

Determination of Sensitive Proteins in Beer by Nephelometry

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on behalf of the Analysis Committee of the European Brewery Convention

Introduction

The EBC Analysis Committee decided to test a modification of Chapon's method (2, 3) for the determination of sensitive proteins in beer by nephelometry.

In autumn 2001 a collaborative trial was carried out using six beer samples with different levels of sensitive proteins. Sixteen laboratories of the members of the EBC Analysis Committee took part in the collaborative trial.

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, 5).

Six stabilized and pasteurized beer samples with different levels of sensitive proteins were circulated to 16 laboratories. Fifteen laboratories analysed all beer samples and carried out two measurements for each (one laboratory analysed 4 beer samples).

Results and Discussion

The original data are given in Table I. Mandel's **k** and **h** statistics are used for testing the consistency of results. Two Mandel's **k** statistical outliers and two Mandel's **h** statistical outliers were excluded from the computation. The precision data are summarized in Table II. From this table it seems clear that both r_{95} and R_{95} tend to increase with higher values of mean. The final r_{95} and R_{95} values are:

$$r_{95} = 0.240 + 0.044 m \quad R_{95} = -0.030 + 0.384 m$$

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. On the other hand, the measured turbidities were results of formation of very small quantities of protein-tannic acid hazes (ppm or less). That means that coefficient of variation of repeatability (CVS_r) and coefficient of variation of reproducibility (CVS_R) are fully in accordance with requirements established by Horwitz (4).

TABLE I – Results: Sensitive Proteins in Beer (EBC units, after addition of 5 mg/L tannic acid)

Laboratory	Level					
	1	2	3	4	5	6
1	1.26/1.19	1.35/1.35	1.53/1.55	2.52/2.69	4.28/4.40	7.26/7.40
2	1.25/1.39	1.55/1.61	1.62/1.66	2.63/2.79	4.72/4.83	7.72/7.60
3	1.54 ⁺⁺ /1.07	1.72/1.71	1.71/1.95	2.49/2.33	4.13/4.65	6.81 ⁺⁺ /5.99
4	1.44/1.04	1.49/1.56	1.59/1.92	2.34/2.15	—/—	—/—
5	0.88/1.01	1.84/1.90	1.77/1.75	2.16/2.51	4.29/3.97	8.77/8.14
6	1.12/0.89	1.74/1.59	1.68/1.52	2.07/1.97	3.14/3.07	7.66/7.58
7	1.61/1.75	1.82/1.83	1.73/1.92	2.95/2.54	4.14/4.10	8.16/8.12
8	1.27/1.28	1.60/1.62	1.79/1.69	2.86/2.88	3.69/3.80	7.52/7.47
9	1.26/1.23	1.38/1.58	1.36/1.60	2.69/2.42	5.56/5.31	7.72/7.91
10	1.54/1.54	1.22/1.26	1.68/1.80	2.28/2.36	3.59/3.69	7.56/7.05
11	1.55/1.44	1.66/1.78	1.97/1.86	2.80/2.72	4.68/4.79	5.37/5.36
12	1.00/0.90	1.31/1.44	1.46/1.49	2.68/2.97	4.16/4.42	6.56/6.40
13	1.10/1.10	1.08/1.28	1.32/1.28	2.76/2.54	4.89/4.64	6.42/6.79
14	1.65/1.55	1.88/1.67	1.79/1.73	2.69/2.81	4.27/4.80	7.28/7.28
15	1.37/1.27	1.75/1.67	1.74/1.86	2.70/2.86	4.44/4.56	7.49/7.39
16	1.52/1.35	1.73/1.86	1.92/1.80	3.32 ^{**} /3.37	1.65 ^{**} /2.18	5.02/5.01

⁺⁺ Mandel's **k** statistic outlier

^{**} Mandel's **h** statistic outlier

TABLE II – Summary of the precision data (calculated from data of Table I)

Sample	Beer 1	Beer 2	Beer 3	Beer 4	Beer 5	Beer 6
Number of laboratories	15	16	16	15	14	14
Grand mean (m)	1.292	1.588	1.689	2.572	4.322	7.143
Repeatability standard deviation (Sr)	0.107	0.082	0.106	0.150	0.182	0.180
Reproducibility standard deviation (S _R)	0.242	0.220	0.184	0.272	0.586	0.984
Repeatability (r ₉₅)	0.301	0.230	0.297	0.420	0.509	0.505
Reproducibility (R ₉₅)	0.676	0.615	0.515	0.761	1.641	2.755
Coefficient of variation of repeatability (CVS _r)	8.31	5.17	6.28	5.83	4.21	2.52
Coefficient of variation of reproducibility (CVS _R)	18.70	13.83	10.90	10.57	13.56	13.77

Conclusions

The method for the determination of sensitive proteins in beer by nephelometry yields results with acceptable repeatability (r₉₅) and reproducibility (R₉₅). The EBC Analysis Committee decided to include the method for the determination of sensitive proteins in beer by nephelometry in Analytica – EBC.

BIBLIOGRAPHY

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