Residual Carbon Dioxide in Beer by a Manometric/Volumetric Method

Subcommittee Members: P. Smith, *Chairman*; G. Austin; M. Cerwinka; B. Cruickshanks; R. Lansbergen; C. Manbeck; J. Miller; G. Nelson; J. Pomeroy; S. Williamson; and R. Crumplen (*ex officio*).

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CONCLUSIONS

1. The method exhibited acceptable repeatability coefficients of variation.
2. The reproducibility coefficients of variation were generally acceptable, although high in relation to the repeatability coefficients of variation.
3. The accuracy of the method was acceptable for sample pairs ranging from 0.30 to 1.60 volumes of carbon dioxide.
4. The method was found to have a detection limit of 0.06 volumes and a determination limit of 0.16 volumes at a precision coefficient of variation of 10%.

RECOMMENDATION

Repeat the collaborative study to determine whether reproducibility can be improved.

This subcommittee was charged with the evaluation of a volumetric/manometric method of determining residual levels of carbon dioxide in beer (3,4). This method is attractive because it is relatively rapid; uses simple, inexpensive, and easily obtainable materials; is easier to perform on-site in the brewery than other existing methods; and uses small sample volumes (approximately 25 ml per test).

PROCEDURE

Collaborators received five sample pairs of beer labeled C/H, B/G, A/F, D/I, and E/J. To provide information on the

TABLE I
Residual Carbon Dioxide in Beer by a Manometric/Volumetric Method (volumes)

	Sample Pair		Sample Pair		Sample Pair		Sample Pair		Sample Pair	
Collaborator	C	H	B	G	A	F	D	I	E	J
1	0.39	0.46	0.60	0.59	0.73	0.72	1.54	1.62	2.37	2.43
2	0.35	0.35	0.49	0.52	0.63	0.63	1.60	1.71	2.63	2.73
3	0.24	0.24	0.35	0.39	0.44[a]	0.85[a]	1.35	1.31	2.32	2.18
4	0.48	0.40	0.59	0.59	0.70	0.70	1.65	1.61	2.68	2.73
5	0.26	0.26	0.43	0.43	0.54	0.57	1.56	1.56	1.92	1.86
6	0.31	0.34	0.48	0.55	0.58	0.61	1.43	1.44	2.32	2.37
7	0.39	0.42	0.59	0.59	0.68	0.72	1.44	1.50	1.81	1.89
8	0.27	0.37	0.57	0.57	0.66	0.67	1.17[a]	1.64[a]	2.40	2.56
Mean[b]	0.336	0.355	0.512	0.529	0.646	0.660	1.510	1.536	2.306	2.344
Grand mean[b]	0.346		0.521		0.653		1.523		2.325	

[a]Outlier at $P \leq 0.01$ based on totals and/or differences (1).
[b]Calculated excluding outliers.

TABLE II
Statistical Summary of Results[a]

Sample Pair	No. of Labs	Grand Mean	Repeatability			Reproducibility		
			s_r	cv_r	r_{95}	s_R	cv_R	R_{95}
C/H	8	0.346	0.038	11.0	0.107	0.079	22.8	0.220
B/G	8	0.521	0.020	3.8	0.055	0.085	16.3	0.237
A/F	7	0.653	0.013	2.1	0.038	0.063	9.6	0.176
D/I	7	1.523	0.042	2.7	0.116	0.120	7.9	0.336
E/J	8	2.325	0.067	2.9	0.187	0.324	14.0	0.908

[a]All calculations were made based on reference 3.

accuracy of the method, carbon dioxide determinations were performed on the sample pairs in the chairman's laboratory using the LAN-1 Laboratory Carbonation Analyzer (5). Sample pairs were prepared in the chairman's laboratory by decarbonating beer to predetermined levels and packaging the treated beer in glass bottles fitted with crowns. The Youden unit block (2) experimental design was used.

The limits of detection and determination (1) were calculated for the method in the chairman's laboratory. To establish these limits, a beer sample was thoroughly decarbonated by method Beer-1A (1) and repeatedly poured between two flasks. This beer was then analyzed by the method a total of 10 times.

RESULTS AND DISCUSSION

The results of eight collaborators are shown in Table I. Outliers at $P < 0.01$ were identified in sample pairs A/F and D/I using Dixon's outlier test (1). Results of the Youden block statistical treatment are listed in Table II.

The method showed acceptable repeatability coefficients of variation for all sample pairs. The reproducibility coefficients of variation ranged from 7.9 to 22.8. In general, reproducibility coefficients of variation decreased as the mean carbon dioxide concentrations increased. This was not true, however, for sample pair E/J, which contained the highest concentration of carbon dioxide. The sampling technique may not adequately prevent losses in samples containing high concentrations of carbon dioxide.

Determinations made on the sample pairs with the LAN-1 Laboratory Carbonation Analyzer showed means of 0.34, 0.55, 0.68, 1.62, and 2.60 volumes for sample pairs C/H, B/G, A/F, D/I, and E/J, respectively. These values compare to the collaborative grand means of 0.346, 0.521, 0.653, 1.523, and 2.325 volumes for the same sample pairs. These data indicate that the accuracy of the method was acceptable for all sample pairs except E/J. Further work with this method may determine whether the reproducibility can be improved on all samples and whether more accurate results can be obtained on samples containing high levels of carbon dioxide.

TABLE III
Limits of Detection and Determination[a]

Run	Results of Replicate Analysis (volumes of carbon dioxide)
1	0.00
2	0.02
3	0.04
4	0.01
5	0.02
6	0.01
7	0.02
8	0.04
9	0.02
10	0.04
Mean	= 0.022
Standard deviation	= 0.014
Limit of detection	= 0.022 + (3) (0.014) = 0.064
Limit of determination	= 0.022 + (10) (0.014) = 0.162

[a]All calculations were made based on reference 1.

Limits of detection and determination for the method are shown in Table III.

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