

COMMENTS ON THE DETERMINATION OF FRIABILITY AND UNMODIFIED GRAINS

SUBMITTED BY M. BENARD AND S. LIE ON BEHALF OF THE ANALYSIS COMMITTEE OF THE EUROPEAN BREWERY CONVENTION

The Analysis Committee of the European Brewery Convention is aware that the Friabilimeter sometimes is used on samples that do not comply with the field of application. The precision of the method is also overlooked by some users. The field of application, precision of the method as well as the need for frequent testing of the instrument is therefore stressed in more detail than in the earlier published method by the EBC Analysis Committee.

INTRODUCTION

The analysis method "Friability and unmodified Grains" was adopted by the EBC Analysis Committee in 1987 and published in the first supplement to *Analytica EBC IV*³ in 1989. The Analysis Committee has recently received some comments on incorrect use of friability values particularly in malt trading. The main problem seems to be that the precision of the method for friability determination is overlooked. In order to clarify this point, the field of applications, the procedure and the precision of the method, are commented upon.

COMMENTS ON THE METHOD

Field of Application

The friabilimeter method can be applied to all commercial pale malt samples produced from two row spring barleys. It is not recommended for other malted cereals. Under section 4.15 in *Analytica EBC* it was taken for granted that the method should be used for pale malts only. It was therefore not stressed that the method did not apply to dark malts.

Procedure

As with all analytical methods there are with in laboratory- and between laboratory errors also in the determination of friability and partly unmodified grains (PUG). The method for determination of friability is an instrumental method and the variations in results appearing between laboratories are therefore dependent upon two important factors:

1. The manufacturer of the apparatus, Pfeuffer GmbH, Germany, must be able to produce instruments of consistent quality. Spare parts, particularly the pressure roller and the spring, must be available in the same quality as in the original instrument.
2. The user of the instrument must test it with the Testkit supplied by the manufacturer. The test must be carried out at regular intervals. In addition to this test, standard malts with known friability should be run frequently.

Precision

The precision of the method varies with the degree of modification of the sample. In *Analytica EBC IV* the precision between laboratories and the reproducibility error, are stated for three levels of modification: 88, 80 and 65% friability. The coefficient of variation of the reproducibility error (CVS_R) is $\pm 1.1\%$, $\pm 2.9\%$ and $\pm 4.4\%$ respectively.

The EBC Analysis Committee determined the repeatability (r) and the reproducibility (R) figures of all methods published in the Malt Section of *Analytica EBC*. Friability and the amount of unmodified grains were also included in this collaborative test. The results of this test have recently been published². For friability the following equations are given for repeatability and reproducibility, where (m) is the mean value:

$$r_{95} = 12 - 0.11m$$

$$R_{95} = 21.7 - 0.192m$$

For partly unmodified grains the equations are:

$$r_{95} = 0.44 + 0.193m$$

$$R_{95} = 0.48 + 0.613m$$

The Friability method is an International Method, tested also by the ASBC¹ in 1988 at two levels of modification, with the following results for coefficient of variation between laboratories: at 85% friability CVS_R = $\pm 1.2\%$, and at 73% friability CVS_R = $\pm 3.0\%$. This is in agreement with the results obtained by EBC.

NATIONAL INTERLABORATORY TESTS

Monthly interlaboratory tests carried out in France in 1990/1991 and 1991/1992 clearly demonstrated an important systematic error between laboratories, very likely due to differences between the instruments.

In each test, all laboratories received 18 paired malts (2 malts each month). For each malt the mean friability was calculated and expressed as a percentage.

At the end of the test the mean differences were calculated for each laboratory, for odd-numbered malts and for even-numbered malts separately, and values obtained plotted on a graph in a way similar to Youden's method⁴, Fig. 1.

According to the results a systematic difference of $\pm 5\%$ in friability, and $\pm 25\%$ in partly unmodified grains may occur between laboratories.

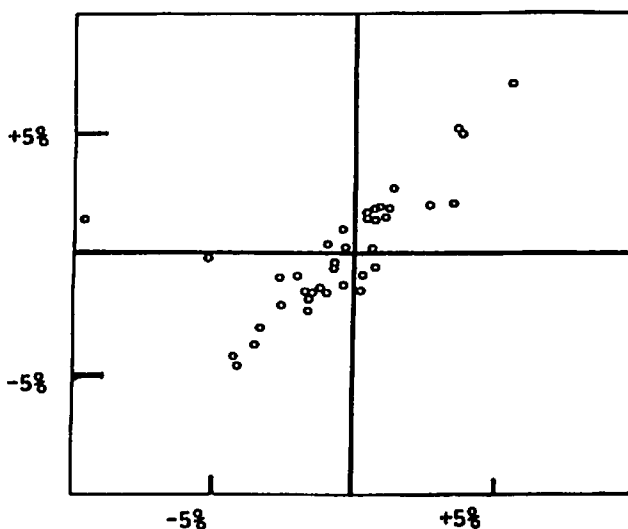


FIG. 1. Mean difference from mean ("true") friability for 38 laboratories in French test 1990/1991.

TABLE I. Reproducibility of friability measurements, France 1990/91

Pair	Mean friability	Reproducibility (R)
1-2	66.5	10.7
3-4	78.5	6.5
5-6	78	6.6
7-8	85	5.7
9-10	68.5	7.8
11-12	79.5	6.4
13-14	81	5.7
15-16	74	7.6
17-18	71	6.8

Reproducibility values (R), calculated from 9 pairs of malts with friabilities ranging from 65% to 85% are tabulated in Table 1 and plotted against friability in Fig. 2. The values obtained are in good agreement with those found in EBC or ASBC tests.

A Norwegian interlaboratory test in 1992 with 10 participating laboratories gave a reproducibility value (R) of 3.3 for a pair of malts with friability 88% (mean value). From Fig. 2 it can be seen that this value is also in good agreement with those mentioned above.

CONCLUSIONS

Both brewers and maltsters should be aware of the precision of the method as well as the field of application of the method when preparing down or accepting malt specifications, or when interpreting laboratory results.

The Friabilimeter is easy to use and can provide fast and valuable information on malt quality. However, it must be checked frequently with standard malts with high (80-85), medium (70-75) and low (60-65) friabilities, and users must

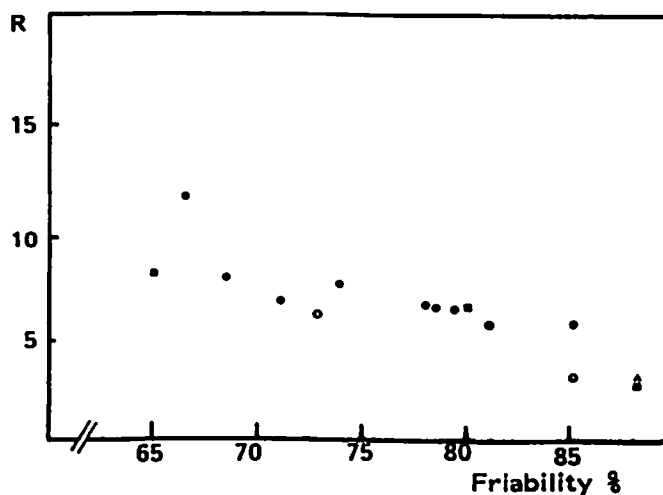


FIG. 2. Reproducibility values (R) for the tests carried out by EBC (■) and ASBC (○) and the national interlaboratory test in France (●) and in Norway (▲).

be aware of a possible systematic deviation of their results due to their own Friabilimeter.

REFERENCES

1. American Society of Brewing Chemists. Report of Subcommittee. *Journal of the American Society of Brewing Chemists* 1988, **46**, 119.
2. Benard, M., *Journal of the Institute of Brewing*, 1992, **98**, 81.
3. European Brewery Convention, *Analytica-EBC*, 4th edition, 1987.
4. Youden, W.J., *Statistical Techniques for Collaborative Tests*, Publication of the Association of Official Analytical Chemists, 1969.