

Atomic Absorption

Authors:

Deborah Bradshaw

Atomic Spectroscopy
Training and Consulting

Kenneth Neubauer

PerkinElmer, Inc.

Analysis of Minerals in Drinking Water with the PinAAcle 500 Atomic Absorption Spectrometer

Introduction

With water quality varying widely with geography and geology, as well as pollution considerations, it is important to know the metal content of waters, both for consumption and industrial use. Although a variety of techniques can measure minerals in water, one of the simplest, least

expensive, and fastest is flame atomic absorption (AA) spectrometry. As a result, the technique continues to enjoy widespread use, despite the increasing popularity of ICP-OES and ICP-MS.

This work focuses on the determination of seven non-toxic elements usually found in drinking waters with the PerkinElmer PinAAcle™ 500 flame atomic absorption spectrometer. Although other lower-level elements can also be measured by flame AA, these are most commonly analyzed by either graphite furnace AA, ICP-OES, or ICP-MS.

Experimental

Samples consisted of municipal and well waters collected locally, spring waters purchased from a local grocery store, and a certified drinking water standard (Trace Metals in Drinking Water – High-Purity Standards™, Charleston, South Carolina, USA). Sample preparation consisted only of acidifying each water with 1% HNO₃ (v/v) and adding 0.1% lanthanum chloride as a releasing reagent for calcium (Ca) and magnesium (Mg) and as an ionization suppressant for sodium (Na) and potassium (K).

All analyses were carried out with the PinAAcle 500 flame AA spectrometer using the conditions in Tables 1 and 2. Due to the high mineral content, the burner was rotated 30 degrees to decrease the signal intensity for the analysis of the minerals. In addition, K and Na were analyzed in emission mode, which allowed the PinAAcle 500 to be auto-configured in such a way to extend the analytical range so that even higher concentrations could be measured. This allowed minimal dilution for K and elimination of dilution for Na.

Samples were introduced via self-aspiration with a high-sensitivity nebulizer, which is standard on the PinAAcle 500 spectrometer. The nebulizer was used without the spacer (providing maximum sensitivity) for the determinations of copper (Cu), iron (Fe), and zinc (Zn). The spacer was inserted for the determinations of Na, K, Mg and Ca.

Table 1. PinAAcle 500 instrument and analytical conditions common to all elements.

Parameter	Value
Air Flow (L/min)	2.5
Acetylene Flow (L/min)	10
Read Time (sec)	3
Replicates	3

Table 2. PinAAcle 500 instrument and analytical conditions specific to each element.

Element	Wavelength (nm)	Slit (nm)	Mode	Burner Angle (degrees)	Calibration Standards (mg/L)	Calibration Curve
Ca	422.67	0.7	Absorption	30	0.5, 1.0, 2.0, 5.0, 10, 20, 40	Non-Linear through Zero
Cu	324.75	0.7	Absorption	0	0.05, 0.10, 0.25, 0.50	Linear Through Zero
Fe	248.33	0.2	Absorption	0	0.05, 0.10, 0.25, 0.50, 1.0	Linear Through Zero
Mg	285.21	0.7	Absorption	30	0.5, 1.0, 2.0, 5.0, 10	Non-Linear Through Zero
K	766.49	0.7	Emission	30	2, 5, 10, 20, 30, 40, 50	Non-Linear Through Zero
Na	589.00	0.2	Emission	30	2, 5, 10, 20, 30, 40, 50	Non-Linear Through Zero
Zn	213.86	0.7	Absorption	0	0.05, 0.10, 0.25, 0.50	Linear Through Zero

Results and Discussion

All calibrations yielded correlation coefficients of 0.999 or greater. The accuracy of the calibrations was assessed with an independent calibration verification (ICV) solution, which was diluted 100 times to fall within the range of the calibration curve. The results of the ICV appear in Table 3 and demonstrate the accuracy of the calibration curves.

Table 3. Results for independent calibration verification (ICV).

Element	Concentration (mg/L)	Experimental (mg/L)	% Recovery
Ca	5.00	4.86	97
Cu	0.25	0.26	104
Fe	1.00	1.00	100
Mg	5.00	4.88	98
K	5.00	4.78	96
Na	5.00	5.12	102
Zn	0.20	0.21	105

To validate the methodology, a reference material was first analyzed, with the results shown in Table 4. All recoveries are within 10% of the certified value, demonstrating the accuracy of the methodology.

Table 4. Results for reference material (all units in mg/L).

Element	Experimental (mg/L)	Certified (mg/L)	% Recovery
Ca	33.4	35.0	95
Cu	0.022	0.020	110
Fe	0.095	0.100	95
Mg	8.69	9.00	97
K	2.28	2.50	91
Na	5.90	6.0	98
Zn	0.070	0.070	100

Table 5. Results for samples (all units in mg/L).

Element	Municipal Water (mg/L)	Well Water-1 (mg/L)	Well Water-2 (mg/L)	Well Water-3 (mg/L)	Spring Water-1 (mg/L)	Spring Water-2 (mg/L)
Ca	17.7	0.148	35.3	32.4	3.43	19.2
Cu	0.048	< DL	0.052	0.017	< DL	< DL
Fe	< DL	< DL	0.019	< DL	< DL	< DL
Mg	6.43	0.026	4.90	5.12	0.799	6.09
K	< 0	233*	4.89	4.10	0.73	0.69
Na	38.4	3.63	10.9	42.9	6.60	7.25
Zn	0.008	0.043	0.010	0.023	< DL	< DL

* Sample required a 10x dilution

With the accuracy of the method established, several drinking water samples from various sources were analyzed. The municipal and well water samples were collected directly from a faucet, while the spring water samples were poured from the bottles in which they were purchased. The results appear in Table 5.

The presence of Cu and Zn in the four samples collected from the faucet is most likely due to leaching from copper pipes, fittings, and solder. Well Water-1 is interesting as it contains the lowest levels of all samples, except for an extraordinarily high level of K. Further investigation determined that this residence has a water softener installed which utilizes K as the counter-ion to remove high levels of Ca and Mg from the well water.

As expected, Cu and Zn are not detected in the spring waters; only the minerals are present. The variation in mineral concentration is indicative of the different geologies of the areas where these waters originate.

Finally, detection limits were determined for Cu, Fe, and Zn as three times the standard deviation of ten blank measurements (i.e. 1% HNO₃), as shown in Table 6. Because of their elevated levels, detection limits were not determined for the mineral elements (i.e. Ca, K, Mg, Na). In addition, since these elements are usually present at high concentrations, the instrument was detuned for their analysis. Therefore, detection limits would be meaningless.

Table 6. Detection limits.

Element	Detection Limit (mg/L)
Cu	0.002
Fe	0.006
Zn	0.004

Conclusion

This work has demonstrated the ability of the PinAAcle 500 to successfully measure mineral elements in drinking water samples, including municipal, well, and spring waters. By taking advantage of the ability to rotate the burner and measure in emission mode, both trace and mineral elements could be measured. With Syngistix Touch™ software, the PinAAcle 500 AA spectrometer can be operated exclusively from a touchscreen interface. For greater flexibility, the ability to run Syngistix™ for AA software from an on-board computer is also available. This flexibility makes the PinAAcle 500 flame AA spectrometer an excellent choice for the analysis of drinking waters.

Consumables Used

Component	Part Number
High Sensitivity Nebulizer	N3160144
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
Zn Hollow Cathode Lamp	N3050191
Quality Control Standard, 21 Elements	N9300281
Initial Calibration Verification Standard	N9300224
Pure-Grade Ca Standard (1000 mg/L)	N9303763 (125 mL) N9300108 (500 mL)
Pure-Grade K Standard (1000 mg/L)	N9303779 (125 mL) N9300141 (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)