Calcium and Magnesium Determination in Beer

Subcommittee Members: P. G. Koski, *Chairman*; E. G. Austin, A. Bacalla, R. L. Berndt, L. Bernstein, B. K. Blenkinship, R. Brisson, M. Frati, S. Frey, J. Grib, E. H. Heger, W. Herwig, T. Lom, A. Mikalajunas, M. Moll (*EBC*), G. P. Skocic, D. D. Thompson, and D. W. Hysert (*ex officio*).

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CONCLUSIONS

1. The titrimetric procedure for calcium provided a clear, distinguishable end point. Results were scattered but reasonable.
2. Magnesium results were inconclusive because the end point was not clear and was difficult to visualize.

RECOMMENDATIONS

1. Reemphasize that collaborators are to follow the method and suggested notes very carefully.
2. pH measurements to be made should be monitored with a pH meter.
3. A sample with known calcium and magnesium levels will be sent with test samples for familiarization and calibration.
4. The subcommittee should continue its work for another year.

PROCEDURE

All reagents, sample preparations, treatments, procedures, and indicators were followed as they appeared in the paper by Ho and Skocic. (1) Sample A had no additions. Sample B had 10 mg/L each of added calcium and magnesium and sample C had 30 mg/L of each. These additions were made to each bottle of sample by removing the appropriate amount of beer and replacing with the same volume of 1,000 mg/L stock solution. The additions were based on an atomic absorption analysis of the beer samples. Sample A contained 33 mg/L of calcium and 77 mg/L of magnesium by atomic absorption analysis.

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RESULTS AND DISCUSSION

Reviewing data from 16 laboratories (Table I) made clear that results from the magnesium titration were excessively variable, whereas those from the calcium titration were reasonable. Collaborators reported problems in visualizing the titrimetric end point in the magnesium titration. Table I has the data from the 16 laboratories with the means compared to atomic absorption data. Table II has the data from Table I with Youden-Block statistics

TABLE I
Calcium and Magnesium (mg/L) in Beer by Titrimetric Method Compared to Atomic Absorption Method

	Calcium				Magnesium			
Laboratory	A	B	C	D	A	B	C	D
1	33.6	43.5	63.8	74.2	78.2	88.3	103.8	112.1
2	27.0	36.0	53.1	68.1	53.5	53.5	54.1	49.9
3	36.1	48.5	82.8	85.0	73.5	81.6	94.1	100.5
4	32.0	44.0	64.0	76.0	77.0	83.0	99.0	92.0
5	33.0	44.0	62.0	70.0	77.0	83.0	99.0	109.0
6	54.0	39.0	80.0	86.0	81.0	74.0	93.0	103.0
7	36.1	44.1	66.5	75.3	77.8	86.3	100.9	113.1
8	36.8	44.9	67.4	73.5	70.6	84.3	99.1	116.5
9	30.5	40.5	60.1	72.1	66.3	65.2	73.0	77.6
10	36.4	48.1	65.3	73.8	50.8	52.8	56.6	73.0
11	34.6	43.1	61.1	70.6	77.2	86.9	105.4	114.3
12	32.0	43.6	65.7	78.6	38.9	39.9	44.2	41.8
13	52.0	53.0	89.0	108.0	43.0	45.0	43.0	36.0
14	42.0	50.0	76.0	78.0	70.0	78.0	92.0	107.0
15	34.0	44.0	62.0	73.0	73.0	82.0	98.0	105.0
16	35.7	33.5	63.2	74.2	52.8	53.5	35.8	67.2
Mean	36.6	43.7	67.6	77.2	65.4	71.0	80.6	88.6
By atomic absorption	33.0	42.0	63.0	72.0	77.0	91.0	105.0	117.0

TABLE II
Statistical Summary for Calcium and Magnesium in Beer

Sample Pair	No. of Labs	Grand Mean[a]	Laboratory Error			c.v.[d]
			Within[b]	Between[b]	Combined[c]	
AB-calcium	16	40.2	5.01	3.59	6.17	8.95
CD-calcium	16	72.4	3.09	8.97	9.49	12.4
AB-magnesium	16	68.7	3.79	14.8	15.2	21.6
CD-magnesium	16	84.6	7.09	25.2	26.2	29.8

[a]Grand mean = GM = $(\bar{A} + \bar{B})/2$ or $(\bar{C} + \bar{D})/2$.
[b]Calculated per Youden and Steiner (2).
[c]Combined laboratory error (S_c) calculated from within-laboratory error (S_r) and between-laboratory error (S_b); $S_c = \sqrt{S_r^3 + S_b^2}$.
[d]Coefficient of variation of S_c = c.v. = 100 (S_c/GM).

applied to results. Outliers were included in the statistical analysis.

LITERATURE CITED

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