European Brewery Convention

EBC PRESS REPORT

Determination of CO_2 in Packaged Beer by a Manual Pressure/Temperature (P/T) Method – Determination of the Repeatability and Reproducibility

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on behalf of the Analysis Committee of the European Brewery Convention

The determination of CO_2 in packaged beer by a manual pressure/temperature (P/T) method was collaboratively tested by the Analysis Committee of the European Brewery Convention according to ISO Standard 5725 in order to determine its suitability for publication in Analytica-EBC. Repeatability (r_{95}) and reproducibility (R_{95}) values are presented.

Repeatability (r_{95}) was found to be independent on the mean value and was 0,12 g/l over the range 4,8 to 7,0 g/l. Reproducibility (R_{95}) was found to be dependent on the mean and was 0,089m g/l over the range of CO_2 tested.

The method has acceptable repeatability and reproducibility and is recommended for inclusion in Analytica-EBC.

At the same time an instrumental thermal conductivity method using the Micro Logger 3654 analyzer from Hach Ultra Analytics was also collaboratively tested in order to determine its repeatability (r_{95}) and reproducibility (R_{95}) values.

Repeatability (r_{95}) was found to be independent on the mean value and was 0,08 g/l over the range 4,7 to 6,9 g/l. Reproducibility (R_{95}) was found to be dependent on the mean and was 0,054m g/l over the range of CO_2 tested.

The instrumental thermal conductivity using the Micro Logger 3654 analyzer from Hach Ultra Analytics is also recommended for inclusion in Analytica-EBC.

INTRODUCTION

The Analysis Committee of the European Brewing Convention (EBC) decided to collaboratively test a manual pressure/temperature procedure for the determination of CO_2 in packaged beer.

23 laboratories participated in the test. Additional information was requested about the instrument used, the calculation formula or system and the corrections applied for the air in the head space.

At the same time a method for the determination of CO_2 using thermal conductivity based on one instrument from the company Hach Ultra Analytics, Switzerland (the Micro Logger 3654) was also tested to evaluate its performance.

EXPERIMENTAL

The organization of the collaborative trial and the statistical treatment of the data were performed according to the International Standard ISO 5725-2[1].

In the trial the method was tested with three beers having a range of CO_2 values between 4,8 and 7,0 g/l. Two bottles of each beer sample were sent in duplicate to each laboratory.

RESULTS AND DISCUSSION

Original data from the collaborative trial (but recalculated from original P/T values in the manual determination using the same Haffmans formula in order to homogenize the way the P & T are converted into CO_2 concentration of the beer) is given in Tables I–III. For the manual P/T method four outliers were detected and excluded from the calculations. For the instrumental thermal conductivity method two outliers were detected and also excluded from the calculation.

Precision data are summarized in Tables IV–VI. Repeatability and reproducibility were calculated excluding the outliers and were considered satisfactory for both the manual and the instrumental methods.

Table I. Manual P/T Method. Raw data recalculated with the same Haffmans formula (g/l).

Lab.	A1	A2	B1	B2	C1	C2	Apparatus	Calculation System	Air Head Space Released	S.G. beers
1	5,39	5,43	4,81*	4,92*	7,06	7,08	?	Wallerstein rule	Not	1,01080 1,00709 1,00705
2	5,18	5,33	4,75	4,84	6,58	6,65	Zahm & Nagel Modified Piercing Device	ASBC table	Not	1,01087 1,00702 1,01071
3	5,57	5,57	5,01**	4,85**	7,37	7,37	Zahm & Nagel D.T Piercing Device	Haffmans formula	Yes	? ? ?
4	5,52	5,58	5,02	5,06	7,30	7,23	Haffmans Inpack 2000	Haffmans formula	Yes	1,01083 1,00704 1,01063
5	5,23	5,30	4,71	4,71	7,08	7,08	?	ASBC table	Not	? ? ?
6	5,22	5,26	4,73	4,77	6,85*	6,75*	Zahm & Nagel Series 11000	Haffmans formula	Yes	1,01083 1,00704 1,01063
7	5,42	5,34	4,94	4,97	6,92	6,85	Haffmans Inpack 2000	Haffmans formula	Yes	1,01083 1,00704 1,01063
8	5,82°°	6,15°°	5,33°°	5,33°°	4,76°°	4,76°°	VLSF 100.07.037800	Haffmans formula	Not	? ? ?
9	4,95°	5,11°	4,52°	4,54°	6,33	6,37	Terris/WQS	Haffmans rule	Yes	1,01083 1,00704 1,01063
10	5,59	5,59	4,97	4,95	7,13	7,14	Steinfurth 91.7.3V	Haffmans formula	Not	1,01083 1,00704 1,01063
11	5,31	5,30	4,82	4,81	7,02	7,04	Haffmans Inpack 2000	Haffmans formula	Not	1,01083 1,00704 1,01063
12	5,72	5,79	4,98	5,02	7,34	7,29	Haffmans CBL	Haffmans formula	Not	1,01055 1,00682 1,01047
13	5,50	5,42	4,99	4,95	7,23	7,17	?	Slide rule	Not	1,01088 1,00709 1,01063
14	5,35	5,35	4,84	4,81	6,91	6,91	Haffmans Inpack 2000	Haffmans formula	Not	1,01083 1,00704 1,01063
15	5,41	5,39	4,92	4,92	7,04	7,04	Haffmans Halu	Slide rule	Not	1,01085 1,00711 1,01074
16	5,35	5,35	4,73	4,81	6,91	6,98	Piercing device + digital pressure meter	Haffmans formula	Yes	? ? ?
17	5,26**	5,60**	5,04	5,02	7,20	7,19	Haffmans Inpack 2000	Haffmans formula	Not	? ? ?
18	5,48	5,43	4,89	4,94	7,06	7,04	Steinfurth	Haffmans rule	Not	1,01083 1,00704 1,01063
19	5,49	5,45	4,93	4,92	7,14	7,05	Haffmans ICM-045	Haffmans formula	Not	1,01083 1,00704 1,01063
20	5,28	5,38	4,86	4,84	6,75	leaking sample	Stadler-Zeller	Haffmans formula	Not	1,0108 1,0072 1,0108
21	5,33	5,29	4,79	4,77	6,94	6,98	Haffmans Inpack CO_2 meter	Haffmans formula	Not	1,01083 1,00704 1,01063
22	5,33	5,34	4,76	4,79	7,01	6,94	Haffmans Inpack CO_2 meter	Haffmans formula	Not	1,01083 1,00704 1,01063
23	5,39	5,38	4,79	4,79	7,05	7,05	Haffmans Inpack CO_2 meter	Haffmans formula	Not	1,01083 1,00704 1,01063
24	5,12	5,14	4,82	4,79	6,75	6,75	Haffmans Inpack 200	Slide rule	Not	1,01083 1,00704 1,01063

** within laboratory outliers, rejected
* within laboratory stragglers, retained
°° between laboratory outliers, rejected
° between laboratory stragglers, retained

Table II. Raw data (g/Kg) Micro Logger 3654.

Lab.	A1	A2	B1	B2	C1	C2
1	5,17	5,13	4,54°	4,57°	6,73	6,67
2	5,40°	5,47°	4,91**	4,79**	7,06	7,06
3	5,34	5,35	4,74	4,74	6,97*	6,88*
4	5,32	5,29	4,74	4,76	6,92	6,93
5	5,19	5,22	4,62	4,68	6,68	6,69
6	5,23**	5,11**	4,60	4,61	6,74	6,67
7	5,25	5,26	4,72	4,71	6,86	6,88
8	5,29	5,26	4,68	4,70	6,95	6,98
9	5,24	5,25	4,67	4,69	6,87	6,90
10	5,26	5,25	4,66	4,66	6,89	6,94

** within laboratory outliers, rejected
* within laboratory stragglers, retained
°° between laboratory outliers, rejected
° between laboratory stragglers, retained

Table III. Raw data for the Haffmans instruments alone (g/l) recalculated with the same Haffmans formula.

Lab.	A1	A2	B1	B2	C1	C2
1	5,52	5,58	5,02	5,06	7,30	7,23
2	5,42*	5,34*	4,94	4,97	6,92	6,85
3	5,31	5,30	4,82	4,81	7,02	7,04
4	5,72°°	5,79°°	4,98	5,02	7,34°	7,29°
5	5,35	5,35	4,84	4,81	6,91	6,91
6	5,41	5,39	4,92	4,92	7,04	7,04
7	5,35	5,35	4,73**	4,81**	6,91	6,98
8	5,49	5,45	4,93	4,92	7,14	7,05
9	5,33	5,29	4,79	4,77	6,94	6,98
10	5,33	5,34	4,76	4,79	7,01	6,94
11	5,39	5,38	4,79	4,79	7,05	7,05
12	5,12	5,14	4,82	4,79	6,75	6,75

** within laboratory outliers, rejected
* within laboratory stragglers, retained
°° between laboratory outlier, rejected
° between laboratory stragglers, retained

Table IV. CO_2 P/T calculated using the same Haffmans formula.

	A	B	C
n	22	22	22
m	5,38	4,85	7,00
$\mathbf{r}_{95}$	0,14	0,11	0,10
$\mathbf{R}_{95}$	0,47	0,35	0,69
$\mathbf{CVS}_r$	0,86	0,79	0,50
$\mathbf{CVS}_R$	2,99	2,47	3,33

r_{95} = 0,12
R_{95}= 0,089m

Table V. Instrumental thermal conductivity – Hach Ultra Analytics.

	A	B	C
n	9	9	10
m (gr/Kg)	5,27	4,67	6,86
$\mathbf{r}_{95}$	0,075	0,059	0,105
$\mathbf{R}_{95}$	0,27	0,21	0,41
$\mathbf{CVS}_r$	0,44	0,39	0,48
$\mathbf{CVS}_R$	1,58	1,38	1,87
m (gr/l)	5,33	4,70	6,93

r_{95} = 0,08
R_{95} = 0,054m

Table VI. Haffmans instruments with CO_2 calculated using the same Haffmans formula.

	A	B	C
n	11	11	12
m	5,36	4,88	7,02
$\mathbf{r}_{95}$	0,080	0,058	0,114
$\mathbf{R}_{95}$	0,335	0,303	0,493
$\mathbf{CVS}_r$	0,48	0,38	0,52
$\mathbf{CVS}_R$	1,99	1,97	2,26

r_{95} = 0,08
R_{95} = 0,066m

CONCLUSION

Repeatability and reproducibility results for the determination of the CO_2 in packaged beer by a manual P/T method or by an instrumental method based on thermal conductivity using a Micro Logger 3654 from Hach Ultra Analytics (Switzerland) are acceptable. The EBC Analysis Committee has recommended the inclusion of both methods in Analytica-EBC.

BIBLIOGRAPHY

1. International Standard ISO 5725-2, 1994.