Analysis of Mineral and Heavy Metal Content in Beverages Using the Teledyne Leeman Lab's Prodigy7 ICP-OES

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Introduction

The presence of toxic elements in foodstuffs and beverages poses a global health risk that has raised public concern and necessitated scientific monitoring. In particular, the elements Cd, Pb and As are known to be detrimental to human health, and have been found in commercially available beverages. In order to provide adequate consumer protection, methods of determination must be both rapid and accurate, identifying contaminant level and source. By analyzing the sample directly, without sample digestion procedures and materials, Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES) provides an efficient and economical means of toxic element determination.

The goal of this application note is to demonstrate the ability of the Teledyne Leeman Lab's Prodigy7 simultaneous ICP-OES, to quickly and accurately measure mineral and heavy metal content in a variety of beverage samples. The obtained results were compared with the maximum allowable limits in drinking water by the United States Environmental Protection Agency (USEPA) and World Health Organization (WHO).[1, 2] Drinking water contaminants and maximum allowable limits are presented in Table I.

Table I Drinking Water Contaminants and Maximum Allowable Limits

	Heavy Metals (mg/L)				
	Ni	**Cu**	**As**	**Cd**	**Pb**
USEPA (2014)	0.1	1.3	0.01	0.005	0.015
WHO (2011)	0.07	2.0	0.01	0.003	0.01

Instrument

A Prodigy7 Inductively Coupled Plasma (ICP) Spectrometer equipped with a dual-view torch was used to generate the data for this application note.

The Prodigy7 is a compact bench-top simultaneous optical emission instrument featuring a 500 mm focal length Echelle optical system coupled with a mega-pixel Large Format CMOS (L-CMOS) detector. At 28 mm^2, the active area of the L-CMOS detector is significantly larger than any other solid-state detector currently used for ICP-OES. This combination allows the Prodigy7 to achieve higher optical resolution than other solid-state detector-based ICP systems. The detector also provides continuous wavelength coverage from 165 to 1100 nm permitting measurement over the entire ICP spectrum in a single reading without sacrificing wavelength range or resolution. This detector design is inherently anti-blooming and is capable of random access, non-destructive readout that results in a dynamic range of more than six orders of magnitude. The Prodigy7 also uses a 40.68 MHz rugged, water cooled, free-running RF Generator, allowing it to handle the most difficult sample matrices as well as common organic solvents.

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Sample Introduction

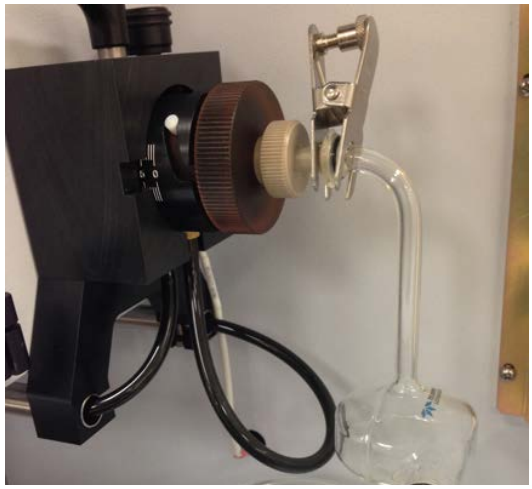
The sample introduction configuration used for this application note is shown in Table II.

Table II Sample Introduction Setup	
Nebulizer	Glass Conikal (PN 120-00463-1)
Spray Chamber	Glass Cyclonic with no Center Knockout Tube (PN 120-00461-2)
Torch Injector Diameter	2.5 mm (PN 318-00161-AQ1)
Sample Uptake Tubing	0.76 mm (PN 309-00069-7)
Sample Drain Tubing	1.14 mm (PN 309-00069-4)

Scandium (Sc) was used as an internal standard and was added to standards and samples using the Glass Expansion Triton Mixing Kit (PN 115-00431).

The volume of the cyclonic spray chamber is low and allows for fast washout between samples. The Prodigy7's torch is mounted using an innovative twist-n-lock cassette system shown in Figure 1. The design permits operators to remove and replace the torch to the exact same position, providing day-to-day reproducibility and simplified training.

Figure 1 Twist-n-Lock Sample Introduction System



Operating Parameters

For all elements of interest, background correction was performed simultaneously with the peak measurement, resulting in improved detection limits. All data was generated using the instrument operating parameters listed in Table III.

Table III Instrument Operating Parameters	
Parameter	**Setting**
RF Power	1.20 kW
Coolant Flow	15 L/min
Auxiliary Flow	1.0 L/min
Nebulizer Pressure	1.0 L/min
Uptake Rate	25 rpm
Integration Time: Axial Radial	 30 seconds 15 seconds

Calibration Standards

Calibration standards were prepared from single-element stock solutions (VHG Labs® Standards, Manchester, NH). The final concentrations are listed in Table IV.

Table IV Calibration Standards, ppm

Element	Wavelength, nm	Blank	STD1	STD2	STD3
Na	589.592 r	0	2.5	5.0	10.0
Mg	279.553 r	0	2.5	5.0	10.0
K	766.491 r	0	2.5	5.0	10.0
Ca	317.933 r	0	2.5	5.0	10.0
As	189.042	0	0.5	1.0	2.0
Cd	214.441	0	0.5	1.0	2.0
Cu	324.754	0	0.5	1.0	2.0
Pb	220.353	0	0.5	1.0	2.0
Zn	206.200	0	0.5	1.0	2.0
Ni	231.604	0	0.5	1.0	2.0
Co	228.615	0	0.5	1.0	2.0
Mn	257.610	0	0.5	1.0	2.0

Example calibration curves are shown in Figure 2 and Figure 3. The curves are based on the calibrations of Mg at 279.553 nm in radial-view mode and Cd at 214.441 in axial-view mode.

Figure 2 Calibration Curve of Mg at 279.553 nm

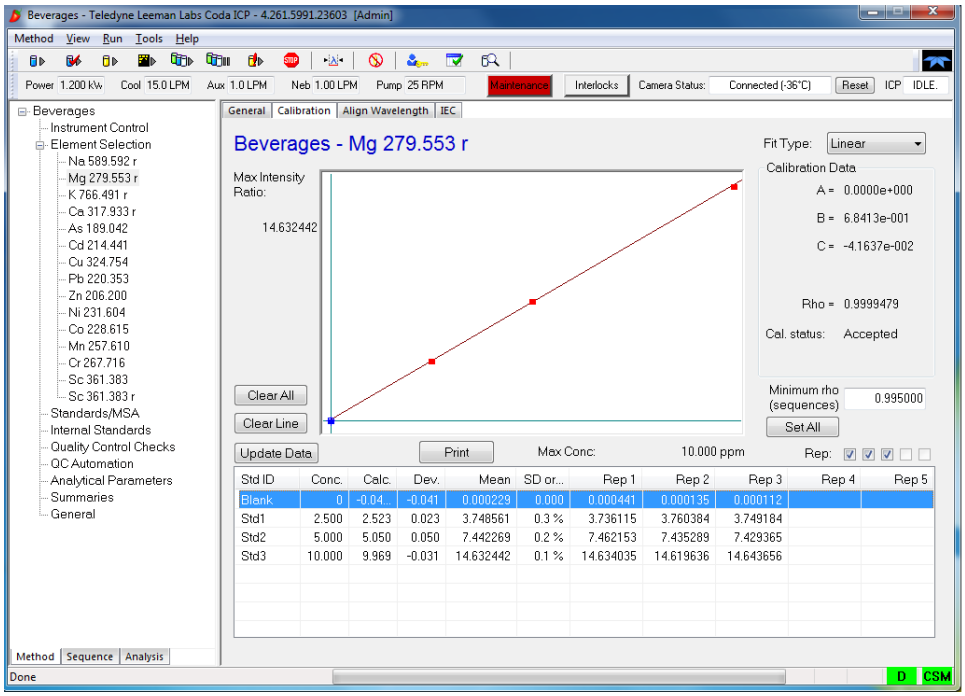


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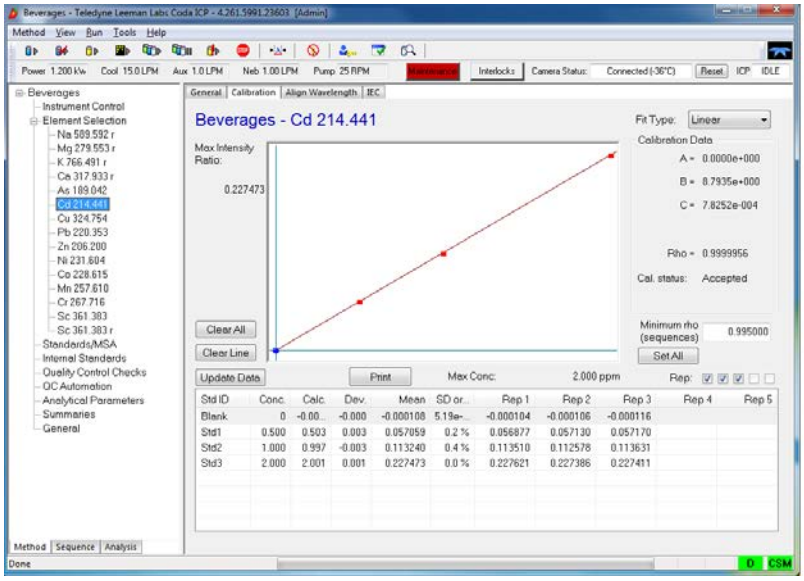
Figure 3 Calibration Curve of Cd at 214.441 nm



Sample Preparation

Three different beverages were analyzed: 100% apple juice, California red wine, and concentrated energy drink. Samples were prepared by weighing 20 g of each sample into 100 mL volumetric flasks for heavy metal content, and 1.0 or 2.0 g of each sample into 100 mL volumetric flasks for mineral content. Flasks were brought to final volume with deionized water. Samples were prepared in duplicates. The first preparation was analyzed without further modification. The second preparation was spiked for purposes of calculating spike recoveries.

Detection Limits

A study was performed to determine the Instrument Detection Limits (IDL) in dual-view mode for the elements of interest. Detection Limits shown in Table V were determined as concentrations corresponding to three times the standard deviation of 10 consecutive measurements of the calibration blank.

Table V Detection Limits (DL)

Element	Wavelength (nm)	DL (ppm)
Na	589.592 r	0.007
Mg	279.553 r	0.007
K	766.491 r	0.012
Ca	317.933 r	0.002
As	189.042	0.005
Cd	214.441	0.0001
Cu	324.754	0.0002
Pb	220.353	0.001
Zn	206.200	0.0001
Ni	231.604	0.0004
Co	228.615	0.0003
Mn	257.610	0.0001

Results

After igniting the plasma and allowing a 15 minute warm-up period, the Prodigy7 was calibrated. Once the calibration was complete, a QC standard was analyzed with acceptance criteria of ±10%. Results are shown in Table VI. Upon successful completion of the QC standard analysis, samples were analyzed.

Table VI QC Standard Results

Element	Wavelength (nm)	Standard Concentration (ppm)	Measured Conc (ppm)	Recovery %	RSD %
Na	589.592 r	5.0	5.0	100.6	0.5
Mg	279.553 r	5.0	5.1	101.9	0.5
K	766.491 r	5.0	5.2	104.9	1.1
Ca	317.933 r	5.0	5.0	100.5	0.5
As	189.042	1.0	1.0	100.5	0.4
Cd	214.441	1.0	1.0	100.7	0.2
Cu	324.754	1.0	1.0	101.3	0.2
Pb	220.353	1.0	1.0	101.5	0.4
Zn	206.200	1.0	1.0	100.6	0.2
Ni	231.604	1.0	1.0	101.2	0.2
Co	228.615	1.0	1.0	101.5	0.1
Mn	257.610	1.0	1.0	101.6	0.2

Results from the sample analysis are shown in Table VII, Table VIII and Table IX. Results for each beverage sample are reported in units of parts per million (ppm) with dilution factors applied. Results are also presented for the recoveries of the 1.0 ppm (heavy metals) and 5.0 ppm (minerals) spikes, along with %RSD values for the measured spike concentrations. Results are listed as Not Detected (ND) if the measured concentration was at or below the IDL.

Table VII 100% Apple Juice Results

Element	Wavelength (nm)	Dilution Factor	Avg. Conc (ppm)	Spike Recovery %	RSD %
Na	589.592 r	100	14.8	119.9	0.07
Mg	279.553 r	100	49.7	114.6	0.2
K	766.491 r	100	1265	96.9	0.2
Ca	317.933 r	100	402	113.3	0.2
As	189.042	5	ND	108.9	1.3
Cd	214.441	5	ND	98.6	0.5
Cu	324.754	5	0.05	98.2	0.2
Pb	220.353	5	ND	96.8	0.5
Zn	206.200	5	0.08	100.6	0.5
Ni	231.604	5	ND	98.1	0.3
Co	228.615	5	0.002	98.2	0.5
Mn	257.610	5	0.3	98.8	0.2

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Table VIII California Red Wine					
Element	Wavelength (nm)	Dilution Factor	Avg Conc (ppm)	Spike Recovery %	RSD %
Na	589.592 r	50	19.8	96.7	0.2
Mg	279.553 r	50	122.8	92.0	0.3
K	766.491 r	200	1140	94.9	0.9
Ca	317.933 r	50	72.8	90.9	0.4
As	189.042	5	0.2	100.7	1.4
Cd	214.441	5	0.001	110.4	0.3
Cu	324.754	5	0.03	94.7	0.2
Pb	220.353	5	0.003	104.5	0.3
Zn	206.200	5	1.2	117.6	0.3
Ni	231.604	5	0.02	106.3	0.2
Co	228.615	5	0.009	105.0	0.2
Mn	257.610	5	2.2	103.6	0.3

Table IX Concentrated Energy Drink					
Element	Wavelength (nm)	Dilution Factor	Avg Conc (ppm)	Spike Recovery %	RSD %
Na	589.592 r	50	255.4	102.4	0.3
Mg	279.553 r	50	ND	102.8	0.07
K	766.491 r	50	236.0	90.2	0.3
Ca	317.933 r	50	1.0	102.0	0.3
As	189.042	5	ND	103.0	1.0
Cd	214.441	5	ND	98.3	0.2
Cu	324.754	5	0.01	98.2	0.4
Pb	220.353	5	ND	97.9	0.4
Zn	206.200	5	0.015	99.5	0.3
Ni	231.604	5	ND	98.0	0.4
Co	228.615	5	0.40	98.1	0.3
Mn	257.610	5	0.012	98.6	0.4

Conclusions

The analysis of beverages was successfully performed using the Teledyne Leeman Labs Prodigy7 ICP-OES. The spike recovery data shows that all analytes were measured within ±20% of the spiked concentrations indicating that the method is free from matrix interferences. The use of an internal standard minimized differences related to sample nebulization efficiency and resulted in improved precision values. The image stabilized plasma combined with the simultaneous collection of both peak and background data provided exceptionally precise and stable results.

Considering the reliability of results and the simplicity of the sample treatment, the Prodigy7 ICP-OES quickly and accurately measured mineral and heavy metal content in a variety of sample beverages.

The results from this study were compared with the maximum acceptable limits in drinking water as defined by the USEPA and WHO. A majority of the trace contaminants were found to be below the allowable limits set by the USEPA and the WHO with the exception of arsenic (As) in California red wine, which exceeded both acceptable regulation limits. The heavy metal content in beverages may be due in part to the concentration of those elements in the beverage's raw ingredients which are affected by soil composition and external conditions throughout fruit growth and harvesting.

References

1. *Maximum Contaminant Levels and Regulatory Dates for Drinking Water - U.S. EPA VS California, Last updated July 2014*; California Environmental Protection Agency - State Water Resources Control Board: Sacramento, CA . 2014. [Online] http://www.waterboards.ca.gov/drinking_water/certlic/drinkingwater/documents/dwdocuments/MCLsEPAvsDWP-2014-07-01.pdf (accessed July 22, 2016)
2. World Health Organization. *Guidelines for Drinking-water Quality - 4th Ed.*; Geneva, Switzerland, 2011. [Online] http://apps.who.int/iris/bitstream/10665/44584/1/9789241548151_eng.pdf (accessed July 22, 2016)

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