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Determination of alcohol chill haze in beer

Submitted on behalf of the Analysis Committee of European Brewery Convention

In order to examine the Chapon's alcohol chill haze test in beer (3, 4) by nephelometry, a collaborative trial was carried out using six beer samples with different levels of alcohol chill haze. Fifteen laboratories of the members of the EBC Analysis Committee took part in the collaborative trial. The method for determination of alcohol chill haze in beer yields results with satisfactory repeatability (r_{95}) and reproducibility (R_{95}). The EBC Analysis Committee decided to include the method for determination of alcohol chill haze in beer in Analytica-EBC and recommend it as a guideline method.

Descriptors: Analysis methods, chill haze, nephelometry

1 Introduction

The EBC Analysis Committee decided to examine the Chapon's alcohol chill haze test in beer (3, 4) by nephelometry. In autumn 2001 a collaborative trial was carried out using six beer samples with different levels of alcohol chill haze. Fifteen laboratories of the members of the EBC Analysis Committee took part in the collaborative trial.

2 Experimental

The organization of the collaborative trial and the statistical treatment of the data were performed according to the procedure given in the International Standard ISO 5725 (1, 6).

Six stabilized and pasteurised beer samples with different alcohol chill haze levels were circulated to 15 laboratories. Fourteen laboratories analysed all beer samples and carried out two measurements on each sample (one laboratory analysed two beer samples) using the modification of the Chapon's alcohol chill haze test (2).

3 Results and discussion

The results of two laboratories showed big systematic deviations as compared to all other results. The original data are given in Table 1. The level 3 of laboratory 3 was irreconcilable with the other data and was excluded from the computation. Mandel's k and h statistics are used for testing the consistency of results. Three Mandel's k statistical outliers, one Mandel's h statistical outlier and two Mandel's h stragglers were excluded from the computation. The precision data are summarized in Table 2. From this table it seems that both r_{95} and R_{95} tend to increase with higher values of mean. The final r_{95} and R_{95} values are:

$$r_{95} = 0,160 + 0,055 m \quad R_{95} = 0,513 + 0,544 m$$

These results were obtained using nephelometers (haze meters) with 90° measuring angle, but with certain differences in measuring wavelength and geometry of the optical systems. In addition,

Table 1 Results: alcohol chill haze in beer (EBC units).

Laboratory	Level					
	1	2	3	4	5	6
1.	0,43	1,33	0,49	1,40	9,18	5,15
	0,45	1,38	0,49	1,35	9,07	5,03
2.	0,40	1,39	0,47	1,53	8,64	4,65
	0,38	1,47	0,54	1,47	8,48	4,57
3.	0,66	3,81	3,88 ^x	1,62	8,81	5,09
	0,58	4,01	4,50 ^x	1,82	8,88	5,86
4.	0,61 ⁺	–	–	1,71	–	–
	0,47 ⁺	–	–	1,45	–	–
5.	0,43	3,12	0,97	1,69	9,74	6,60
	0,50	3,11	0,87	1,73	9,67	6,73
6.	0,38	3,85	0,93	1,80	9,65	5,76 ⁺⁺
	0,42	3,90	0,95	1,75	9,48	7,02 ⁺⁺
7.	0,39	1,41	0,40	1,20 ⁺⁺	7,60	3,92
	0,32	1,36	0,45	1,63 ⁺⁺	7,34	4,15
8.	0,58	2,23	0,71	1,30	7,41	4,30
	0,63	2,31	0,81	1,39	7,75	4,18
9.	0,37	2,28 ⁺⁺	0,37	1,41	9,06	5,15
	0,40	1,56 ⁺⁺	0,38	1,17	9,08	4,75
10.	0,24 [*]	0,54	0,33	1,55	5,67	1,68 [*]
	0,20 [*]	0,50	0,30	1,66	5,56	1,80 [*]
11.	0,36	0,64	0,41	0,83 ^{**}	5,02	3,04
	0,28	0,84	0,34	0,76 ^{**}	5,20	2,41
12.	0,45	2,11	0,64	1,60	6,37 ⁺	3,09
	0,45	2,16	0,61	1,50	5,95 ⁺	2,60
13.	0,54	2,45	0,70	1,74	8,90	5,05
	0,46	2,50	0,77	1,69	8,70	5,33

* = Mandel's h statistic straggler

** = Mandel's h statistic outlier

^x = excluded from the computation

⁺ = Mandel's k statistic straggler

⁺⁺ = Mandel's k statistic outlier

Table 2 Summary of the precision data (calculated from data of table 1).

Sample	Beer 1	Beer 2	Beer 3	Beer 4	Beer 5	Beer 6
Number of laboratories	12	11	11	11	12	10
Grand mean (m)	0,456	2,110	0,588	1,560	7,967	4,583
Repeatability standard deviation (Sr)	0,048	0,070	0,042	0,097	0,147	0,280
Reproducibility standard deviation (S _R)	0,101	1,162	0,224	0,180	1,579	1,208
Repeatability (r ₉₅)	0,133	0,195	0,119	0,272	0,412	0,785
Reproducibility (R ₉₅)	0,283	3,255	0,629	0,505	4,421	3,383
Coefficient of variation of repeatability (CVSr)	10,44	3,31	7,21	6,23	1,85	6,12
Coefficient of variation of reproducibility (CVS _R)	22,15	55,09	38,20	11,55	19,82	26,36

the measured turbidities were results of formation of very small quantities of alcohol chill hazes (ppm or less). However, the coefficient of variation of repeatability (CVSr) and the coefficient of variation of reproducibility (CVS_R) are in accordance with requirements established by Horwitz (5).

4 Conclusions

The method for determination of alcohol chill haze in beer yields results with satisfactory repeatability (r₉₅) and reproducibility (R₉₅). The EBC Analysis Committee decided to include the method for determination of alcohol chill haze in beer in Analytica-EBC and recommend it as a guideline method.

5 Bibliography

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