APPLICATION NOTE

Atomic Absorption

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Analysis of Major Elements in Drinking Water Using FAST Flame Sample Automation for Increased Sample Throughput

Introduction

Water quality is an ongoing issue that continues to present itself globally. As populations around the world come to identify the quality of their drinking water as a major concern and regulations continue to expand, the ability to measure and monitor the impurities in drinking water becomes paramount. In areas where simplicity, accessibility and system cost are major concerns, analysis via flame atomic absorption (AA) remains a useful tool that can be used to monitor inorganic impurities in drinking water.

Municipal and bottled water must conform to regulatory standards at all times. The facilities that process the water, the regulatory bodies with oversight, and the consumer all have great interest in ensuring that an excursion above the limits does not occur. The desire to maintain compliance at all times can result in sampling protocols that require continuous analysis of a large number of samples in a short amount of time. Consistent accuracy, precision and productivity are critically important for a laboratory to be able to successfully process these samples without excessive re-prep or additional sample handling.

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Elements of interest can vary greatly by region and regulatory body, and this application will examine some commonly regulated high-level elements in a variety of drinking water samples, including municipal and bottled waters. Table 1 lists the elements measured, along with representative regulatory limits.

Table 1. Select Elements in Drinking Water.

Element	Limit (mg/L)
Copper (Cu)	1.3
Iron (Fe)	0.3
Magnesium (Mg)	50
Zinc (Zn)	5
Sodium (Na)	200
Calcium (Ca)	200

Experimental

All analyses were performed on a PerkinElmer PinAAcle™ 900T atomic absorption spectrometer operating in flame mode (equivalent results would also be obtained on PinAAcle 900H and 900F models.) Instrument conditions for the analysis of the water samples are outlined in Table 2. A high-sensitivity nebulizer was used with the standard spray chamber and a 10 cm burner head. External calibrations were performed using a single intermediate standard made in 2% HNO_3/deionized water which was then diluted in-line using the capabilities of the PerkinElmer FAST Flame 2 sample automation accessory. The water samples were acidified with nitric acid and then run directly without further preparation other than spiking.

The FAST Flame 2 accessory is a combination of high-speed autosampler, peristaltic pump and switching valve which provides quick sample turnaround with fast rinse-out, short signal stabilization times and no sample-to-sample memory effect. The FAST Flame 2 rapidly fills a sample loop via vacuum and then switches to inject the sample loop while the autosampler moves to the next sample. This removes the time delay associated with self-aspiration or peristaltic pumping and eliminates the long rinse-in and rinse-out times as a result of autosampler movement and flushing, resulting in complete sample-to-sample analytical times as short as 15 seconds.

The ability of the FAST Flame 2 to mechanically pump the sample during injection allows for ideal optimization of nebulizer and flame conditions, eliminates variability due to changes in sample viscosity, dissolved solids, and tubing length and also provides long-term sample flow stability. The in-line dilution capability allows the analyst to create a single intermediate standard, and then the FAST Flame 2 system automatically generates all calibration standards in-line, as required. In addition, the instrument can be set to identify QC over-range samples and then utilize the in-line dilution capability to automatically re-run a sample that falls outside the calibration range at an increased dilution factor, bringing the signal within the calibration range and providing accurate measurement along with a successful QC check.

The water samples were spiked at levels well below the regulatory guidelines to demonstrate the accuracy of the method at lower-than-regulated levels.

Table 2. PinAAcle Instrument and Analytical Conditions.

Parameter	Cu	Fe	Mg	Zn	Na	Ca
Wavelength (nm)	324.75	248.33	285.21	213.86	589.00	422.67
Slit (nm)	0.7	0.2	0.7	0.7	0.2	0.7
Air Flow (L/min)	2.5	2.5	2.5	2.5	2.5	2.5
Acetylene Flow (L/min)	10	10	10	10	10	10
Acquisition Time (sec)	3	3	3	3	3	3
Replicates	3	3	3	3	3	3
Sample Flow Rate (mL/min)	6	6	6	6	6	6
Intermediate Standard (mg/L)	1	2	1	2	20	20
Auto-Diluted Calibration Standards (mg/L)	[0.05] [0.1] [0.25] [0.5] [1]	[0.1] [0.2] [0.5] [1] [2]	[0.05] [0.1] [0.25] [0.5] [1]	[0.1] [0.2] [0.5] [1] [2]	[0.5] [1.0] [2.0] [5.0]	[1.0] [2.0] [5.0] [10.0]
Calibration Curve Type	Non-Linear Through Zero	Non-Linear Through Zero	Non-Linear Through Zero	Non-Linear Through Zero	Non-Linear Through Zero	Non-Linear Through Zero

Results and Discussion

The calibration curves were created from a single intermediate standard using the in-line dilution capabilities of the FAST Flame 2 accessory. Calibration results are shown in Table 3. The excellent correlation for the calibration standards demonstrates the value of the automatic in-line sample and standard dilution available on the FAST Flame 2 accessory. The independent calibration verification recoveries ensure that the calibration is valid and that the creation of standards via the dilution system is accurate.

Tables 4-9 show the results for the analyses of the water samples. The measured concentrations of magnesium, sodium and calcium in the samples varied enough to fall outside the calibration curve. The in-line dilution capability of the FAST Flame 2 accessory then automatically diluted and re-ran these samples so that the absorbance was within the calibration curve and the results represented accurate analysis. The calcium in the Bottled "Mineral" Water A sample was of such a high concentration that the spike should have been higher than 10 mg/L for ideal recovery. In all cases, the water samples were in compliance for the elements tested. With the exception of a single calcium value, spike recoveries were within 10% of the spiked values, even when spiked at well below the regulated values.

In addition, the FAST Flame 2 accessory reduced the creation of standards from one intermediate and five final standards to a single intermediate standard with a commensurate reduction in human error during creation. The FAST Flame 2 accessory was also able to react to the over-range samples and auto-dilute the samples accurately and consistently without interaction from an analyst, saving time and eliminating additional sample handling and re-prep.

When analyzing a large number of samples or over a long period, there is always concern that the analytical system will drift so that QC checks will fail. At worst, analytical samples will have to be re-prepped and re-run, while under the best case scenario the instrument might be recalibated and the samples in the autosampler re-analyzed. To avoid this problem altogether, the PinAAcle 900 AA spectrometer and FAST Flame 2 sample automation accessory have been designed to provide outstanding long-term stability, as demonstrated in Figure 1. Bottled Water B was spiked with 0.600 mg/L of magnesium and then continuously analyzed over the course of four hours with a sample frequency of once per minute. As can be seen, other than an initial settling time, the running average remains within +/- 1% of the mean throughout the entire duration with the actual samples well within +/- 3% of the mean. In addition, there is no indication of a rising or falling trend, indicating that the instrument could continue to successfully analyze samples and pass QC checks well beyond four hours without any input from the analyst and with no recalibrations.

These results demonstrate the robustness and accuracy of the analysis and the speed and increased productivity available from the PinAAcle 900 and the FAST Flame 2 system.

Table 3. Calibration Results.

Element	Correlation Coefficient	ICV Concentration (mg/L)	Measured ICV (mg/L)	ICV (% Recovery)
Copper (Cu)	0.99994	0.500	0.494	98.8
Iron (Fe)	0.99989	1.00	0.979	97.9
Magnesium (Mg)	0.99999	0.500	0.508	102
Zinc (Zn)	0.99999	1.00	1.03	103
Sodium (Na)	0.99997	5.00	5.05	101
Calcium (Ca)	0.99954	5.00	5.34	107

Table 4. Magnesium in Drinking Water.

Sample	Measured Concentration (mg/L)	In-line Dilution Factor	Measured Sample + Spike Concentration (0.250 mg/L Spike)	% Spike Recovery
Municipal Water A	0.062	1	0.311	99.6
Municipal Water B	1.09	5	1.34	99.6
Bottled Water A	0.010	1	0.262	101
Bottled Water B	0.232	1	0.487	102
Bottled Water C	1.15	4	1.40	101
Bottled "Mineral" Water A	0.354	2	0.611	103
Bottled "Mineral" Water B	6.52	10	6.75	93.2

Table 5. Iron in Drinking Water.

Sample	Measured Concentration (mg/L)	In-line Dilution Factor	Measured Sample + Spike Concentration (0.500 mg/L Spike)	% Spike Recovery
Municipal Water A	0.136	1	0.654	104
Municipal Water B	0.008	1	0.538	106
Bottled Water A	0.018	1	0.522	101
Bottled Water B	0.000	1	0.509	102
Bottled Water C	0.037	1	0.510	94.6
Bottled "Mineral" Water A	0.057	1	0.547	98.0
Bottled "Mineral" Water B	0.048	1	0.524	95.2

Table 6. Copper in Drinking Water.

Sample	Measured Concentration (mg/L)	In-line Dilution Factor	Measured Sample + Spike Concentration (0.500 mg/L Spike)	% Spike Recovery
Municipal Water A	0.100	1	0.600	100
Municipal Water B	0.187	1	0.683	99.2
Bottled Water A	0.004	1	0.499	99.0
Bottled Water B	0.006	1	0.476	94.0
Bottled Water C	ND	1	0.494	102
Bottled "Mineral" Water A	0.002	1	0.463	92.2
Bottled "Mineral" Water B	0.000	1	0.463	92.6

Table 7. Zinc in Drinking Water.

Sample	Measured Concentration (mg/L)	In-line Dilution Factor	Measured Sample + Spike Concentration (0.500 mg/L Spike)	% Spike Recovery
Municipal Water A	0.080	1	0.589	102
Municipal Water B	0.002	1	0.500	99.6
Bottled Water A	ND	1	0.492	99.0
Bottled Water B	ND	1	0.500	101
Bottled Water C	ND	1	0.502	101
Bottled "Mineral" Water A	0.003	1	0.520	103
Bottled "Mineral" Water B	ND	1	0.498	100

Table 8. Sodium in Drinking Water.

Sample	Measured Concentration (mg/L)	In-line Dilution Factor	Measured Sample + Spike Concentration (10.00 mg/L Spike)	% Spike Recovery
Municipal Water A	9.55	10	19.0	94.6
Municipal Water B	20.3	15	30.0	97.1
Bottled Water A	2.84	10	12.3	94.1
Bottled Water B	0.996	15	10.9	98.8
Bottled Water C	15.6	10	25.2	96.2
Bottled "Mineral" Water A	9.81	10	19.3	94.9
Bottled "Mineral" Water B	37.4	25	47.1	97.7

Table 9. Calcium in Drinking Water.

Sample	Measured Concentration (mg/L)	In-line Dilution Factor	Measured Sample + Spike Concentration (10.00 mg/L Spike)	% Spike Recovery
Municipal Water A	7.18	5	17.2	100
Municipal Water B	17.9	10	27.8	99.3
Bottled Water A	2.08	5	12.5	105
Bottled Water B	0.027	5	10.0	100
Bottled Water C	5.24	5	15.2	99.1
Bottled "Mineral" Water A	146	40	*	*
Bottled "Mineral" Water B	10.9	10	21.2	103

*For Bottled "Mineral" Water A, the Ca concentration was too high to obtain a meaningful spike recovery.

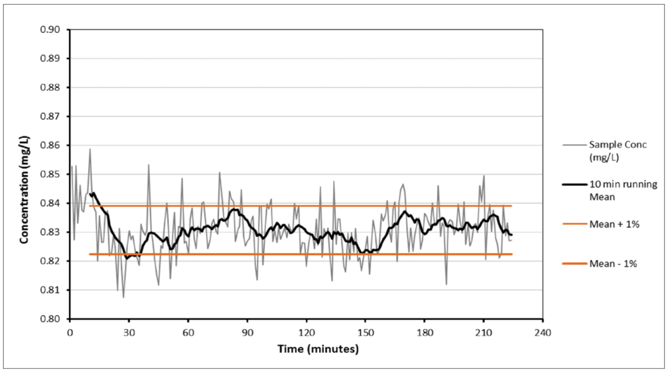

Figure 1. Four-hour stability of Mg spiked at 0.600 mg/L in Bottled Water B.

Conclusion

This work has demonstrated the ability of the PinAAcle 900 AA spectrometer to reliably and effectively analyze drinking water samples for Mg, Fe, Cu, Zn, Na and Ca over a wide range of concentrations. Using the FAST Flame 2 sample automation accessory along with the PinAAcle minimizes user errors when performing dilutions and making calibration standards, increases throughput, and provides excellent long-term stability, increasing productivity for the laboratory. However, the same analyses can also be done without the use of a FAST Flame 2 if/when analyzing smaller sample batches.

Consumables

Component	Part Number
Red/Red PVC Pump Tubing	N8145158
Black/Black PVC Pump Tubing	N8145153 (unflared) N8145202 (flared)
Autosampler Tubes	B0193233 (15 mL) B0193234 (50 mL)
Ca Hollow Cathode Lamp	N3050114
Cu Hollow Cathode Lamp	N3050121
Fe Hollow Cathode Lamp	N3050126
Mg Hollow Cathode Lamp	N3050144
Na Hollow Cathode Lamp	N3050148
Zn Hollow Cathode Lamp	N3050191
Pure-Grade Ca Standard (1000 mg/L)	N9303763 (125 mL) N9300108 (500 mL)
Pure-Grade Cu Standard (1000 mg/L)	N9300183 (125 mL) N9300114 (500 mL)
Pure-Grade Fe Standard (1000 mg/L)	N9303771 (125 mL) N9300126 (500 mL)
Pure-Grade Mg Standard (1000 mg/L)	N9300179 (125 mL) N9300131 (500 mL)
Pure-Grade Na Standard (1000 mg/L)	N9303785 (125 mL) N9300152 (500 mL)
Pure-Grade Zn Standard (1000 mg/L)	N9300178 (125 mL) N9300168 (500 mL)

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