

DETERMINATION OF REPEATABILITY AND REPRODUCIBILITY OF EBC ACCEPTED METHODS-II PALE MALT COLOUR, HOPS, SOLID ADJUNCTS

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Methods for the determination of pale malt colour, hops analysis and solid adjuncts analysis published in Analytica EBC have been collaboratively tested by the European Brewery Convention Analysis Committee. Repeatability (r) and Reproducibility (R) are presented.

Key Words: Collaborative test, analysis methods, hops, solid adjuncts, precision.

1. INTRODUCTION

The general procedure followed was the same as for malt and laboratory wort, which was described in a preceding paper¹, where definitions and an example can also be found.

samples for determination of moisture, (method 7.1), lead conductance value (method 7.3.2) and resins (method 7.3.4). - the second. In which 23 laboratories (EBC, AHA and ASBC) received 8 samples (4 pellets and 4 extracts) for determination of α and β acids by HPLC (method 7.4.1).

TABLE I. Repeatability (r_{95}) and Reproducibility (R_{95}) of hops analysis according to Analytica EBC 4th ed.

Method		Number of levels	Range	r_{95}	R_{95}	Number of results
7.1-Moisture	%	4	3-10 10-15	0.27 0.40	1.2 2.0	15
7.3.2-L.C.V.	% as is	4	2.5-10	0.20	1.0	12
7.3.4-Total Resins	% as is	4	10-35	0.55	1.4	11-12
7.3.4-Soft Resins	% as is	4	9-30	$r = 0.17 + 0.016 m$	$R = 1.17 + 0.052 m$	11-12
7.3.4-L.C.V.	% as is	4	3-16	$r = 0.024 + 0.033 m$	$R = 0.41 + 0.057 m$	11
7.3.4- β Fraction	% as is	4	5-15	$r = 0.046 + 0.039 m$	$R = 0.97 + 0.066 m$	11-12
7.3.4-Hard resins	% as is	4	1-5	0.42	1.5	12
7.4.1- α Acids	% as is	8	4-48	$r = 0.1 + 0.032 m$	$0.2 + 0.08 m$	16-18
7.4.1- β Acids	% as is	8	2-32	$r = 0.17 + 0.023$	$0.3 + 0.08 m$	17-19

Particular procedures connected with these interlaboratory tests are described under each chapter.

2. PALE MALT COLOUR

In a previous test (1), the range was too small to obtain reliable results, and a new test was organized.

2.1 Procedure

Seven malt samples were sent to EBC members for determination of wort colour in duplicate according to method 4.7.

Results from 14 laboratories were collected.

2.2 Conclusions

The following values have been accepted by the Statistics Subcommittee.

- Range: 2.5 to 6.5 EBC units
- Repeatability: $r_{95} = 0.3$
- Reproducibility: $R_{95} = 1.0$

There is no relation between r or R and the measured value within the range observed, which corresponds to pale malt.

3. HOPS

3.1 Procedure

Two interlaboratory tests were organized:

- the first. In which 15 laboratories (EBC and AHA, Arbeitsgruppe für Hopfen Analyse) received 4 hop pellets

3.2 Results and Conclusions

Results are given in Table 1.

Special comments are given for the following methods:

7.3.2 Lead Conductance Value (Toluene extraction):

One of the 4 pellets samples was hop powder containing whole lupulin glands and results showed a very high dispersion, due to incomplete extraction in some laboratories. Much better results were observed on that sample with method 7.3.4. It is concluded that such samples *should not* be analyzed with method 7.3.2. This statement should be included in the paragraph: "Field of application".

Note: For determination of lead conductance value, it was observed that method 7.3.4 gives systematically higher results than method 7.3.2. (see table 2)

TABLE II. Compared Lead Conductance Values (L.C.V.) % obtained by methods 7.3.2 and 7.3.4

Sample	LCV 7.3.2	LCV 7.3.4
1	2.47	3.12
2	9.80	10.01
3	2.79	3.20
4*	(12.23)	16.03

* Hop powder containing whole lupulin glands.

TABLE III. Repeatability (r) and reproducibility (R) of solid adjuncts analysis according to Analytica EBC, 4th ed.

Method:		Number of levels	Range	r		R		Number of results
6.1-Moisture	%	4	11-13	0.15	0.94	11-13		
6.2.1-Extract	% as is	4	78-91	0.85	3.2	7		
6.2.2-Extract	% as is	4	77-91	0.85	2.0	7		
6.2.3-Extract	% as is	4	77-91	0.53	1.9	7		
6.4-Fatty substances	% as is	3	0.6-1.2	0.09	0.19	10		

7.4.1 Alpha acids (N + Ad + Cohumulone) by HPLC

One laboratory using a 3 years old standard extract obtained systematically high results. Its results were discarded and it was decided to add the following statement in the method, under reagent:

"The standard extract should be kept in a refrigerator when not in use and replaced every year"

4. SOLID ADJUNCTS

4.1 Procedure

Four samples:

- 2 maize grits with different fatty substances contents
- 1 maize flour with a high fatty substances contents
- 1 refined maize gritz (maize starch)

were sent to EBC and ASBC laboratories. Results from 13 laboratories were collected (12 EBC and 1 ASBC).

TABLE IV. Compared Fatty Substances results (% as is) obtained by method 6.4 and alternative method

Sample	F.S. 6.4(CCl ₄)	F.S. alternative method (Petroleum ether)
1	0.60	0.51
2	0.82	0.77
3	1.20	1.13

4.2 Results and Conclusions

Table 3 summarizes the results obtained.

Special comments are given for the following methods:

EXTRACT

Three methods for extract determination appear in Analytica EBC, 4th ed. For maize grits, method 6.2.1 has a tendency to give higher results than method 6.2.3.

For maize starch, the 3 methods gave the same mean values.

FATTY SUBSTANCES

An alternative method, with the same procedure as method 6.4 but in which petroleum ether replaced carbon tetrachloride was tried. (Petroleum ether is used both in ASBC and EEC methods and is less toxic than Carbon Tetrachloride) Repeatability and reproducibility were equivalent, but systematically lower results were obtained. (see Table 4)

REFERENCES

1. Benard, M., *Journal of the Institute of Brewing*, 1992, 98, 2, 81.