

Evaluation of Established Methods of Decarbonating Beer¹

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ABSTRACT

Various methods of decarbonating beer were evaluated to identify the best method of preparing a beer sample for chemical analysis, particularly for the determination of alcohol and real extract. Beer samples were decarbonated by ultrasonic bath, filtration, a combination of ultrasonic bath and filtration, pouring (the repeated transfer of beer from one beaker to another), and ASBC method Beer-1A. The samples then were analyzed by a Servo Chem Automated Beer Analyzer for alcohol and real extract and by a LAN-1 carbonation analyzer for carbon dioxide. Samples prepared by ASBC method Beer-1A and the pouring method showed the lowest levels of carbon dioxide and real extract. An alcohol loss of from 0.05 to 0.07% (w/w) was observed in samples prepared by the pouring method; samples prepared by all other methods showed alcohol values within 0.02% (w/w), with samples prepared by ultrasonic bath tending to show the highest alcohol values. In addition, samples prepared by ultrasonic bath, filtration, and a combination of ultrasonic bath and filtration all showed approximately the same level of carbon dioxide and real extract. The error introduced by residual carbon dioxide in a sample was calculated to be approximately 0.0125% (w/w) real extract for each 0.1 volume of carbon dioxide.

Keywords: Alcohol, Carbon dioxide, Decarbonation, LAN-1 carbonation analyzer, Real extract, SCABA

Most analytical methods for the evaluation of beer require that carbon dioxide be removed from a sample before analysis. Among the methods requiring decarbonated beer are those used for the determination of alcohol and real extract (1). Several in-house investigations have shown that the technique used to decarbonate beer can significantly affect the alcohol and real extract concentrations of samples. The effect of sample decarbonation on alcohol content has likely been observed by others, since the American Society of Brewing Chemists subcommittee charged with coordination of new and alternate methods of analysis has deemed the current decarbonation method in use by the Society inadequate for accurate alcohol measurement and has requested an investigation of decarbonation methods (3). In addition, Patino et al, in their work on the influence of carbonation on beer density (6), concluded that optimal degassing techniques should be developed to achieve a consistent carbon dioxide level without sample evaporation. This article presents the results of an evaluation of several decarbonation methods currently in use for their impact on a beer sample's alcohol and real extract concentrations.

EXPERIMENTAL

Methods

Beer and diluent water samples were prepared for analysis according to five different decarbonation methods. All beer samples used in this study were packaged in cans or bottles and contained approximately 2.60 volumes of carbon dioxide. The diluent water used in this study was acidified and contained approximately 2.30 volumes of carbon dioxide. In each method, the samples were adjusted to 20°C before treatment and allowed to reach ambient temperatures (20–23°C) during decarbonation. The decarbonation methods were as follows.

Ultrasonic bath. Two hundred milliliters of beer was poured into a 500-ml Erlenmeyer flask. The flask then was covered with aluminum foil and placed in an ultrasonic bath containing 2.5 cm of water. The samples were treated in the ultrasonic bath for 15 min.

Ultrasonic bath and filtration. Samples were prepared as above but were treated in the ultrasonic bath for only 10 min. The beer was then poured into a funnel lined with 24-cm, grade 513 fluted filter paper and collected in a 250-ml Erlenmeyer flask. After the beer was added to the funnel, the funnel was covered tightly with aluminum foil to minimize evaporation.

Filtration. Two hundred milliliters of beer was poured into a funnel lined with 24-cm, grade 513 fluted filter paper and collected in a 250-ml Erlenmeyer flask. After the beer was added to the funnel, the funnel was covered tightly with aluminum foil to minimize evaporation.

Pouring. Samples were decarbonated by repeated transfers between two 1,000-ml beakers; 200 ml of beer was placed into one of the beakers and then repeatedly poured from one beaker to the other for a total of 60 pours.

ASBC method Beer-1A. Two hundred milliliters of beer was placed in a 500-ml Erlenmeyer flask and decarbonated by shaking, gently at first and then vigorously, until gases no longer escaped from the beer.

Any foam that remained on the beer samples after decarbonation was allowed to collapse before the samples were analyzed for alcohol, real extract, and carbon dioxide.

Equipment

Alcohol and real extract values were obtained in this study with a Servo Chem Automatic Beer Analyzer (SCABA) (2,4) equipped with densitometer, pH meter, and an automatic sampling tray. The SCABA measures alcohol by evaporating and then oxidizing the alcohol in a sample. The heat created by oxidation is transformed into a voltage reading, which, based on a calibration, is used to determine a sample's alcohol concentration. The SCABA also measures specific gravity by passing the sample through the unit's densitometer. Using the alcohol and specific gravity measurements, the SCABA then calculates related values such as real extract, apparent extract, and original gravity.

Carbon dioxide determinations were made with a LAN-1 laboratory carbonation analyzer (7), equipped with a sample holder and a source of pressurized air. This analyzer directly measures the intensity of the infrared carbon dioxide absorption band in a sample, using infrared analysis. Before use, the analyzer was zeroed with deionized water and calibrated to match the ASBC carbon dioxide chart. To accomplish this calibration, 24 bottles of beer from the same production run were obtained. Twelve bottles were subjected to temperature-pressure determinations of carbon dioxide (1), and 12 bottles were analyzed with the LAN-1 laboratory carbonation analyzer. Using the instrument's calibration dial, the average response of the LAN-1 laboratory carbonation analyzer was made to match the average carbon dioxide value obtained via the ASBC temperature-pressure method.

The ultrasonic bath used in this study was equipped with an ultrasonic generator producing 1,000W average output at simultaneous multiple frequencies (40–90 kHz). The tank portion of the ultrasonic bath measured 40.64 cm wide, 55.88 cm long, and 50.8 cm high. A sample tray was placed in the bottom of the tank that maintained approximately 1.25 cm clearance between samples and the bottom of the tank. Approximately 2.5 cm of water was placed in the bath.

RESULTS AND DISCUSSION

Nineteen replicate samples of a premium beer were analyzed for alcohol, real extract, and carbon dioxide after decarbonation

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by the various methods. The means, standard deviations, and ranges found for these samples for each decarbonation method are listed in Table I, and the means are shown graphically in Figure 1. Five replicate samples of diluent water also were prepared by each decarbonation method, and determinations were made for specific gravity, real extract, and carbon dioxide. Table II shows the means, standard deviations, and ranges found for these samples.

The data in Table I show several differences among the decarbonation methods. First, the data show that the lowest concentrations of carbon dioxide were found in samples prepared by ASBC method Beer-1A (0.04 volumes) and pouring (0.06 volumes), whereas samples prepared by ultrasonic bath, filtration, and ultrasonic bath and filtration showed significantly higher concentrations of carbon dioxide (0.56, 0.53, and 0.41 volumes, respectively). Second, the data show that the pouring method yielded samples that were from 0.06 to 0.07% (w/w) lower in alcohol than samples prepared by the other methods. Alcohol concentrations were very close (within 0.02% w/w) for samples prepared by ultrasonic bath, filtration, ultrasonic bath and filtration, and ASBC method Beer-1A. However, even among these four methods, a consistent pattern was observed, as samples prepared by the ultrasonic bath consistently showed the highest alcohol values, and samples prepared by ASBC method Beer-1A consistently showed alcohol values of 0.01–0.02% (w/w) lower

than those prepared by the ultrasonic bath. Although within the published error for the SCABA of 0.02% (w/w) (5), the reproducibility nature of this difference suggests that it should not be overlooked. And third, the data show that a direct relationship exists between the concentrations of carbon dioxide and real extract, so that samples prepared by ASBC method Beer-1A and pouring showed real extract values from 0.04 to 0.05% (w/w) lower than samples prepared by the other methods in this study. Although data on premium beer are shown in Table I, similar results were obtained with reduced-calorie and super-premium beers.

The direct relationship between carbon dioxide and real extract is illustrated in Figure 2, a plot of a beer sample analyzed at frequent intervals during decarbonation by pouring. This figure shows that as carbon dioxide was removed from the sample, the real extract concentration decreased. The decrease in real extract is a result of the reduction in the specific gravity of the beer as carbon dioxide is removed. It is clear from Figure 2 that when carbon dioxide was present in the beer, even at residual concentrations, the sample's real extract value was elevated. Therefore, decarbonation methods evaluated in this study that resulted in the most complete removal of carbon dioxide also resulted in the most accurate determinations for real extract.

A calculation of the error introduced to real extract values by incomplete decarbonation can be made from the data of this

TABLE I
Effect of Decarbonation Methods on Alcohol, Real Extract, and Carbon Dioxide Analysis of 19 Replicate Beer Samples

Decarbonation Method	Alcohol (% w/w)			Real Extract (% w/w)			CO ₂ (volumes)		
	Mean	Range	SD	Mean	Range	SD	Mean	Range	SD
Ultrasonic bath	3.624	3.61–3.64	0.009	3.561	3.53–3.59	0.018	0.560	0.49–0.66	0.065
Filtration	3.617	3.59–3.63	0.014	3.559	3.52–3.58	0.015	0.529	0.46–0.60	0.041
Ultrasonic bath and filtration	3.617	3.59–3.63	0.015	3.549	3.52–3.57	0.015	0.410	0.31–0.51	0.058
ASBC method Beer-1A	3.610	3.58–3.63	0.018	3.510	3.49–3.53	0.012	0.039	0.00–0.10	0.031
Pouring	3.554	3.52–3.59	0.022	3.512	3.49–3.53	0.011	0.060	0.00–0.13	0.038

TABLE II
Effect of Decarbonation Methods on Real Extract, and Carbon Dioxide Analysis of Five Replicate Diluent Water Samples

Decarbonation Method	Specific Gravity			Real Extract (% w/w)			CO ₂ (volumes)		
	Mean	Range	SD	Mean	Range	SD	Mean	Range	SD
Ultrasonic bath	1.00044	1.00042–1.00047	0.00002	0.114	0.11–0.12	0.005	0.430	0.40–0.46	0.028
Filtration	1.00046	1.00043–1.00050	0.00003	0.118	0.11–0.13	0.008	0.496	0.43–0.58	0.055
Ultrasonic bath and filtration	1.00045	1.00041–1.00047	0.00003	0.114	0.10–0.12	0.009	0.402	0.36–0.43	0.028
ASBC method Beer-1A	1.00026	1.00025–1.00027	0.00002	0.062	0.06–0.07	0.004	0.000	0.00–0.00	0.000
Pouring	1.00025	1.00025–1.00026	0.00002	0.060	0.06–0.06	0.000	0.000	0.00–0.00	0.000

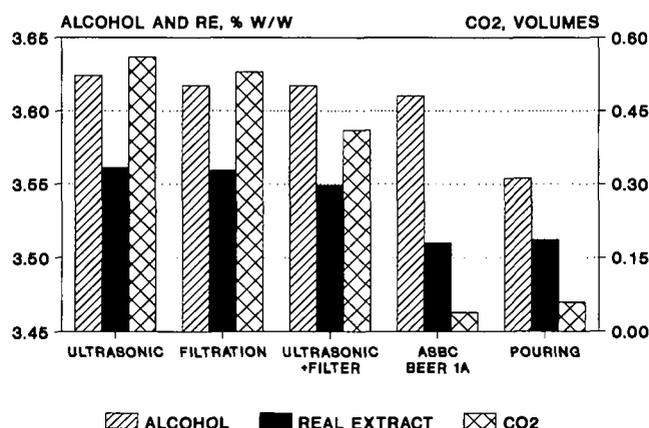


Fig. 1. Alcohol, real extract (RE), and carbon dioxide concentrations in a premium beer sample after decarbonation by various methods.

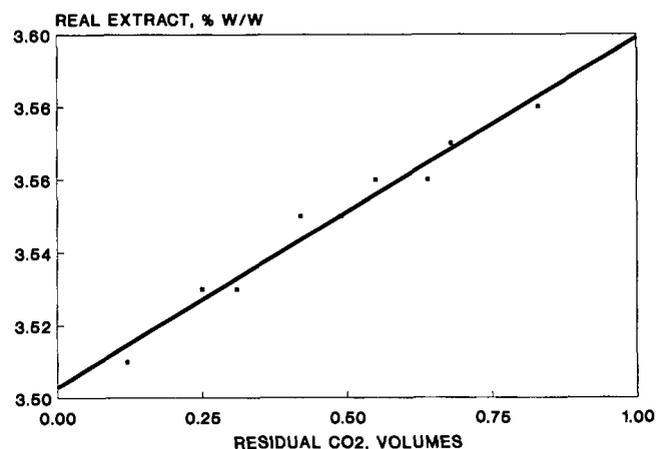


Fig. 2. Relationship between carbon dioxide and real extract in a beer sample undergoing decarbonation.

study. A review of the diluent water data of Table II shows that samples prepared by both ASBC method Beer-1A and pouring contained 0.00 volumes of carbon dioxide and 0.06% (w/w) real extract (dissolved solids in the water are responsible for this real extract concentration), whereas samples prepared by the other methods contained from 0.40 to 0.50 volumes of carbon dioxide and from 0.11 to 0.12% (w/w) real extract. The difference in real extract between the samples can therefore be attributed to residual carbon dioxide. An example using the results of the samples prepared by pouring and ultrasonic bath and filtration from Table II gives the equation:

$$0.11\% \text{ (w/w) R1} - 0.06\% \text{ (w/w) R2} = 0.05\% \text{ (w/w) R3}$$

$$0.40 \text{ volumes (V)} = 0.05\% \text{ (w/w) R3}$$

$$0.10 \text{ volumes} = 0.0125\% \text{ (w/w)}$$

where R1 = real extract of decarbonated sample containing residual carbon dioxide, R2 = real extract of sample with carbon dioxide completely removed, R3 = portion of sample's real extract due to carbon dioxide, and V = volumes of carbon dioxide measured in sample.

The mean of 28 diluent water samples yielded an error value of 0.0123% (w/w) for each 0.1 volume of residual carbon dioxide in a sample. Similarly, when the differences in real extract and carbon dioxide were compared in 20 beer samples, a mean error value of 0.0127% (w/w) for each 0.1 volume of residual carbon dioxide was obtained. This error value is similar to the theoretical

value of 0.0172% (w/w) for each 0.1 volume calculated from density changes at various carbon dioxide levels as reported by Patino et al (6). If an average calculated error value of 0.0125% (w/w) is applied to the average residual carbon dioxide concentrations listed for the various methods in Table I, samples prepared by ultrasonic bath, filtration, ultrasonic bath and filtration, ASBC method Beer-1A, and pouring would have average errors of 0.070, 0.066, 0.051, 0.005, and 0.008% (w/w) real extract, respectively.

Because it was clear that the presence of residual carbon dioxide could lead to elevated real extract values, efforts were made to modify the decarbonation methods that resulted in samples with higher carbon dioxide levels. Figure 3 shows the results of a trial to modify the ultrasonic bath method for more complete decarbonation. A sample was treated for 60 min in the ultrasonic bath and analyzed at frequent intervals for alcohol, real extract, and carbon dioxide. The carbon dioxide concentration decreased slowly from 0.66 volumes after 15 min to 0.48, 0.42, 0.37, and 0.28 volumes after 20, 25, 30, and 40 min, respectively. After 40 min, no further reduction in carbon dioxide concentration was observed. With respect to alcohol, it was found that a sample could be treated (with the temperature controlled at approximately 20°C and the sample flask covered) as long as 50 min without significantly reducing the alcohol concentration. This trial indicated that increasing the ultrasonic sample treatment times could result in lower carbon dioxide concentrations and, therefore, lower real extract values. However, even with extended treatment times, the ultrasonic bath was unable to completely decarbonate the samples. Moreover, if extended treatment times were used, this method would have the further disadvantage of decreased sample preparation efficiency.

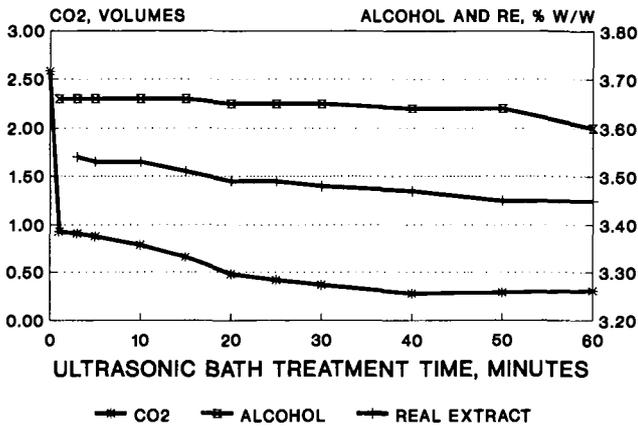


Fig. 3. Influence of extended sample treatment time in ultrasonic bath on concentrations of alcohol, real extract (RE), and carbon dioxide in a beer sample.

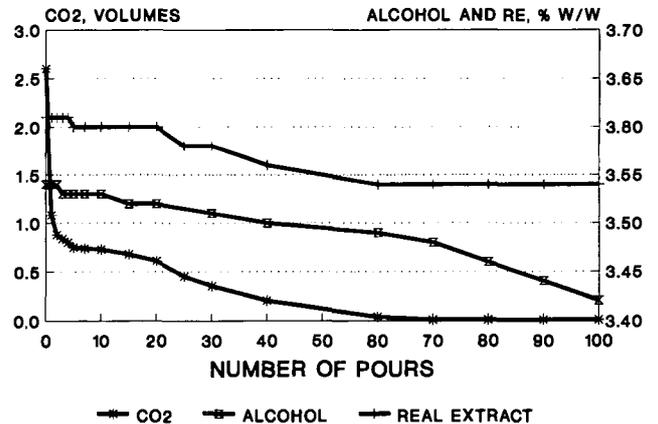


Fig. 5. Plot of alcohol, real extract (RE), and carbon dioxide concentrations of a beer sample undergoing decarbonation by pouring.

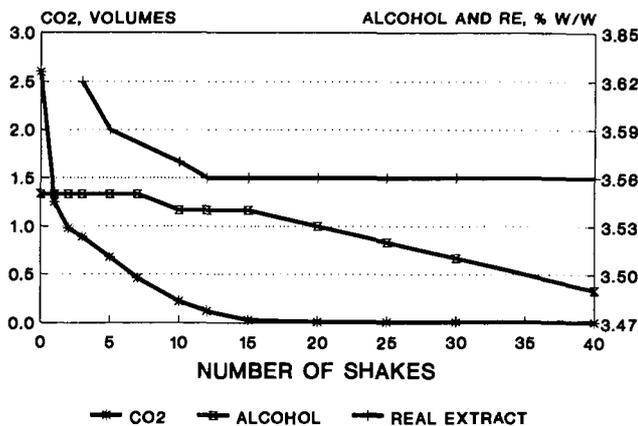


Fig. 4. Plot of alcohol, real extract (RE), and carbon dioxide concentrations of a beer sample undergoing decarbonation by ASBC method Beer 1A.

TABLE III
Time Required to Prepare Samples by Decarbonation Methods

Decarbonation Method	Analyst Time (min)	Total Time (min)
Per one sample		
Ultrasonic bath	1	20
Filtration	2	15
Ultrasonic bath and filtration	3	20
ASBC method Beer-1A	4	25
Pouring	3	3
Per 10 samples		
Ultrasonic bath	4	22
Filtration	5	20
Ultrasonic bath and filtration	7	25
ASBC method Beer-1A	25	25
Pouring	25	25

To further reduce the carbon dioxide concentration of samples prepared by filtration, a trial was conducted to filter samples multiple times. The carbon dioxide concentration of a sample that showed 0.53 volumes after one filtration was reduced to 0.45, 0.28, 0.23, and 0.10 volumes after receiving two, three, four, and five filtrations, respectively, without reducing the alcohol concentration of the sample. Again, increasing sample treatment produced lower carbon dioxide levels, with the trade-off of longer sample preparation times.

No further efforts were required to reduce the carbon dioxide levels of samples prepared by ASBC method Beer-1A, as this method proved very efficient at removing carbon dioxide from beer samples. However, efforts were required to reduce the loss of alcohol in samples prepared by ASBC method Beer-1A. Figure 4 shows a plot of the alcohol, real extract, and carbon dioxide concentrations of a beer sample during decarbonation by ASBC method Beer-1A. Figure 4 shows that 0.01% (w/w) alcohol was lost before the sample was completely decarbonated, and when the sample was treated past the point of complete decarbonation, its alcohol concentration was further reduced. Thus, to obtain the most accurate alcohol values for samples prepared by ASBC method Beer-1A, it was necessary to precisely determine the point when, as written in the method, "gases no longer escape from the beer," thus ensuring that the sample was not over-treated. In this study, it was determined that 15 shake cycles were required to completely decarbonate a sample. (A shake cycle was defined as a 10-sec period in which the beer was shaken and swirled as vigorously as possible without overfoaming the flask.) This process required approximately 25 min per sample, including rest periods between shake cycles.

The pouring method resulted in very low levels of carbon dioxide and lower alcohol levels than the other methods. Attempts to limit the alcohol loss seen in samples prepared by this method were not successful. Figure 5 shows a plot of the alcohol, real extract, and carbon dioxide concentrations of a beer sample as it was decarbonated by pouring.

Table III shows the amount of time required in this study to prepare samples using each method. Time is expressed both as the "hands-on time" required by an analyst and as the total time (including analyst time) required to perform each of the methods. The table also lists time values per one and per 10 samples. It is evident from the table that the ultrasonic bath method was the most efficient in terms of analyst time, whereas the pouring method required the least amount of total time to prepare one sample. When evaluated for the total time required to prepare 10 samples, filtration and ultrasonic bath, which are the two least labor-intensive methods, showed a time savings.

CONCLUSIONS

Beer samples prepared by the decarbonation methods evaluated in this study showed differences of up to 0.05% (w/w) real extract

and up to 0.07% (w/w) alcohol. Also, the presence of residual carbon dioxide in samples was shown to contribute an error of approximately 0.0125% (w/w) real extract for each 0.1 volume of carbon dioxide. In addition, this study showed that methods can be modified to improve results. However, when evaluated for accuracy of alcohol and real extract values and ease of sample preparation, none of the methods discussed in this study is perfect. When deciding on which decarbonation method to use, each laboratory must decide what errors it can tolerate and what characteristics of a method are most important. Efforts should continue to develop a better decarbonation method. Ideally, such an improved decarbonation method will be widely and uniformly practiced by the Society; will result in complete removal of carbon dioxide, no loss of alcohol, and no change in other attributes such as bitterness units, color, or protein; and will allow rapid and perhaps automated sample preparation.

ACKNOWLEDGMENTS

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