscopy in whole kernels*

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ISO 5725-1, *Accuracy (trueness and precision) of measurement methods and results — Part 1: General principles and definitions*

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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*

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

3 Terms and Definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- IEC Electropedia: available at <http://www.electropedia.org/>
- ISO Online browsing platform: available at <https://www.iso.org/obp>

4 Interlaboratory tests

4.1 FOSS

An interlaboratory test, organized by the FOSS Analytical AB (Sweden) company 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 laboratories of European grain networks. The grain networks did also assist in the collection of the samples (Table 1).

Table 1 — Samples for the interlaboratory study organized by FOSS Analytical

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

The prediction model used for the interlaboratory test is WB003034.

This 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 and 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 and analysed by different laboratories using the reference methods given in bibliography. The performance (accuracy) is in accordance with EN 15948, all the details about the prediction model are given in Annex A.

The results of the interlaboratory test were subjected to statistical analysis in accordance with ISO 5725-1 and ISO 5725-2 to calculate the precision data shown in Tables 2 to 5 and Figures 1 and 2.

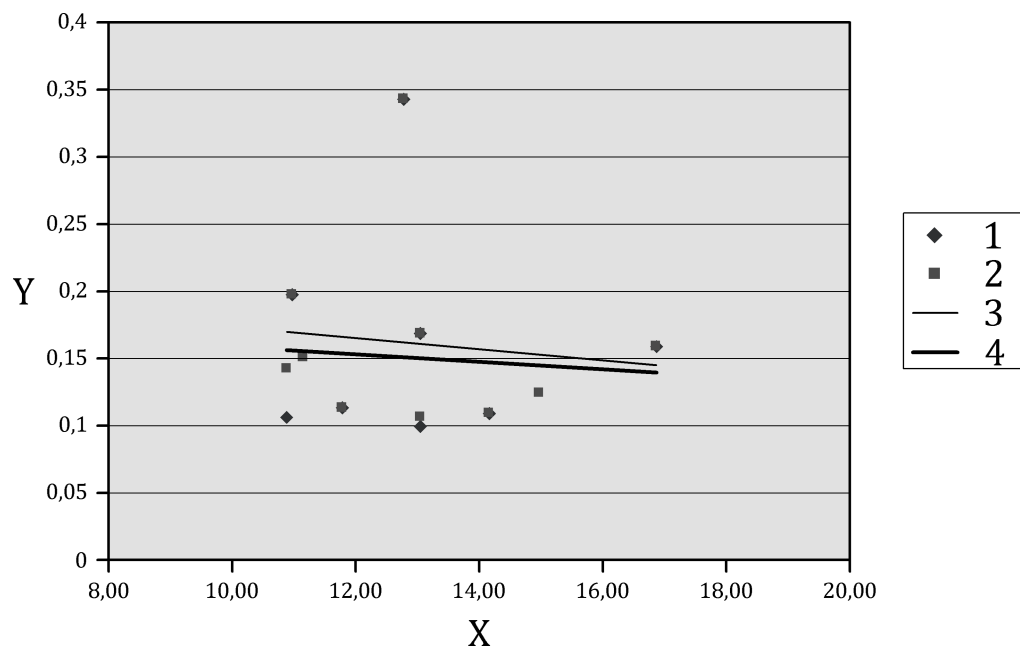
Table 2 — Results of statistical analysis for the determination of the protein content in wheat by the ANN model WB003034

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 (%DM)	16,883	11,789	13,047	10,876	14,985	14,173
Repeatability standard deviation s_r (%DM)	0,159a	0,113a	0,099	0,106	0,087	0,109a
Repeatability relative stand. dev. s_r (%)	0,943	0,958	0,76	0,979	0,583	0,771
Repeatability limit r [$r = 2,77 \times s_r$] (%DM)	0,440	0,313	0,274	0,294	0,241	0,302
Reproducibility stand. dev. s_R (%DM)	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 [$R = 2,77 \times s_R$] (%DM)	0,440	0,313	0,296	0,396	0,346	0,302
Best estimate of true protein value (%DM) ^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 (%DM)	0,01	0,04	0,03	0,01	0,06	0,33
<p>^a $s_r > s_R$, s_R set to be equal s_r.</p> <p>^b Average protein value (after elimination of outliers) generated by 17 Master laboratories of the European grain networks, using Kjeldahl (EN ISO 20483 or EN ISO 5983-2) and Dumas (EN ISO 16634-2) methods.</p>						

Table 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 (%DM)	10,964	13,05	11,15	12,772
Repeatability standard deviation s_r (%DM)	0,198a	0,169a	0,123	0,343 ^a
Repeatability relative stand. dev. s_r (%)	1,804	1,292	1,105	2,682
Repeatability limit r [$r = 2,77 \times s_r$] (%DM)	0,548	0,468	0,341	0,95
Reproducibility stand. dev. s_R (% DM)	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 [$R = 2,77 \times s_R$] (%DM)	0,548	0,468	0,418	0,950
Best estimate of true protein value (%DM) ^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 (%DM)	0,30	-0,06	-0,12	-0,20
<p>^a $s_r > s_R$, s_R set to be equal s_r.</p> <p>^b Average protein value (after elimination of outliers) generated by 17 Master laboratories of the European grain networks, using Kjeldahl (EN ISO 20483 or EN ISO 5983-2) and Dumas (EN ISO 16634-2) methods.</p>				

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**Key**

X protein content (%DM)

Y s (%DM)

1 s_r 2 s_R 3 Linear (s_r)4 Linear (s_R)

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Figure 1 — Standard deviations for the repeatability, s_r , and reproducibility, s_R , as a function of the protein content (wheat and barley)

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

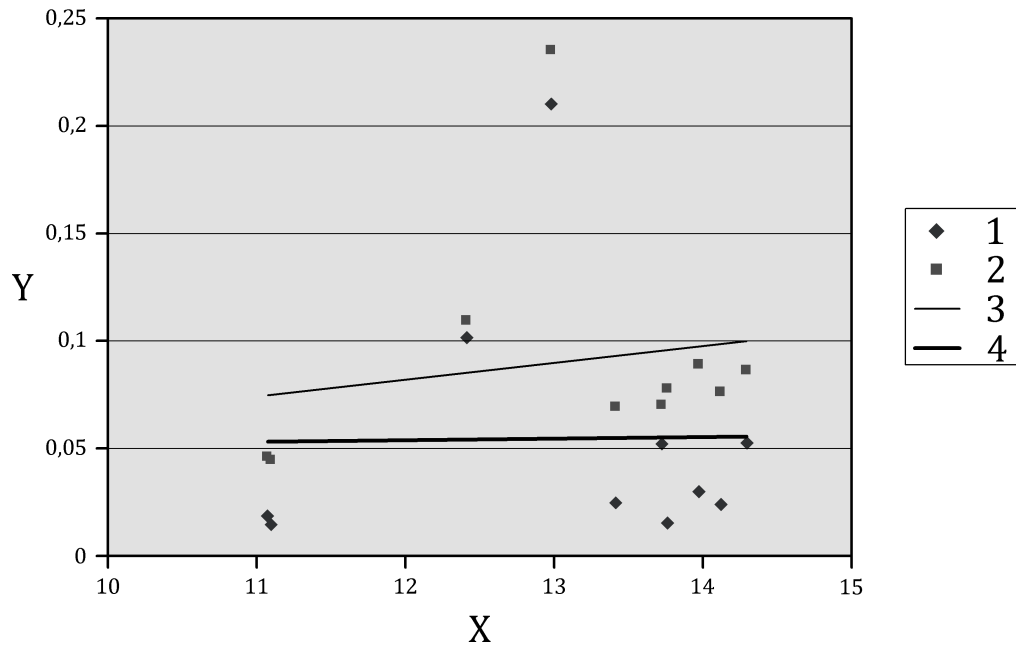
Sample	WG 1	WG 2	WG 3	WG 4	WG 5	WG 6
Number of laboratories	20	20	20	20	20	20
Mean predicted moisture content (%)	14,125	13,415	13,978	13,764	11,102	11,074
Repeatability standard dev s_r (%)	0,024	0,025	0,030	0,016	0,015	0,019
Repeatability relative stand. dev. s_r (%)	0,170	0,187	0,215	0,116	0,138	0,167
Repeatability limit r [$r = 2,77 \times s_r$] (%)	0,066	0,069	0,083	0,044	0,042	0,053
Reproducibility stand. dev. s_R (%)	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 [$R = 2,77 \times s_R$] (%)	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 laboratories of the European grain networks, using EN ISO 712 method.

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

Sample	B1	B2	B3	B4
Number of laboratories	20	20	20	20
Mean predicted moisture content (%)	12,417	13,731	14,299	12,986
Repeatability standard dev s_r (%)	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 [$r = 2,77 \times s_r$] (%)	0,285	0,147	0,150	0,590
Reproducibility stand. dev. s_R (%)	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 [$R = 2,77 \times s_R$] (%)	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 laboratories of the European grain networks, using EN ISO 712 method.

**Key**

X moisture (%)

Y s (%)

1 s_r 2 s_R 3 Linear (s_r)4 Linear (s_R)

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Figure 2 — Standard deviations for the repeatability, s_r , and reproducibility, s_R , as a function of the moisture content (wheat and barley)

4.2 CHOPIN Technologies

An interlaboratory test, organized by the CHOPIN Technologies (France) company 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 6 and Table 7).

Table 6 — Wheat Samples for the interlaboratory study organized by CHOPIN

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

Sample	Description	Country of origin
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 7 — Barley Samples for the interlaboratory study organized by CHOPIN

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

The Infraneo prediction models for the determination of protein and moisture contents on whole grain of wheat and barley have been developed on samples worldwide collected and coming from crop year 2006 to 2011. They 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, and analysed on different Infraneo using the reference methods given in bibliography. The performance