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Alcohol by Rapid Distillation

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Overview:

Determining alcohol content accurately and precisely is essential in the brewing industry for a variety of reasons: to conform to government laws ensuring that proper taxes are paid, to confirm that the product meets label specifications, to meet quality control parameters, and to maintain consistency in products. While the NIR/density method (Beer-4G) is quite suitable for measuring the alcohol content of beer, flavored malt beverages present a challenge. Interference with the instrument measurement can occur as a result of flavor additions and/or high alcohol content. A more accurate way to measure alcohol in this type of product is by using standard distillation (Beer-4). Unfortunately, this method is time consuming and labor intensive. Due to these drawbacks, an alternative method was investigated. A rapid distillation unit, which distills using the power of steam, was tested.

Experimental Methods:

Sample Preparation & Analysis: Samples were degassed via either filtration through fluted filter paper or by sonication. (Note that if degassing by filtration exceeded five minutes, degassing by sonication was performed as an alternative to filtration to prevent the loss of ethanol.) Samples remained in a sealed Nalgene bottle and were placed in a 20°C water bath for a minimum of 25 minutes in order to attemporate prior to pipetting. Five drops of a diluted anti-foam solution (1:10) were added to the bottom of the 300 ml digestion tube. After the anti-foam was added, 100 ml of the degassed sample was pipetted with a volumetric pipette and transferred into the digestion tube. The digestion tube was swirled gently to mix the sample with the anti-foam. The steam inlet tube of the instrument was then inserted into the digestion tube, the protection door was closed, and the automated distillation was started. The distillation time applied was 4 minutes and 10 seconds. The steam power applied was 70%. Distillate samples were collected in 100 ml volumetric flasks and placed in a 20°C water bath. After samples were brought to temperature, they were diluted to volume with Milli-Q grade (18 MΩ) water and analyzed for ABV based on specific gravity, similar to the Beer-4A method.

Image 1: View of the Rapid Distillation Apparatus

Precision Study: The instrument was first evaluated for precision by three analysts. Five different brands including both beer and flavored malt beverages were analyzed. Package dates were the same within each brand to eliminate that as a variable. The alcohol content of these brands ranged from 4.2%-14.4%, v/v. Each analyst tested six replicates of one brand within the same day to determine repeatability. This was done on 4 separate days over a 2 week time period to determine reproducibility.

Accuracy Study: Data were generated based on twenty-seven different brands analyzed by both standard distillation and rapid distillation. Samples were chosen in attempt to encompass a large range of alcohol levels. The alcohol content of these brands ranged from 3.64%- 14.4 % v/v.

Results: **Table 1** shows the standard deviation and the %CV for each analyst for each of the five samples.

Table 1: Precision Study-Investigation of Repeatability

Sample A- Label Claim 4.2%		
Analyst:	Standard Deviation:	%CV:
1	0.005	0.113
2	0.004	0.095
3	0.003	0.082
Sample B- Label Claim 5%		
Analyst:	Standard Deviation:	%CV:
1	0.005	0.104
2	0.011	0.227
3	0.006	0.115
Sample C- Label Claim 5.5%		
Analyst:	Standard Deviation:	%CV:
1	0.004	0.078
2	0.004	0.068
3	0.008	0.139
Sample D- Label Claim 8%		
Analyst:	Standard Deviation:	%CV:
1	0.016	0.199
2	0.011	0.131
3	0.131	0.131
Sample E- Label Claim 14.4%		
Analyst:	Standard Deviation:	%CV:
1	0.008	0.054
2	0.030	0.209
3	0.019	0.129

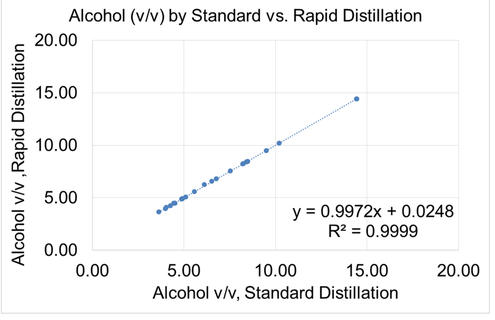
For the second half of the precision study, reproducibility was investigated. For each brand, the results from all analysts were combined. The standard deviation, the %CV and the confidence intervals are shown in **Table 2** below.

Table 2: Precision Study-Investigation of Reproducibility

Sample:	Standard Deviation:	%CV:	95% CI	99% CI
A	0.006	0.145	0.003	0.004
B	0.008	0.155	0.004	0.005
C	0.006	0.112	0.003	0.004
D	0.015	0.193	0.008	0.011
E	0.020	0.137	0.010	0.014

For the accuracy study, 27 brands were analyzed by both standard distillation and rapid distillation for alcohol levels ranging from 3.6-14.4% v/v. Results were plotted on **Graph 1** below to compare the two sets of data. Results were also assessed using a paired t-test. The resulting p-value =0.30.

Graph 1: Accuracy Study- Standard vs. Rapid Distillation



Conclusions:

Repeatability and reproducibility coefficients of variation for the determination of alcohol were all well below 1% (0.05-0.23% and 0.11-0.19%, respectively for alcohol levels ranging from 4.2-14.4%, v/v). The p-value of 0.30 that was obtained from the accuracy study, showed that there was no statistically significant difference between the means of the data sets at a 95% confidence level. In addition, according to **Graph 1** above, the slope of 0.9972 showed the two methods produced nearly identical results. The coefficient of determination =0.9999, denoted a strong linear association between the two sets of data.

References:

1.) American Society of Brewing Chemists. *Methods of Analysis*, Beer-4A Beer and Distillate Measure Volumetrically, Beer 4G Near-Infrared and Original Extract Content, ASBC, St. Paul, MN, 2004.

2.) Gerhardt Analytical Systems. *Operating Instructions*, Vapodest 200. Konigswinter, Germany, 2014.