



BREWUP

Elaboration,
homogenization, sampling
and packaging of an EBC
Reference Malt and
determination of its
analytical characteristics

Guidance

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FIELD OF APPLICATION

SCOPE

Procedures to determine manipulations employed for the selection of a batch of EBC reference malt, its homogenization, sieving, sampling, and packaging in order to obtain representative samples of malt for analytical purposes

RESPONSABILITIES

The TIL chairman is responsible for realizing all these steps using resources at his disposal. He can outsource all physico-chemical analyses to the Qualtech laboratory. He has to check the good conservation of the EBC malt and its analytical characteristics.

PRE REQUISITES

GENERALITIES

First of all, before the preparation of a new EBC reference malt:

- Check the conformity of the homogenization tool
- Make sure that an area is empty and safe to store all the materials regarding security specifications.
- Take into account the estimated total duration of 12 months in order to achieve all these steps, so that the results can be presented to one of the EBC Analysis Group biannual meetings.

PHYSICO-CHEMICAL ANALYSIS

In order to simplify the following procedure, the **Table 1** lists the different malt quality parameters evaluated, their units and the method applied to determine them.

Table 1 : Malt quality parameters evaluated throughout the procedure

Parameter	Unit	EBC Method
Moisture	%	4.2
Total proteins	% dm	4.3.1
Extract fine grind	% dm	4.5.1
Wort colour spectro	EBC units	4.7.1
Wort viscosity	mPa.s	4.8
Wort soluble proteins	% dm	4.9.1
Kolbach Index	%	4.9.1
Wort FAN	mg/L	4.10
Diastatic power	WK dm	4.12
Alpha amylase	DU dm	4.13
Modification	%	4.14
Homogeneity	%	4.14
Friability	%	4.15
Boiled wort colour	EBC units	4.19
Betaglucans	mg/L	8.13.2

r_{95} = Repeatability : the value less than or equal to which the absolute difference between two test results obtained under repeatability conditions may be expected to be with a probability of 95 %

R_{95} = Reproducibility : the value less than or equal to which the absolute difference between two test results obtained under reproducibility conditions may be expected to be with a probability of 95 %.

VALIDATION OF A BATCH

The validation of a new batch (malt EBC N) must be performed, either when the previous EBC N-1 stock is equal to 2000 units (around 12 months before its depletion), or when one of the analytical characteristic of the malt EBC N is significantly modified.

The procedure to follow is presented on *Figure 1*

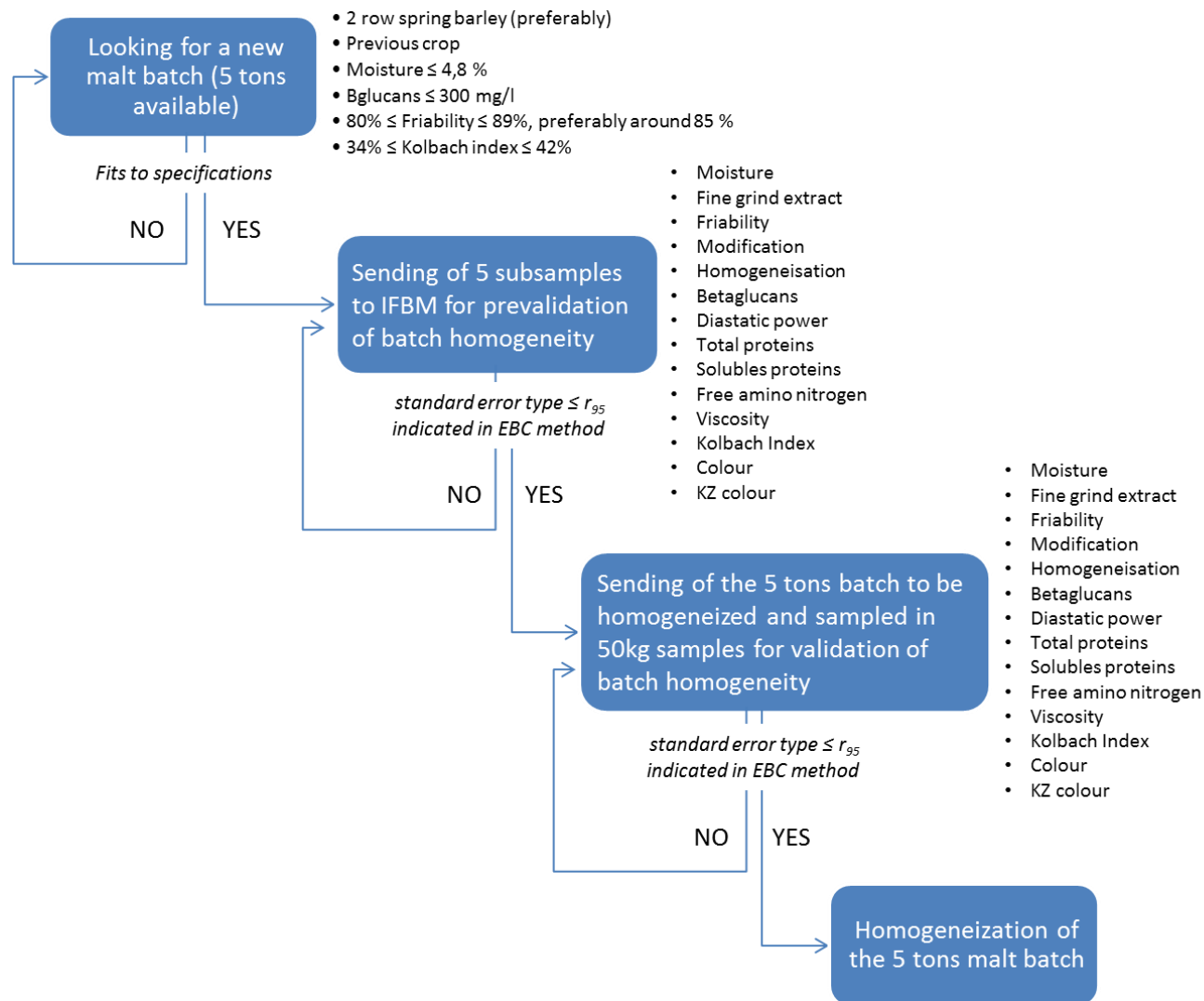


Figure 1 : Procedure to validate a new malt batch

HOMOGENIZATION OF BATCH

At the end of the previous step (*Validation of a batch*), 100 bags containing 50kg of malt are collected.

The principle of homogenization/sampling can be achieved thanks a mechanical divider (Boerner type) with ten outputs and a charging hopper (50 kg malt), see *Figure 2*.

In order to be homogenized, each bag must be loaded in the given order from n°1 to n°100 one after each other on the divider to recover 100 news bags of 50 kg by series of 10 bags placed under the 10 outputs. Afterwards, the first bag of each series is once again loaded on the divider to recover 10 new bags, and so on. Finally, 100 bags of homogenized malt are obtained.

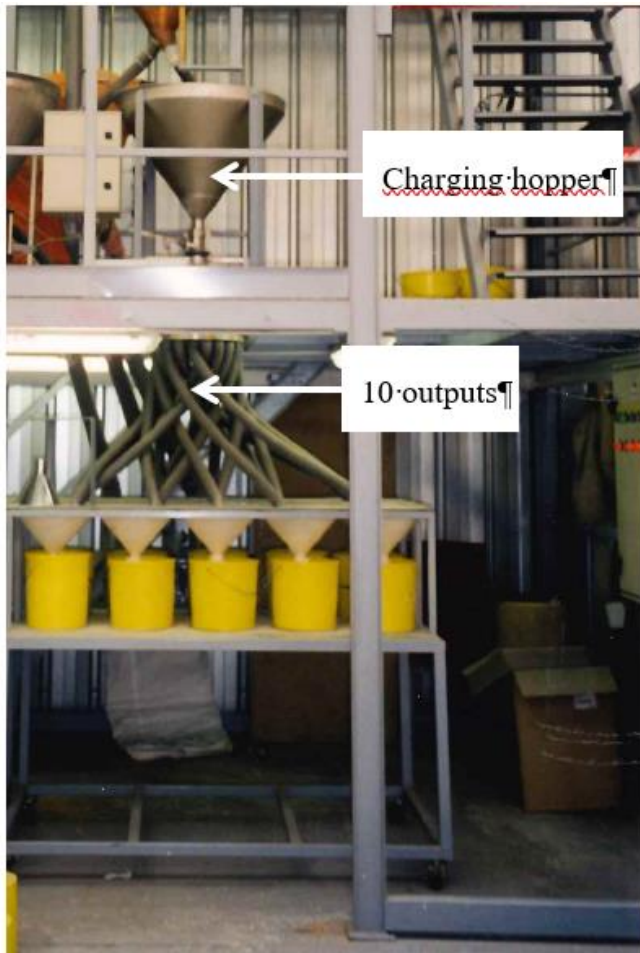


Figure 2 : Mechanical divider with 10 outputs and a 50 kg charging hopper

CLEANING AND CALIBRATION OF THE BATCH

After the homogenization, the 100 bags coming from the 5 tons batch are cleaned and graded through a grader/barley cleaner and recovered in hundred 50kg containers hermetically sealed. The equipment available at IFBM is presented on **Figure 3**



Figure 3 : Grader/Barley cleaner available at IFBM

SAMPLING

The 100 containers containing cleaned and graded malt are loaded successively in the given order from n°1 to 100 on the divider previously cleaned (*Figure 2*). The malt is in this way sampled by series of ten 1L boxes (~ 0,625 kg net malt) placed at the several outputs. Each box is labelled with a serial number corresponding to its filling rank and then hermetically sealed. Around 6000 boxes should be obtained at the end of this procedure with a 5 tons sample at the beginning.

ANALYTICAL CHARACTERIZATION OF THE MALT EBC

Once the sampling step is finalized, two boxes are sent to 30 laboratories in order to determine the value of each malt parameter cited in *Physico-chemical analysis*. Results are collected by IFBM and treated statistically. In that way, two limits (r_{95} and R_{95}) are calculated and associated to the parameter value, after outliers have been excluded from analysis (*procedure in Annex*).

The EBC Analysis Group will have a look at the results of the ring test of validation during one of their annual meetings. After comparison of the standard deviation for the most important parameters with the r_{95} and R_{95} of the corresponding methods, the EBC Analysis Group will validate (or not) the new EBC standard malt batch. Thereafter, the new batch can be sold with the reference values. Informations on the new batch will be available on EBC website.

STORAGE AND QUALITY FOLLOW-UP OF THE MALT EBC

Boxes containing the new “malt EBC” are stored in the storage hall from IFBM at room temperature on wooden pallets (type EUR) in layers separated by PVC spacers. In these conditions, these samples are considered to be stable over about a 3 years period.

In order to be able to follow an eventual drift of the quality of the malt EBC, it can be incorporated anonymously into malt ring tests organized by IFBM.

ANNEX

Scope of the file is the evaluation of collaborative trials for the determination of precision figures (repeatability limit and reproducibility limit) in accordance with ISO 5725-2:1994.

The file can be applied for the evaluation of collaborative trials with not more than 30 participating laboratories, not more than six levels and not more than five parallel measurements (repetitions) in a single laboratory for the same parameter under repeatability conditions.

A short guide for the use of the file:

1. Open "AC collaborative trial.XLS" file.
2. Save the file with a name as "XXX collaborative trial.XLS" or whatever you want to use it for.
3. Enter the raw data into the B4:F33 ranges of Sample A, Sample B,...and Sample F worksheets, respectively.

Now you should decide whether you want to use graphical consistency tests or numerical tests to seek outliers and stragglers.

4. If you decided to use Mandel's statistics, look at "Mandel's k" and "Mandel's h" diagrams for graphical consistency tests. Adjust the diagrams to the raw data arrays. The default set is 30 data per series that is the maximum number of participants. After due consideration please decide which test results will be rejected from the further calculations and go to point 9.
5. If your decision is to use numerical tests for the outliers and stragglers, please click to "Numerical tests" worksheet.
6. In the cells of B7:G7 range you can find the Cochran's statistics for sample A, sample B,...sample F. In the B8:G8 range there are the critical values of the Cochran's statistics for 1% and in B9:G9 range the critical values of the Cochran's statistics for 5%. If any of the calculated Cochran's statistics in the range of B7:G7 is larger than the critical value at 1% for the same sample, at this level the test results with the highest variance (standard deviation or difference) have been indicated as outlying results. In the 10th row in blue colour the ordinal number of the outlying laboratory can be found. Come to a decision on the rejection and/or retain of the outliers from level to level. Complete the rejection as put down in point 9. Repeat the test if necessary.
7. In the cells of B12:G12 range you can find the computed Grubbs' statistics for the largest mean value for sample A, sample B,...sample F. In the B13:G13 range there are the Grubbs' statistics for the smallest mean value for sample A, sample B,...sample F. The critical values of the Grubbs' statistics for 1% are in the B14:G14 range and those for 5% in B15:G15 range. If any of the calculated Grubbs' statistics in the range of B12:G13 is larger than the critical value at 1% for the same sample, at this level the test results which belong to the highest or to the smallest mean value have been indicated as outlying results. In the 16th row in blue colour the ordinal number of the outlying laboratory with the highest mean and in the 17th row in blue colour the ordinal number of the outlying laboratory with the smallest mean may be found. Come to decision on the rejection and/or retain of the outliers from level to level. Complete the rejection as put down in point 9. Repeat the test if necessary.

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8. At levels where no outlier or straggler was indicated by the Grubbs' test, accomplish the Grubbs' test for the two largest and two smallest mean values.

DO NOT MAKE ANY TEST FOR THE TWO LARGEST OR SMALLEST VALUES IF OUTLIER AND/OR STRAGGLER WAS INDICATED AT THE GIVEN LEVEL BY GRUBBS' TEST FOR THE LARGEST OR FOR THE SMALLEST MEAN.

In range B21:G21 of the "Numerical tests" worksheet you will find the computed Grubbs' statistics for two largest and in range B22:G22 the computed Grubbs' statistics for two smallest mean values. If any of the computed Grubbs' statistics are smaller than the critical Grubbs' value for 1% then the two largest or smallest mean values have been indicated as outliers. Come to decision on the rejection and/or retain of the outliers from level to level. Complete the rejection as put down in point 9.

DO NOT REPEAT THE TEST.

9. Look for the outlying test result at the worksheet of the appropriate level and in the red (!) array B37:F66 delete the outlying test results, which are all parallel results of the outlying laboratory. DO NOT DELETE THEM FROM THE RAW DATA IN THE BLACK AREA.
10. If all rejections have been completed, click to "Summary" worksheet to see the results of the collaborative trial.
11. Clicking on "Diagram" the relationships between r and m between R and m can be studied. In case of less than 30 participants, the series should be adjusted to the actual ranges in the "Summary" worksheet. The equations of the trend lines as well as the coefficients of determination (R^2) are visible on the sheet. To make the evaluation easier, please check the significance of the relationship clicking to the "Significance" worksheet.
12. Compose a report and save the file.

NOTE:

EBC Analysis Group traditionally uses the terms repeatability and reproducibility for the repeatability limit and reproducibility limit, respectively. For the calculation of these figures we use the formulas:

$$r_{95} = 2,8 \cdot s_r \text{ and } R_{95} = 2,8 \cdot s_R.$$

The factor 2,8 is really $t \cdot \sqrt{2}$, where t is the Student's statistic. It means that for the Student statistic we use 2 at all degrees of freedom with a probability level of 95%. It was put down in ISO 5725:1988 on the supposition that normally the number of participating laboratories is above 25. ISO 5725:1994 doesn't give any formula for the calculation of repeatability limit and reproducibility limit. It suggests reporting the appropriate standard deviations. However, 2 is acceptable for the Student's statistic only if the degree of freedom is sufficiently high. It means at least 25-30 participants.

The table gives some figures to demonstrate the situation:

Number of participants	Student's statistic	Multiplication faktor
6	2,57	3,64
8	2,36	3,34
10	2,26	3,20
12	2,20	3,11
14	2,16	3,06
16	2,13	3,01
18	2,11	2,98
20	2,09	2,96
25	2,06	2,92
30	2,05	2,89
35	2,03	2,87
40	2,02	2,86
45	2,02	2,85
50	2,01	2,84
55	2,00	2,84
60	2,00	2,83

Taken into consideration the number of participants in a usual EBC Analysis Group collaborative trial, in the "Summary" worksheet you will find a factor that slightly deviates from 2.8 and depends on the degree of freedom that is on the number of participants.

ACKNOWLEDGEMENT

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DISCLAIMER

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