

## DETERMINATION OF THE MOISTURE AND NITROGEN CONTENTS OF BARLEY AND MALT BY NEAR INFRARED SPECTROSCOPY (NIRS)

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**The determination of the moisture and nitrogen contents of barley and malt by near infrared spectroscopy (NIRS) has been tested by the Analysis Committee of the European Brewery Convention. In the collaborative trial four samples of barley and malt were analysed by 17 laboratories. Repeatability ( $r_{95}$ ) and reproducibility ( $R_{95}$ ) values of 0.3 and 1.5 % m/m respectively were obtained for barley moisture over the range 12.7 to 15.8 % m/m. For malt moisture these values were 0.2 and 1.3 % m/m over the range 4.0 to 4.3 % m/m, for barley nitrogen 0.1 and 0.3 % m/m on dry matter over the range 1.57 to 2.14 % m/m, and for malt nitrogen 0.1 and 0.2 % m/m on dry matter over the range 1.58 to 1.82 % m/m, respectively.**

**Key Words:** *Collaborative trial, near infrared spectroscopy, moisture, nitrogen, barley, malt.*

### INTRODUCTION

The Analysis Committee of the European Brewery Convention decided to evaluate near infrared spectroscopy (NIRS\*) as a guideline method for the analysis of moisture and nitrogen in barley and malt. An interlaboratory trial was organised in which 17 laboratories participated. Due to differences in equipment and calibration procedures, it was decided that each laboratory would use its own calibrations.

### EXPERIMENTAL

The organisation of the collaborative trial and the statistical treatment of the data were carried out according to the procedures given in the International Standard ISO 5725<sup>1</sup>. A uniform design was employed and four samples of barley and four samples of malt were circulated to the participating laboratories. Thirteen laboratories used NIRS equipment of a particular make and type operating in the transmission mode and using whole kernels. The other laboratories used various equipment of other make operating in the reflection or transmission mode and using whole kernels or ground samples. Some laboratories reported the results from two NIRS instruments. In the framework of this report these will be considered to be different laboratories. Participating laboratories were requested to determine moisture and nitrogen contents of the samples in duplicate, using their own equipment and calibration. The only requirements were that the instruments should be calibrated using the following methods: EBC method 3.1.1 for barley moisture<sup>2</sup>, EBC method 4.1 for malt moisture<sup>3</sup>, and EBC method 3.2 for nitrogen<sup>4</sup>.

### RESULTS AND DISCUSSION

As the aim of this study was not to evaluate a potential reference method, but to compare results from different laboratories using their own calibrations, no attempts were made to remove Dixon outliers. However, to obtain a reasonable impression of the repeatability any Cochran outliers were removed.

The raw data, as received, are presented in Tables I–IV. A summary of the results of statistical treatment of the data is given in Table V. For barley moisture (Table I) two sets were identified as outliers and therefore rejected.

\*In this report the term Near Infrared Spectroscopy (NIRS) is used to cover both Near Infrared Reflectance Spectroscopy (NIR) and Near Infrared Transmittance Spectroscopy (NIT).

The relatively large data set obtained for the nitrogen method permitted an investigation to determine whether there is a significant difference between the average values obtained with the group of 13 instruments and the average values obtained with other equipment. Statistical analysis revealed that this was not the case.

### CONCLUSION

This collaborative trial showed that the repeatabilities for the determination of moisture and nitrogen in barley and malt using NIRS (Table V) are at a level comparable with EBC accepted methods (Table VI). The values for the reproducibility obtained with NIRS (Table V) are in general much higher than those given for the reference methods (Table VI). This is probably due to the different NIRS calibration procedures employed by the participating laboratories.

The method should not be used as a reference method or in commercial transactions.

TABLE I. Raw data as received for the NIR moisture determination in barley (units % m/m)

Laboratory	Sample 1		Sample 2		Sample 3		Sample 4	
1	12.5	12.6	13.5	13.6	15.6	15.6	14.3	14.5
2	13.4	13.5	13.8	13.8	16.1	16.1	15.6	15.5
3	13.0	13.0	13.3	13.3	15.1	15.1	14.9	14.9
4	12.8	12.8	13.6	13.7	16.1	15.5	15.5	15.5
5	12.3	12.0	13.1	13.3	15.7	15.9	14.5	14.6
6	12.1	12.4	13.2	13.2	15.7	15.6	14.5	14.6
7	12.7	12.8	13.5*	15.5*	16.8*	15.9*	15.1	15.0
8	12.7	12.7	13.7	13.7	15.3	15.3	13.9	13.9

\* Rejected as a Cochran outlier.

TABLE II. Raw data as received for the NIR moisture determination in malt (units % m/m)

Laboratory	Sample 1		Sample 2		Sample 3		Sample 4	
1	4.7	4.6	4.4	4.4	3.9	4.0	4.0	3.9
2	3.9	3.9	3.8	3.9	3.6	3.6	3.8	3.8
3	4.6	4.7	4.6	4.6	4.5	4.5	4.3	4.3
4	4.3	4.2	4.2	4.2	3.9	3.9	3.9	3.8
5	4.6	4.5	4.4	4.4	4.3	4.3	4.3	4.3
6	3.3	3.4	3.5	3.5	3.4	3.3	3.2	3.3
7	4.5	4.4	4.5	4.6	4.3	4.3	4.1	4.1
8	4.8	4.8	4.9	5.0	4.8	4.7	4.6	4.7

TABLE III. Raw data as received for the NIR nitrogen determination in barley (units % m/m on dry matter)

Laboratory	Sample 1		Sample 2		Sample 3		Sample 4	
1	1.76	—	2.26	—	1.57	—	1.25	—
2	1.73	1.74	2.13	2.16	1.74	1.78	1.52	1.54
3	1.76	1.78	2.14	2.16	1.71	1.73	1.60	1.63
4	1.78	1.79	2.14	2.14	1.79	1.76	1.70	1.70
5	1.74	1.74	2.10	2.18	1.74	1.74	1.63	1.62
6	1.63	1.66	2.00	2.00	1.54	1.52	1.49	1.49
7	1.73	1.74	2.13	2.11	1.71	1.71	1.52	1.52
8	1.62	1.63	2.02	2.03	1.55	1.57	1.42	1.44
9	1.68	1.70	2.03	2.02	1.66	1.66	1.54	1.55
10	1.78	1.71	2.13	2.26	1.70	1.66	1.60	1.47
11	1.82	1.86	2.22	2.22	1.87	1.84	1.78	1.78
12	1.92	1.90	2.29	2.32	1.81	1.79	1.68	1.70
13	1.71	1.74	2.16	2.14	1.63	1.65	1.55	1.54
14	1.65	1.63	2.10	2.11	1.63	1.63	1.50	1.54
15	1.76	1.73	2.14	2.13	1.70	1.74	1.60	1.63
16	1.78	1.79	2.02	2.03	1.78	1.78	1.70	1.71
17	1.76	1.76	2.22	2.24	1.70	1.73	1.57	1.55
18	1.73	1.70	2.11	2.08	1.70	1.74	1.54	1.54
19	1.76	1.74	2.16	2.11	1.71	1.70	1.62	1.66

TABLE IV. Raw data as received for the NIR nitrogen determination in malt (units % m/m on dry matter)

Laboratory	Sample 1		Sample 2		Sample 3		Sample 4	
1	1.78	1.81	1.84	1.82	1.82	1.86	1.58	1.63
2	1.82	1.82	1.78	1.84	1.92	1.92	1.63	1.60
3	1.76	1.76	1.70	1.68	1.71	1.70	1.47	1.50
4	1.81	1.78	1.76	1.81	1.79	1.76	1.60	1.58
5	1.89	1.86	1.89	1.87	1.82	1.81	1.65	1.65
6	1.84	1.76	1.78	1.74	1.78	1.81	1.58	1.57
7	1.79	1.82	1.78	1.78	1.76	1.74	1.52	1.50
8	1.79	1.79	1.89	1.90	1.89	1.82	1.70	1.68

TABLE V. Summary of NIRS precision data

	Range	Number of laboratories	r <sub>95</sub>	R <sub>95</sub>
Barley moisture (% m/m)	12.7–15.8	7	0.3	1.5
Barley nitrogen (% m/m on dry matter)	1.57–2.14	19	0.1	0.3
Malt moisture (% m/m)	4.0–4.3	8	0.2	1.3
Malt nitrogen (% m/m on dry matter)	1.58–1.82	8	0.1	0.2

TABLE VI. Summary of precision data of EBC barley and malt reference methods

	Range	Number of laboratories	r <sub>95</sub>	R <sub>95</sub>
Barley moisture (% m/m) (5)	11–13	14–15	0.13	0.55
Barley nitrogen (% m/m on dry matter) (5)	1.5–2.1	26–27	0.04	0.10
Malt moisture (% m/m) (6)	3.8–7.3	17–26	0.13	0.6
Malt nitrogen (% m/m on dry matter) (6)	1.56–1.87	12–26	0.05	0.13

## REFERENCES

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