

DETERMINATION OF THE SOLUBLE IRON CONTENT OF FILTER AIDS: A COLLABORATIVE TRIAL

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The international method for the determination of the soluble iron content of filter aids has been collaboratively tested by members of the Analysis Committee of the European Brewery Convention to obtain repeatability (r_{95}) and reproducibility (R_{95}) values. It was judged that precision values were dependent on the quantity of the soluble iron content of the filter aids over the range 32 to 220 mg/kg. Repeatability (r_{95}) value of 0.050 m and reproducibility value (R_{95}) of 0.255 m were obtained over this range.

Key Words: Collaborative trial, filter aids (analysis methods for), soluble iron (determination of)

INTRODUCTION

The European Brewery Convention Analysis Committee formed a sub-committee to supervise the methods for the analysis of filter aids. After due consideration it was decided to collaboratively test the method for the determination of soluble iron content of filter aids (EBC-Analytica 11.5) to obtain precision values.

EXPERIMENTAL

The organisation of the collaborative trial and the statistical treatment of the data were carried out according to the procedures given in the International Standard ISO 5725⁴. A uniform-level experiment was employed and six samples were circulated to twelve laboratories. Participating laboratories were asked to determine the soluble iron content of filter aids, in duplicate, to one decimal place, according to Analytica-EBC 11.5 Soluble Iron (International Method). Although the participants were permitted to use their own routine procedures

TABLE II Summary of precision data (units mg/kg)

Sample	A	B	C	D	E	F
No of labs	10	11	11	11	11	10
Mean	102.2	67.6	219.0	92.2	179.8	32.4
s_p^2	0.44	0.58	19.38	3.96	14.07	0.73
atL^2	91.14	19.72	174.55	104.46	418.26	7.41
CVr	0.65	1.12	2.01	2.16	2.09	2.63
CVR	9.34	6.57	6.03	11.09	11.38	8.41
r_{95}	1.9	2.1	12.5	5.6	10.6	2.4
R_{95}	27.1	12.7	39.4	29.5	58.8	8.1

for the determination of the iron concentration of the extract, eleven out of the twelve participants followed the spectrophotometric method of Analytica-EBC 9.10.11. Laboratory No. 5 carried out AAS-analysis. Two of the participating laboratories determined the iron concentration both spectrophotometrically and by AAS and ICP-AES methods, respectively.

RESULTS AND DISCUSSION

Raw data of the interlaboratory precision test are presented in Table I. One laboratory (No. 8) was identified an outlier and was rejected. Two outliers of the rest of the results were identified and rejected. The precision data are summarised in Table II. It was judged that both repeatability (r_{95}) and reproducibility (R_{95}) were dependent on the mean value (m). Values of r_{95} and R_{95} respectively were: 0.050 m and 0.255 m over the range of 32 to 220 mg/kg soluble iron content.

CONCLUSIONS

The Analysis Committee of the European Brewery Convention judged as acceptable both the repeatability and reproducibility values, obtained in the collaborative trial, for the determination of the soluble iron content of the filter aids. The Committee has approved a reworded version of the method 11.5 Soluble Iron (International Method) in Analytica-EBC.

REFERENCES

1. European Brewery Convention, Analytica-EBC, 4th Edition, 1987. Method 11.5.
2. Methods of Analysis of the American Society of Brewing Chemists, Filter Aids-4, Iron pickup by beer, 1976.
3. Brautechnische Analysemethoden Band III, Methodensammlung der Mitteleuropäischen Brautechnischen Analysenkommission (MEBAK), Selbstverlag der MEBAK, 1982.
4. International Standard, Precision of test methods—Determination of repeatability and reproducibility for a standard test method by interlaboratory tests, ISO 5725, 1986.

TABLE I Raw data, as received for soluble iron content

Laboratory	A	B	C	D	E	F
1	90.3	63.9	217.8	82.9	141.2	31.2
	90.6	64.2	217.8	83.2	141.4	29.8
2	103.9	71.9	200.3	84.7	157.5	31.4
	103.8	71.8	200.3	84.6	157.7	31.4
3	102.7	70.4	227.0	101.6	187.4	35.2*
	101.6	67.8	225.3	103.3	178.6	41.7*
4	116.6	70.2	193.0	87.2	171.5	34.9
	114.5	70.2	203.7	89.5	178.2	36.4
5	92.4	58.8	212.4	96.8	164.0	31.6
	92.8	59.6	211.6	95.2	162.4	30.8
6	107.0	72.4	223.1	107.3	193.8	36.2
	107.5	70.2	229.3	105.5	200.2	35.2
7	114.4	73.6	245.6	106.0	195.0	35.3
	114.8	73.6	244.1	106.8	196.5	36.0
8	109.8	140.0**	228.3	140.6**	128.8	54.7**
	108.1	141.3**	222.3	144.2**	130.2	54.8**
9	130.0**	67.6	236.0	72.0	208.0	27.6
	120.4**	67.2	225.2	78.0	212.0	29.2
10	110.0	70.0	210.0	95.0	180.0	35.0
	110.0	70.0	220.0	100.0	175.0	35.0
11	94.4	64.3	225.4	89.5	196.8	31.9
	93.5	64.5	224.7	90.2	206.8	29.9
12	92.7	62.5	210.3	86.3	176.7	30.4
	91.4	62.9	215.6	83.0	176.1	29.1
7	113.3	70.6	229.6	102.4	199.8	24.8
AAS	111.8	70.1	236.8	101.3	212.0	35.3
13	99.3	75.9	233.6	94.2	203.7	33.8
ICP AES	99.5	69.4	229.9	95.4	205.2	31.7

*rejected as outlier (Cochran's test; $p \leq 0.01$)

**rejected as outlier (Dixon's test; $p \leq 0.01$)