Efficient Analysis of Alcohol Concentration in RTD (Ready-To-Drink) Alcoholic Beverages Using Near-Infrared System - BCOJ Collaborative Work -

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ABSTRACT

RTD is one of the categories of alcoholic beverages in Japan. It stands for "Ready to Drink" and refers to beverages that can be consumed straight out of the bottle. Japan National Tax Agency (NTA) establishes official analytical methods for alcohol in RTD. Alcoholic drink makers must use these methods to record alcohol concentration for liquor tax management. Currently the official methods include the distillation method, gas chromatography method and oxidation method. But these methods are not efficient because they are laborious and time-consuming. Alcolyzer (Anton Paar GmbH) uses near-infrared principle, and takes a few minutes per sample to measure alcohol concentration in alcohol drinks. However, the Alcolyzer had not yet been approved for use by NTA for determining and recording alcohol concentrations. Here we evaluated the Alcolyzer as a candidate alternative method of alcohol analysis for RTD beverages.

Liquors that were permitted to use Alcolyzer by Japanese Government (Aug. 2021)

Samples	Items
Beer	Alcohol, Extract
Sake, Pitching yeast for beer, Beer during fermentation	Alcohol

Under the Japanese taxation system, RTD beverages are categorized into two types: beverages containing extracts at <2 g/100 ml and those containing extracts at ≥2 g/100 ml. The former are considered spirits and the latter are considered liqueurs. Spirits have a low concentration of extracts, so the extracts do not interfere with the measurements by the Alcolyzer even when the color of the spirit is dark. In contrast, liqueurs contain more extracts, and there are many kinds of liqueurs, e.g., some have a very high extract content, some are highly viscous, and some contain fruits. As such, after some evaluation tests, we determined the upper limits of the alcohol and extract concentrations for the Alcolyzer. According to the Alcolyzer system specifications, the upper alcohol limit is 20% for spirits. We found that extract concentrations higher than 13% interfere with the alcohol analysis by the Alcolyzer, and we determined the upper alcohol concentration limit to be 10%, and the upper extract concentration limit to be 13% for liqueurs.
To examine the repeatability, we obtained data from the distillation method, one of the official methods, for comparison. To examine the reproducibility, three spirit samples and three liqueur samples were analyzed at 10 different laboratories. We reported these results to the regional taxation bureau in Japan, and the Alcolyzer method was approved as an official method for analyzing RTD beverages.

PROCEDURES

1. Selection of the Alcolyzer analysis mode and determination of the sample range
The Alcolyzer ME has three analysis modes, beer, wine and spirit modes. The spirit mode is for whisky, brandy and such kind of high alcohol products. We compared the modes beer and wine. We also examined how high alcohol and extract the Alcolyzer can analyze. The sample range was roughly checked using market products, and was precisely determined using the samples made in the lab. The sample temperature is determined 15℃ by liquor tax law in Japan.

2. Validation of Alcolyzer measurements
(1) Repeatability and trueness
For the repeatability test, five spirit samples and five liqueur samples were prepared, and each sample was analyzed five times using the Alcolyzer. For the trueness test, the average value of each sample was compared to that obtained by the official distillation method.
(2) Reproducibility
For the reproducibility test, three spirit samples and three liqueur samples were analyzed using the Alcolyzer at 10 different laboratories.

RESULTS AND DISCUSSION

1. Selection of the Alcolyzer analysis mode and determination of the sample range
Compared to the values obtained in the beer analysis mode, the values obtained in the wine analysis mode were more similar to those obtained by the distillation method (Table 1). The difference between values from the distillation method and the Alcolyzer method increased with increasing alcohol or extract concentration. In the wine analysis mode, the Alcolyzer was able to analyze alcohol up to 20% v/v for the spirit samples, because the extract concentration of spirits was <2%. However, for the liqueur samples, the Alcolyzer could only analyze alcohol up to about 10% v/v when the samples contained a high concentration of extracts.
The alcohol concentration difference between the distillation method and the Alcolyzer was 0.10% v/v (Table 2) and acceptable when the extract was 15.29 % w/v, but the alcohol concentration difference between the distillation method and the Alcolyzer was 0.14% v/v and unacceptable when the alcohol was 11.23% v/v.

Table 1. Comparison of Alcolyzer wine and beer mode

	Alcohol(% v/v)					Extract(% w/v)
	Distillation	Alcolyzer Wine	Alcolyzer Beer@15℃	-	-	Distillation
	A	B	C	B-A	C-A	
Spirits 1	14.68	14.62	14.60	-0.06	-0.08	0.75
Spirits 2	18.43	18.33	18.25	-0.10	-0.18	0.73
Liqueur 1	4.79	4.83	4.70	0.04	-0.09	10.87
Liqueur 2	8.86	8.79	8.75	-0.07	-0.11	9.57
Liqueur 3	14.60	14.37	14.39	-0.23	-0.21	9.05
Liqueur 4	19.54	19.14	19.15	-0.40	-0.39	8.50

Table 2. Test results to confirm the highest alcohol and extract limits by Alcolyzer

Items	Alcohol(% v/v)			Extract(% w/v)
Analysis modes	Distillation	Alcolyzer Wine	-	Distillation
	A	B	B-A	
Liqueur 5	9.52	9.50	-0.02	13.55
Liqueur 6	10.22	10.14	-0.08	9.84
Liqueur 7	10.30	10.20	-0.10	15.29
Liqueur 8	11.23	11.09	-0.14	15.12

2. Validation of Alcolyzer measurements
(1) Repeatability and trueness for spirits
The results for the spirit samples are shown in Table 3. The relative standard deviation (RSD) values of the five samples were between 0.00% and 0.09%, and were below the upper limit of 0.3% established by NTA. The Grubbs's test results are shown in Table 4. The Grubbs's test statistic for Alcolyzer sample 2 and sample 5 was 1.789, which is larger than the critical value of 1.715 when the tests are repeated five times and the significance level is 0.05. However, the alcohol analysis values were only 0.01% v/v higher than the average values at these points. As such, we considered that these values were not outliers, and we included them in the RSD calculation.
The trueness test results are shown in Table 5. The mean difference in the alcohol concentration between the distillation method and the Alcolyzer method was between -0.05% and 0.07% v/v, and remained within the limit of ±0.1% v/v established by NTA. The Grubbs's test statistics for the Alcolyzer method have already been described in the previous paragraph, and all data were used even when the Grubbs's statistic was higher than the critical value of 1.715.
(2) Repeatability and trueness for liqueurs
The results for the liqueur samples are shown in Table 6. The RSD values of the five samples were between 0.00% and 0.17%, and were below the upper limit of 0.3% established by NTA. The Grubbs's test results are shown in Table 7. The Grubbs's test statistic for Alcolyzer sample 9 was 1.789, which is larger than the critical value of 1.715 when the tests are repeated five times and the significance level is 0.05. However, the alcohol analysis value was only 0.01% v/v higher than the average value at this point. As such, we considered that this value was not an outlier, and we included it in the RSD calculation.
The trueness test results are shown in Table 8. The mean alcohol concentration between the distillation method and the Alcolyzer method was between -0.08% and 0.05% v/v, and remained within the limit of ±0.1% v/v established by NTA. The Grubbs's test statistics for the Alcolyzer method have already been described in the previous paragraph, and all data were used even when the Grubbs's statistic was higher than the critical value of 1.715. However, the alcohol analysis value was only 0.01% v/v higher than the average value at this point. As such, we considered that this value was not an outlier, and we included it in the trueness evaluation.

Table 3. Repeatability of spirits samples using Alcolyzer (% v/v)

	1	2	3	4	5	Average	S.D.	RSD(%)
Sample 1	0.52	0.52	0.52	0.52	0.52	0.520	0.000	0.00
Sample 2	5.30	5.31	5.30	5.30	5.30	5.302	0.004	0.08
Sample 3	9.27	9.27	9.27	9.27	9.27	9.270	0.000	0.00
Sample 4	15.37	15.35	15.35	15.35	15.33	15.350	0.014	0.09
Sample 5	20.34	20.35	20.34	20.34	20.34	20.342	0.004	0.02

Table 4. Grubbs's test results of spirits

	Statistic of distillation method					Statistic of Alcolyzer method				
	1	2	3	4	5	1	2	3	4	5
Sample 1	0.730	0.730	1.095	0.730	1.095	-	-	-	-	-
Sample 2	1.414	0.707	0.707	0.707	0.707	0.447	1.789	0.447	0.447	0.447
Sample 3	0.074	0.824	0.972	0.972	1.194	-	-	-	-	-
Sample 4	1.228	0.351	0.351	0.526	1.403	1.414	0.000	0.000	0.000	1.414
Sample 5	0.447	0.447	1.193	1.043	1.043	0.447	1.789	0.447	0.447	0.447

Table 5. Alcohol concentration difference between distillation and Alcolyzer method, spirits (alcohol:% v/v)

	Distillation method						(Alcolyzer method) − (Distillation method)
	1	2	3	4	5	Average	
Sample 1	0.50	0.50	0.51	0.50	0.51	0.50	0.02
Sample 2	5.24	5.27	5.27	5.25	5.27	5.26	0.04
Sample 3	9.20	9.21	9.19	9.19	9.21	9.20	0.07
Sample 4	15.30	15.31	15.31	15.32	15.33	15.31	0.04
Sample 5	20.40	20.40	20.41	20.38	20.38	20.39	-0.05

Table 6. Repeatability of liqueur samples using Alcolyzer (% v/v)

	1	2	3	4	5	Average	S.D.	RSD(%)
Sample 6	0.52	0.52	0.52	0.52	0.52	0.520	0.000	0.00
Sample 7	3.15	3.14	3.15	3.15	3.14	3.146	0.005	0.17
Sample 8	5.32	5.32	5.32	5.32	5.32	5.320	0.000	0.00
Sample 9	8.11	8.11	8.11	8.12	8.11	8.112	0.004	0.06
Sample 10	10.46	10.46	10.46	10.46	10.46	10.460	0.000	0.00

Table 7. Grubbs's test results of liqueur

	Statistic of distillation method					Statistic of Alcolyzer method				
	1	2	3	4	5	1	2	3	4	5
Sample 6	0.730	1.095	0.730	1.095	0.730	-	-	-	-	-
Sample 7	1.565	0.671	0.671	0.447	0.671	0.730	1.095	0.730	0.730	1.095
Sample 8	1.230	0.671	1.006	0.112	1.006	-	-	-	-	-
Sample 9	0.671	0.671	0.671	0.447	1.565	0.447	0.447	0.447	1.789	0.447
Sample 10	1.789	0.447	0.447	0.447	0.447	-	-	-	-	-

Table 8. Alcohol concentration difference between distillation and Alcolyzer method, liqueur (alcohol:% v/v)

	Distillation method						(Alcolyzer method) − (Distillation method)
	1	2	3	4	5	Average	
Sample 6	0.48	0.49	0.48	0.49	0.48	0.48	0.04
Sample 7	3.08	3.10	3.10	3.09	3.10	3.09	0.05
Sample 8	5.26	5.27	5.30	5.28	5.30	5.28	0.04
Sample 9	8.09	8.09	8.09	8.10	8.11	8.10	0.02
Sample 10	10.53	10.54	10.54	10.54	10.54	10.54	-0.08

(3)Reproducibility for spirits
The results are not shown in the tables. The relative standard deviation for reproducibility (RSDR) values were 0.16% for the sample containing 5% v/v alcohol, 0.13% for the sample containing 7% v/v alcohol, and 0.08% for the sample containing 9% v/v alcohol; they were all below the upper limit of 0.5% established by NTA. The largest Cochran statistic was 0.333, which is below the critical value of 0.602 when 10 laboratories conduct the tests two times each, and there were no outliers.
(4) Reproducibility for liqueurs
The results are not shown in the tables. The RSDR values were 0.49% for the sample containing 3% v/v alcohol, 0.24% for the sample containing 5% v/v alcohol, and 0.15% for the sample containing 7% v/v alcohol; they were all below the upper limit of 0.5% established by NTA. The Cochran statistic of sample O analyzed at laboratory F was 1.000, which is larger than the critical value of 0.602 when 10 laboratories conduct the tests two times each. However, the difference between the two tests performed for sample O by laboratory F was only 0.01%. As such, we considered that this value was not an outlier, and we included it in the RSDR calculation.

CONCLUSIONS

1. It was concluded that the Alcolyzer method is capable of analyzing RTD.
2. The highest alcohol concentration that can be analyzed by the Alcolyzer is 20% v/v for spirit samples and 10% v/v for liqueur samples. The highest extract concentration that can be analyzed by the Alcolyzer is 13% w/v for liqueur samples.
3. The Alcolyzer method is accepted NTA as a rational and accurate method to analyze RTD.
4. The BCOJ Analysis Committee recommends that the alcohol concentration analysis in RTD by Alcolyzer be adopted for inclusion in *the Methods of Analysis of BCOJ*.

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