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# Determination of Calcium in Wort by Atomic Absorption Spectrophotometry

Submitted on behalf of the Analysis Committee of the European Brewery Convention

BC 35 Wort

(Descriptors: Collaborative trial.

Deskriptoren: Ringanalyse).

## Introduction

The Analysis Committee of the European Brewery Convention (EBC) decided to test the determination of calcium in wort according to Analytica-EBC method 9.19 Calcium in Beer by Atomic Absorption Spectrophotometry (1).

In spring 2001 an inter-laboratory trial was organised using four samples of wort with different calcium levels in the range of 20 – 150 mg/l. Ten members of the EBC Analysis Committee and one laboratory from Weihenstephan participated in this collaborative trial.

## Experimental

The organisation of the collaborative trial and the statistical treatment of the data were performed according to the procedure given in the International Standard ISO 5725 (2). The trials were undertaken using a uniform design.

Industrial pasteurised worts with different calcium levels were circulated among the participating laboratories which were asked to determine the calcium content in duplicate according to method 9.19 given in Analytica-EBC and report the results to one decimal place.

Cochran's test was used for dealing with stragglers and outliers in the within-laboratory variances; among the cell averages the Grubbs' test was used.

## Results and discussion

The original data are given in table 1.

The Cochran's test classed an outlying standard deviation in laboratory 10 in the case of wort B. Grubbs' test showed two outlying results (wort A and wort B) and one straggling result (wort C) from laboratory 1, so, although the result for wort D from this laboratory does not show a significant deviation, it is considered unreliable and for that reason all results from laboratory 1 have been excluded from the calculations.

The precision data are summarized in table 2. From this table it seems clear that both  $r$  and  $R$  tend to increase with higher values of  $m$ , so a functional relationship between  $r$  and  $m$  as well as between  $R$  and  $m$  exists. The final values for  $r$  and  $R$  are:

$$r = 0.3 + 0.073 m$$

$$R = 4.0 + 0.16 m$$

**Table 1 Original data of the collaborative trial (all results as mg/l calcium)**

Lab	Wort A	Wort B	Wort C	Wort D
1	33.3 / 36.5 <sup>2</sup>	54.1 / 53.9 <sup>2</sup>	75.1 / 72.3 <sup>3</sup>	161.4 / 151.2 <sup>4</sup>
2	17.6 / 17.8	38.1 / 38.2	57.7 / 58.1	144.0 / 144.0
3	21.3 / 20.7	39.4 / 39.3	59.9 / 66.2	150.7 / 158.1
4	22.0 / 22.0	36.5 / 36.0	59.0 / 52.0	128.0 / 140.0
5	18.5 / 18.5	35.6 / 35.0	54.0 / 54.0	147.6 / 147.6
6	15.6 / 15.6	33.9 / 33.9	53.5 / 52.6	138.9 / 138.2
7	19.4 / 18.8	36.2 / 35.7	57.0 / 56.1	148.0 / 150.0
8	18.1 / 16.5	31.7 / 32.4	50.8 / 52.7	141.8 / 138.1
9	16.0 / 18.8	28.0 / 28.0	49.2 / 47.3	133.3 / 137.8
10	24.9 / 24.1	34.3 / 40.7 <sup>1</sup>	57.9 / 57.1	156.1 / 162.5
11	18.3 / 18.5	33.6 / 33.3	56.4 / 55.4	157.0 / 161.0

1. Outlier according to Cochran's test. Not included in calculations.
2. Outlier according to Grubbs' test. Not included in calculations.
3. Straggler according to Grubbs' test. Not included in calculations.
4. Not included in calculations.

**Table 2 Summary of the precision data (calculated from the results of table 1 after the statistical evaluation according to Cochran's and Grubbs' test)**

Sample	Wort A	Wort B	Wort C	Wort D
Number of laboratories	10	9	10	10
Grand mean	19.2	34.7	55.4	146.1
$r_{95}$	2.2	0.8	6.2	10.7
$R_{95}$	7.5	9.5	12.0	27.1
$CVS_r$	4.1	0.8	4.0	2.6
$CVS_R$	14.0	9.8	7.7	6.6

## Conclusion

The results of this collaborative trial show that the repeatability ( $r_{95}$ ) and reproducibility ( $R_{95}$ ) values for the determination of calcium in wort are at an acceptable level, so the EBC Analysis Committee decided to include this method in Analytica-EBC.

## References

1. European Brewery Convention, Analytica-EBC, Grundwerk 1998. Verlag Hans Carl, Nürnberg 1998.
2. International Standard ISO 5725, 1994.