

## DETERMINATION OF ALUMINIUM IN BEER BY GF-AAS

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**A method for the determination of aluminum in beer, has been collaboratively tested. The method tested relies on atomic absorption spectrometry with atomization in a graphite furnace. Five pairs of beers, with concentrations ranging from 96 to 1000 µg/litre, were analysed by eleven laboratories. The repeatability ( $r_{95}$ ) values ranged from 10 to 69 µg/l, and the reproducibility ( $R_{95}$ ) values ranged from 70 to 465 µg/l. A second collaborative trial with a slightly different method protocol gave no improvement. Due to the high reproducibility values the method was not adopted for inclusion in Analytica-EBC.**

TABLE 1. Raw data, collaborative trial 1 (units: µg/litre)

Lab	Sample Pair A		Sample Pair B		Sample Pair C		Sample Pair D		Sample Pair E	
1a	178	189	287	291	72	78	986	946	140	135
1b	198	213	344	322	84	93	938	902	171 <sup>3</sup>	133 <sup>3</sup>
2	224	223	358	330	103	108	1065	1096	155	172
3	212	174	252	272	144 <sup>1</sup>	128 <sup>1</sup>	1120	1180	110	114
4	238	255	415	425	112	113	1297	1339	268 <sup>1</sup>	300 <sup>2</sup>
5	201	196	306	285	93	97	908	890	139	146
6	199	202	313	290	89	99	936	920	148	156
7	292	265	412	445	153	154	1190	1170	178	170
8	160	150	321	322	53	70	916	948	110	121
9	196	208	325	323	94	100	860	910	149	151
10	123	167	218	145	70 <sup>1</sup>	112 <sup>1</sup>	733	739	98	95
11	208	157	304	355	74	74	1119 <sup>1</sup>	894 <sup>1</sup>	112	130

1. The difference is an outlier according to Dixon's test.
2. The average is an outlier according to Dixon's test.
3. The difference is a straggler according to Dixon's test.

TABLE 2. Summary of precision data, collaborative trial I.

	Sample Pair A	Sample Pair B	Sample Pair C	Sample Pair D	Sample Pair E
Number of results	12	12	10	11	10
Grand mean, (µg/l)	201, 2	319, 1	95, 7	1000	136, 4
$s_r$ (µg/l)	18, 5	22, 7	3, 6	24, 8	6, 3
$r_{95}$ (µg/l)	51, 9	63, 6	10, 1	69, 4	17, 6
$cv_r$ (%)	9, 2	7, 1	3, 8	2, 5	4, 6
$s_r$ (µg/l)	38, 7	67, 3	25, 6	166, 0	25, 0
$R_{95}$ (µg/l)	108, 3	188, 6	71, 7	465, 0	70, 1
$cv_R$ (%)	19, 2	21, 1	26, 8	16, 6	18, 4

**Key Words:** Aluminium (analysis of), beer, atomic absorption spectrometry, graphite furnace, collaborative trial.

### INTRODUCTION

The aim of the subcommittee was to evaluate a method for the analysis of aluminum in beer. The method evaluated was published by Wagner and McGarrity: "Determination of Aluminium in Beer by Graphite Furnace Atomic Absorption Spectrometry" (1). Two changes in this method were introduced as proposed by the authors (2): potassium dichromate was substituted for magnesium nitrate as the matrix modifier, and a cool down step prior to atomization was included in the temperature programme. This modified version of the

method was also tested by the American Society of Brewing Chemists (ASBC).

### TESTING PROCEDURE

The organisation of the collaborative test and the statistical evaluation of the data were carried out according to the procedures given in the International Standard ISO 5725 (3). A split level design was employed.

Five pairs of beer, with aluminium contents from approx. 100 to 1000 µg/litre were sent to 11 participating laboratories. The different levels were obtained by addition of an aluminium standard solution. The samples were bottled in acid washed polypropylene bottles and pasteurized. The analyses were performed within four weeks after bottling. An aqueous aluminium standard was distributed to all participating laboratories together with the samples, and this was used for the calibration.

### RESULTS AND DISCUSSION

The raw data of the collaborative trial are presented in Table 1, and a summary of the statistical calculations is given in Table 2. One laboratory returned two sets of results, one using deuterium and one using Zeeman background correction. Five pairs of results were omitted after applying Dixon's test, see tables.

Repeatabilities,  $r_{95}$ , ranged from 10 to 69 µg/l in the five sample pairs;  $CV_r$  below 10% at all levels. The reproducibility values ( $R_{95}$ ) were considerably higher: 70 to 465 µg/l and  $CV_R$  ranged from 17 to 27%.

The purity of the matrix modifier, potassium dichromate, and its influence on the results, was questioned as it is not possible to obtain this as a high purity reagent. A second collaborative trial was organised, in which the original version of the method was tested, i.e. using magnesium nitrate as modifier and without cool down step before atomization. The trial was performed in the same way as the first one, but the concentration at the highest level was approx 500 µg/litre. Both repeatability and reproducibility values were higher in this study than those obtained in the first one;  $CV_R$  ranged from 30 to 40%.

### CONCLUSIONS

Due to the poor reproducibility values that were obtained in the two collaborative trials, the Analysis Committee of the EBC judged that the method was not suitable for inclusion in Analytica-EBC.

### REFERENCES

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2. Wagner, H. P. and McGarrity, M. J., *J. ASBC*, 1991, 49(4), 151.
3. International Standard, Precision of test methods, ISO 5725, 2nd edition, 1986.