

Atomic Absorption

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Elemental Analysis of Beer by Flame Atomic Absorption Spectrometry with the PinAAcle 900 AAS

Introduction

Beer is a widely consumed beverage with both organic and inorganic components. The concentrations of the inorganic components may vary depending on raw materials and brewing processes. Knowledge of

the type and concentration of inorganic components in beer is of considerable interest from various perspectives, as they may affect taste, appearance, product stability, and health of the consumer¹. The determination of elements in beer by flame atomic absorption spectrometry (FAAS) is a well-known procedure². For example, the American Society of Brewing Chemists (ASBC) in St. Paul, Minnesota, USA, is proposing the regular determination of calcium (Ca), copper (Cu), iron (Fe), and sodium (Na) in beer by FAAS³.

FAAS has the benefit of providing precise and accurate measurements at a lower cost per element than more advanced elemental techniques, and also requires less operator training than many other trace elemental techniques. The PinAAcle™ 900 FAAS provides an intuitive, highly efficient system capable of simplifying analyses while maintaining peak performance and unmatched productivity.

Experimental

Instrumentation

All measurements were performed on a PerkinElmer PinAAcle 900T atomic absorption spectrometer equipped with high sensitivity nebulizer (HSN) and ceramic impact bead. An air-C₂H₂ flame with a 10 cm 3-slot solid titanium burner head was used for the determination of copper (Cu), iron (Fe), zinc (Zn), and manganese (Mn). Aluminum (Al) was determined with N₂O-C₂H₂ flame on a 5 cm solid titanium burner head. A nebulizer spacer was used for calcium (Ca) and sodium (Na) to reduce sensitivity, and for Al to improve N₂O flame stability and minimize interferences. Lumina™ cableless hollow cathode lamps were used for all elements.

Sample Preparation

Several brands of beer were purchased in a local supermarket in Singapore. When available, the same brand was purchased in two different packaging materials: a glass bottle and a metal can. A total of five bottled and six canned beers were analyzed. Sample aliquots for analyses were obtained by pouring the beers in 50 mL polyethylene autosampler tubes with caps. Samples were degassed of CO₂ by ultra-sonication at full power for 30 minutes and then acidified to 2 % (v/v) with HNO₃ (70 % w/v, Clean Room Chemical, Air Products and Chemicals Inc, Allentown, Pennsylvania, USA).

All elements were measured against external calibration curves with linear-through-zero regression, except Na, which used a non-linear through zero regression. Standards were prepared by serial dilutions of 1000 mg/L PerkinElmer Pure single-element standards in 2 % HNO₃ (v/v).

Elements usually present at trace levels (Al, Cu, Fe, Mn, and Zn) were determined directly in the undiluted beers. The calibration solutions for these elements were prepared in 5 % (v/v) ethanol (99.5 % GR grade, Kanto Chemical Co. Inc., Japan) for matrix matching. For Al determination, 0.2 % lanthanum (La) (w/v) was

added to all samples and standards as an ionization buffer (La₂O₃ 99.5 % LAB grade, Merck, Germany).

For the determination of Ca and Na, the samples were diluted 30 fold with ≥ 18 M Ω ultrapure water (MilliQ system, Millipore, Billerica, Massachusetts, USA). Calibration standards were prepared in 1 % HNO₃ (v/v). No ethanol was added, due to the dilution factor. La 0.2 % (w/v) was added as a releasing agent (to avoid phosphate suppression on Al) and as an ionization suppressant for Na and Ca (releasing agents are cations that react preferentially with an interferent). Table 1 shows the instrumental conditions used for this work.

Results and Discussion

Each beer sample was given a number to identify the brand and container type. Samples labeled “G” were from glass bottles, while samples labeled “M” were from metal cans. Results, reported in Table 2, show that Ca and Na are present at high concentrations (mg/L), while other elements are present at μ g/L levels, as expected. The data showed good quality for all beers tested, with respect to their elemental contents, based on the current ASBC guidelines. These results indicate that the container material (glass or can) does not significantly contribute to the element content of the beer, with the exception of Mn, which is always a little higher in the bottled beers.

Due to the low level of Al in the beer samples tested, it could not be detected by FAAS in most samples. Instead, a more sensitive technique, such as graphite furnace atomic absorption spectrometry (GFAAS), should be used for Al determination. The PinAAcle 900T (and 900H) can easily be switched between flame and graphite furnace modes, offering the capability to determine the low concentration elements by GFAAS using a single system. For analysis using flame-only atomic absorption, the PinAAcle 900F is also available.

Table 1. Instrument settings for the analysis of beer.

Element	Wavelength (nm)	Slit (nm)	Lamp Current (mA)	Units	Calibration Standards	Air (L/min)	Nitrous Oxide (L/min)	Acetylene (L/min)
Al	309.27	0.7	25	mg/L	2, 5	---	10.0	7.98
Ca	422.67	0.7	10	mg/L	0.5, 0.8, 2.5	8.68	---	2.48
Cu	324.75	0.7	15	μ g/L	40, 100, 200	10.0	---	3.16
Fe	248.33	0.2	30	μ g/L	100, 250, 500	10.0	---	3.16
Mn	279.48	0.2	20	μ g/L	50, 125, 250	10.0	---	3.16
Na	589.00	0.2	8	mg/L	0.5, 0.8, 2.5	8.68	---	2.48
Zn	213.86	0.7	15	μ g/L	50, 125, 250	10.0	---	3.16

Table 2. Results for the analysis of multiple beer samples using flame atomic absorption.

Sample	1M	2G	3M	3G	4M	4G	5M	5G	6G	7M	7G
Al (mg/L)	0.14	ND	ND	ND	ND	ND	ND	ND	ND	0.19	ND
Ca (mg/L)	79	104	72	93	139	143	107	143	96	77	76
Cu (μ g/L)	35	31	63	62	37	33	45	44	46	36	35
Fe (μ g/L)	43	42	45	31	22	30	52	68	54	25	25
Mn (μ g/L)	62	144	107	136	87	118	123	153	190	64	72
Na (mg/L)	40	47	35	46	86	82	72	69	207	196	187
Zn (μ g/L)	2.4	0.7	6.1	3.6	1.2	0.3	2.5	29	1.2	4.4	5.3

ND = not detected

Quality Control

For beer analysis, there are no certified reference materials (CRMs) available with certified elemental content. For this reason, quality control (QC) procedures were implemented by running selected samples in duplicate and after performing a spike to demonstrate the method's capability for precision and recovery. Due to the spread in concentration levels, spike additions were performed at mid-range of calibration curves, to provide a detectable signal increase (Table 3). Due to limited sample, not all elements were analyzed in all samples.

Analytical results of some samples run in duplicate were utilized to demonstrate analytical precision. Sample duplicates were carried through the full sample preparation process. The obtained results, reported in Table 4, show a good level of repeatability, even when using disposable plastic-ware, which was used in this application, instead of the typical calibrated glassware.

Conclusions

The present work reports the usage of the PinAAcle 900T AAS in flame mode for the determination of several elements relevant to the beer industry. The procedure is simple, fast, and accurate, requires no sample digestion, and can be applied to the quality control of beer manufacturing products when using a customer-validated application. The reported results prove that the PinAAcle 900 FAAS has the capability to determine elements in beer with high accuracy and precision.

Table 3. Spike recovery tests.

Element	Spike Level	% Recovery					
		3M	3G	4M	4G	6G	7G
Ca	1 mg/L		90	105			103
Cu	100 µg/L	93			97	95	
Fe	250 µg/L	98			98	101	
Mn	125 µg/L		100	95			94
Na	1 mg/L		103	110			103
Zn	125 µg/L		99	96			93

Table 4. Duplicate tests.

Sample	3M		3G		4M		4G		6G		7G	
	1	2	1	2	1	2	1	2	1	2	1	2
Ca (mg/L)			93	93	139	134					76	75
Cu (µg/L)	63	59					33	32	46	43		
Fe (µg/L)	45	42					30	31	54	40		
Mn (µg/L)			136	135	87	89					72	69
Na (mg/L)			46	46	86	85					187	189
Zn (µg/L)			3.6	3.2	1.2	1.2					5.3	4.0

References

1. S. Caroli "The Determination of Chemical Elements in Food: Applications for Atomic and Mass Spectrometry", Wiley 2007.
2. PerkinElmer Flame-AAS Cook Book 03030152 rev. 2009.
3. American Society of Brewing Chemists (ASBC), <http://www.asbcnet.org>, International Check Sample Service, May 2010.

Consumables Used

Component	Part Number
Al Hollow Cathode Lamp	N3050103
Ca Hollow Cathode Lamp	N3050114
Cu Hollow Cathode Lamp	N3050121
Fe Hollow Cathode Lamp	N3050126
Mn Hollow Cathode Lamp	N3050145
Na Hollow Cathode Lamp	N3050148
Zn Hollow Cathode Lamp	N3050191
10 cm 3-slot Titanium Burner Head	N0400103
5 cm 1-slot Titanium Burner Head	N0400101
Al – 1000 mg/L Standard	N9300184 (125 mL) N9300100 (500 mL)
Ca – 1000 mg/L Standard	N9303763 (125 mL) N9300108 (500 mL)
Cu – 1000 mg/L Standard	N9300183 (125 mL) N9300114 (500 mL)
Fe – 1000 mg/L Standard	N9303771 (125 mL) N9300126 (500 mL)
Mn – 1000 mg/L Standard	N9303783 (125 mL) N9300132 (500 mL)
Na – 1000 mg/L Standard	N9303785 (125 mL) N9300152 (500 mL)
Zn – 1000 mg/L Standard	N9300178 (125 mL) N9300168 (500 mL)
Autosampler Tubes	B0193233 (15 mL) B0193234 (50 mL)