

Iso- α -Acids in Beer by Solid-Phase Extraction and High-Performance Liquid Chromatography

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Keyword: Beer bitterness

RECOMMENDATION

Repeat the collaborative study of both methods with a larger number of participants to determine whether the reproducibility can be improved.

CONCLUSIONS

1. The number of collaborators was marginal to permit a valid statistical analysis.
2. The repeatability coefficients of variation for both methods for all sample pairs were acceptable.
3. The reproducibility coefficients of variation for the method of Bloomfield et al were unacceptable. However, those of the Donley method were acceptable.

This subcommittee was charged with the evaluation of methodology to determine the concentration of iso- α -acids in beer using solid-phase extraction techniques for sample preparation. Following sample preparation, the samples were analyzed by the HPLC method developed by Ono et al (2) for the determination

TABLE I
Iso- α -acids in Beer Using Bloomfield et al Method (mg/L)

Collaborator	Sample Pair		Sample Pair		Sample Pair		Sample Pair	
	A	B	C	D	E	F	G	H
1	3.87	2.37	4.35	4.18	8.11	6.55	10.92	10.33
2	4.25	4.54	7.68	8.15	11.78	13.02	20.99	19.47
3	7.22	6.92	10.78	10.48	14.44	13.98	23.70	22.94
4	6.30	6.90	11.80	11.93	16.51	16.35	24.10	23.94
5	6.66	6.63	11.45	11.39	15.72	16.76	26.65	27.49
6	5.53	5.56	9.77	9.70	14.97	14.58	22.83	22.72
7	5.20	4.70	8.60	9.40	12.60	12.30	19.40	20.90
Mean	5.576	5.374	9.204	9.319	13.447	13.363	21.227	21.113
Grand mean	5.475		9.261		13.405		21.170	

TABLE II
Iso- α -acids in Beer Using Donley Method (mg/L)

Collaborator	Sample Pair		Sample Pair		Sample Pair		Sample Pair	
	A	B	C	D	E	F	G	H
1	6.72 ^a	5.14 ^a	12.47	11.13	17.15	17.92	28.77	29.67
2	5.38	5.18	9.21	8.87	13.90	13.84	21.29	20.04
3	6.78	7.04	11.07	11.16	16.66	15.49	22.41	24.93
4	6.23	6.33	10.55	10.16	15.48	15.05	23.59	23.23
5	6.62	6.61	11.28	11.52	17.36	17.74	28.03	27.86
6	5.79	5.81	9.91	10.21	15.45	15.53	25.13	24.64
7	4.90	5.40	8.10	8.40	12.70	12.80	20.00	20.90
Mean ^b	5.950	6.062	10.370	10.207	15.529	15.481	24.174	24.467
Grand mean ^b	6.006		10.289		15.505		24.321	

^aOutlier at $P \leq 0.01$ based on totals and/or differences (1).

^bCalculated excluding outliers.

TABLE III
Statistical Summary of Results^a

Method and Sample Pair	No. of Labs	Grand Mean	Repeatability			Reproducibility		
			s_r	cv_r	r_{95}	S_R	cv_R	R_{95}
Bloomfield et al								
A/B	7	5.475	0.479	8.7	1.341	1.464	26.7	4.099
C/D	7	9.261	0.276	3.0	0.773	2.602	28.1	7.284
E/F	7	13.405	0.676	5.0	1.893	3.160	23.6	8.848
G/H	7	21.170	0.715	3.4	2.003	5.241	24.8	14.674
Donley								
A/B	6	6.006	0.171	2.9	0.480	0.726	12.1	2.034
C/D	7	10.289	0.421	4.1	1.178	1.323	12.9	3.705
E/F	7	15.505	0.437	2.8	1.225	1.804	11.6	5.051
G/H	7	24.321	0.882	3.6	2.469	3.401	14.0	9.522

^aCalculations were made based on reference 5.

of iso- α -acids in hop extracts. Two sample preparation techniques were evaluated; those of Bloomfield et al (3) and of Donley (4).

PROCEDURE

Four sample pairs of beers labeled A/B, C/D, E/F, and G/H were sent to collaborators. The bitterness unit levels of the samples ranged from 6 to 21. The four sample pairs were prepared and analyzed for concentration of iso- α -acids using the methods of Bloomfield et al and Donley. Results were reported as milligrams of iso- α -acids per liter of beer. The Youden unit block experimental design (5) was used.

RESULTS AND DISCUSSION

The results of seven collaborators are shown in Tables I and II. One outlier in the A/B pair analyzed by the Donley method (Table II) was found and excluded from calculations. The results of the Youden unit block statistical treatment are shown in Table III.

The repeatability coefficients of variation were acceptable for both methods. The reproducibility coefficients of variations for the Donley method were acceptable, while those of the Bloomfield et al method were unacceptable.

The overall results from both methods were promising enough to repeat the evaluation with additional collaborators.

LITERATURE CITED

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