

## Analysis of Mineral and Heavy Metal Content in Beverages Using the Teledyne Leeman Labs' Prodigy Plus 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.

Specifically, cadmium is toxic to the kidney, lead can have an effect on the brain as well as interfere with intellectual development in children, and arsenic is a carcinogen. 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 Labs' Prodigy Plus 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).<sup>1,2</sup> 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
<b>USEPA (2014)</b>	0.1	1.3	0.01	0.005	0.015
<b>WHO (2011)</b>	0.07	2.0	0.01	0.003	0.01

### Instrument

A Prodigy Plus High Dispersion Inductively Coupled Plasma (ICP) Spectrometer equipped with a dual-view twist-n-lock torch and a Teledyne CETAC ASX-280 120-position autosampler was used to generate the data for this application note.

The Prodigy Plus is a compact bench-top simultaneous ICP-OES system featuring an 800 mm focal length Echelle optical system coupled with a CMOS (Complementary Metal Oxide Semiconductor) detector. At 28 x 28 mm, the active area of the detector is significantly larger than any other solid-state detector currently used for ICP-OES. This combination allows the Prodigy Plus to achieve significantly higher optical dispersion 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 Prodigy Plus uses a 40.68 MHz free-running, water-cooled oscillator, allowing it to handle the most difficult sample matrices. A high-sensitivity sample introduction system ensures that sufficient and steady emission signals are transmitted to the spectrometer.

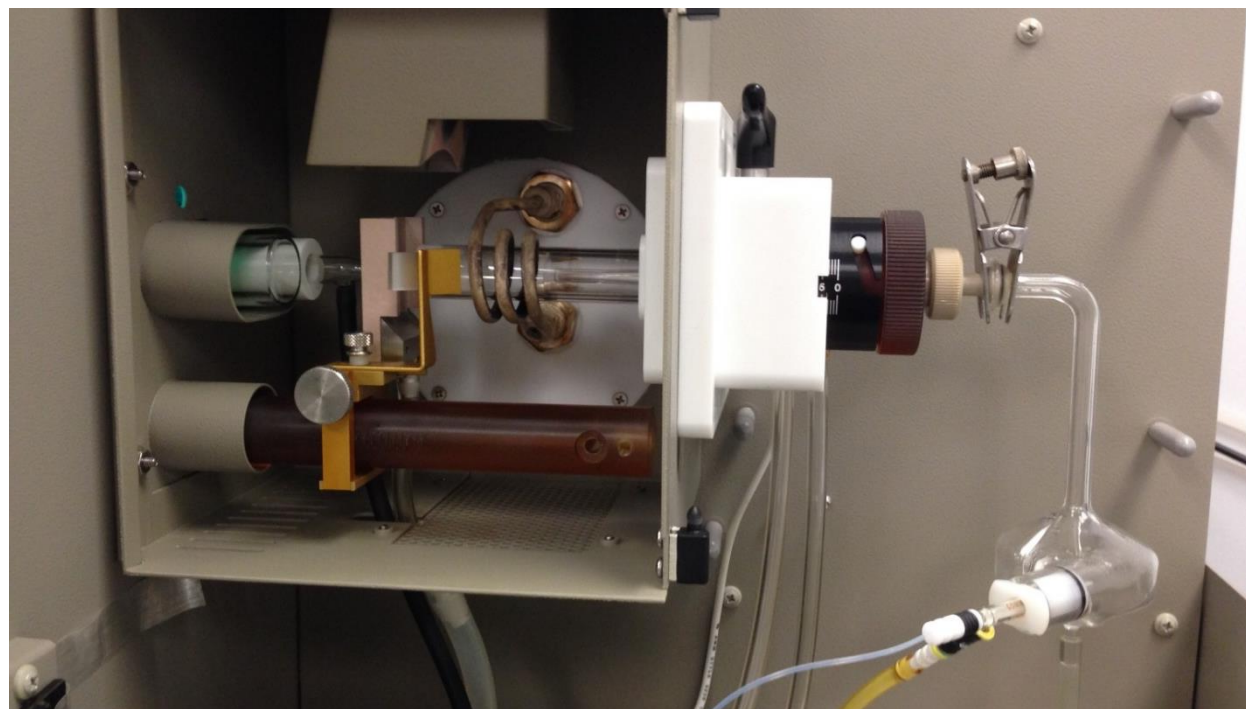
### Sample Introduction

The sample introduction configuration used for this application note is listed in [Table II](#) and shown in [Figure 1](#).

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 Prodigy Plus'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



### 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	35 PSI
Uptake Rate	25 rpm
Integration Time:	
Axial	30 seconds
Radial	15 seconds

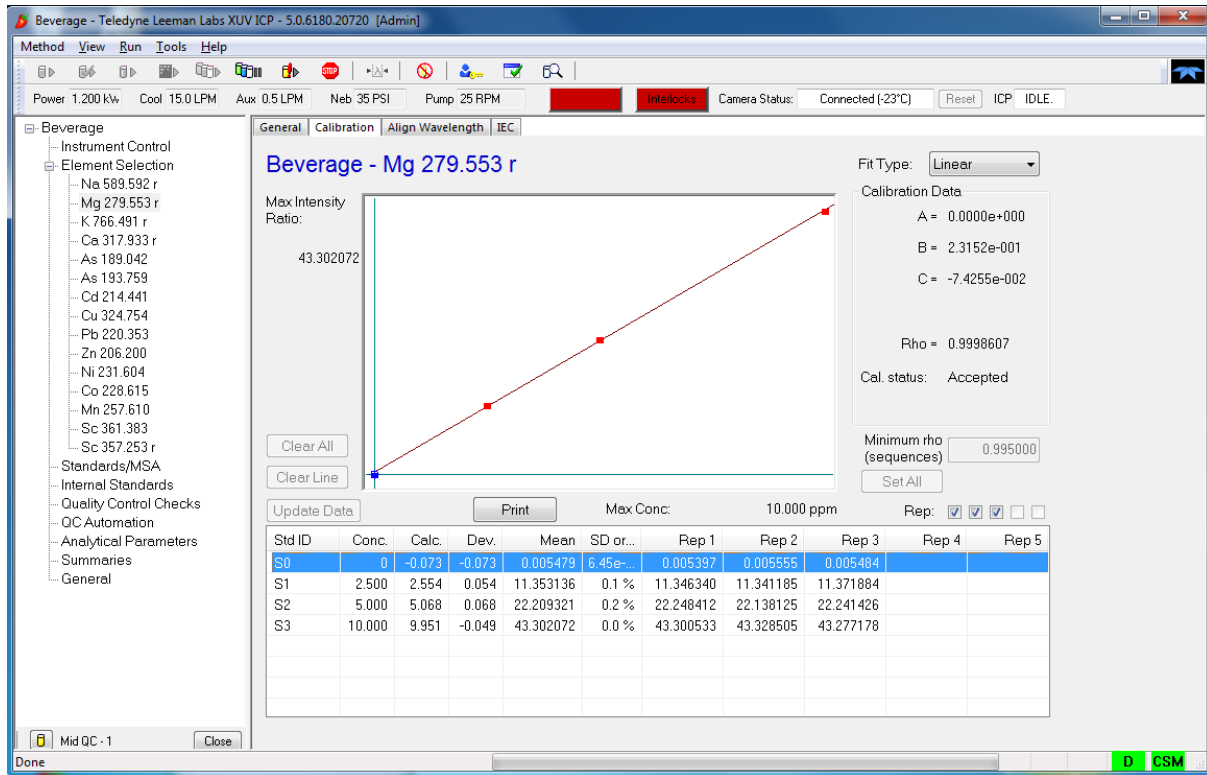
### Calibration Standards

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

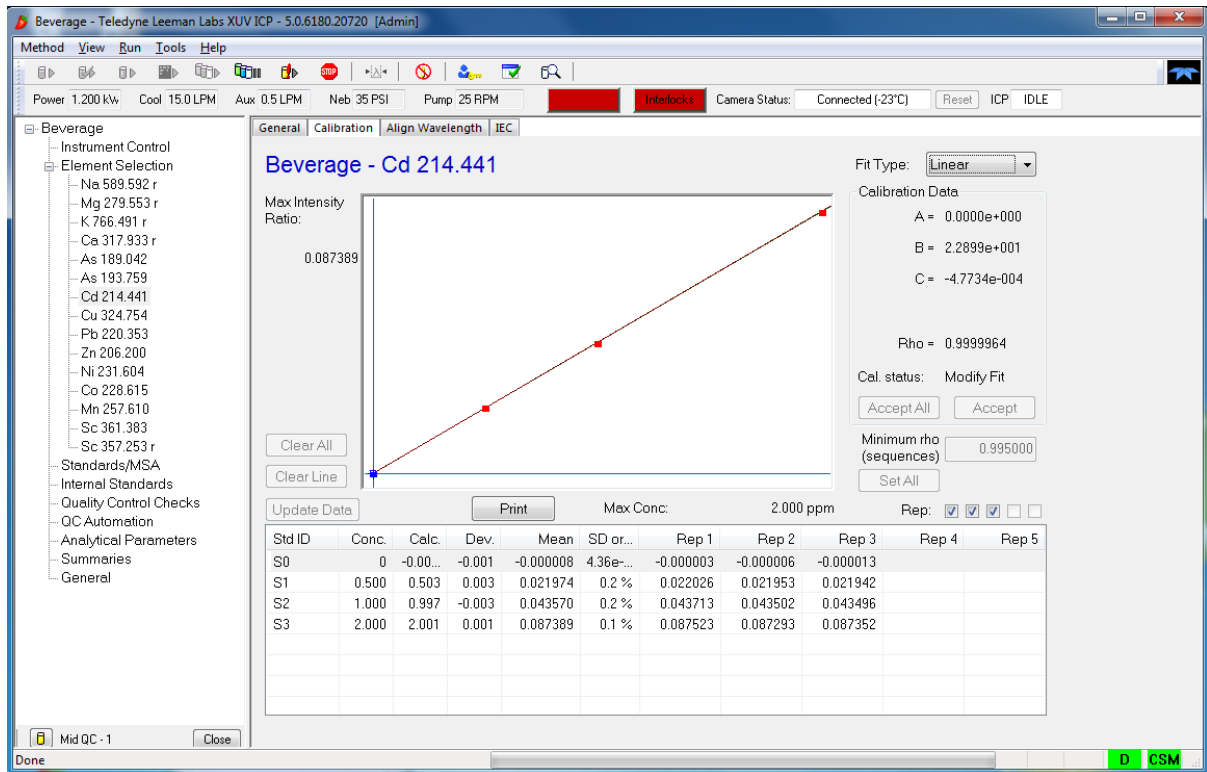
Table IV Calibration Standards, ppm					
Element	Wavelength, nm	Blank	Std 1	Std 2	Std 3
Na	589.592 r	0	2.5	5.0	10.0
Mg	279.553 r	0	2.5	5.0	10.0
K	766.491 r	0	2.5	5.0	10.0
Ca	317.933 r	0	2.5	5.0	10.0
As	189.042	0	0.5	1.0	2.0
Cd	214.441	0	0.5	1.0	2.0
Cu	324.754	0	0.5	1.0	2.0
Pb	220.353	0	0.5	1.0	2.0
Zn	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



**Figure 3** Calibration Curve of Cd at 214.441 nm



## Sample Preparation

Three different beverages were analyzed: 100% apple juice, sports drink and orange juice. Samples were prepared by weighing 20 g of each sample into 100 mL volumetric flasks for heavy metal content, and 1.0, 0.5 or 0.20 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 duplicate. 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		
Element	Wavelength (nm)	DL (ppm)
Na	589.592 r	0.004
Mg	279.553 r	0.0001
K	766.491 r	0.014
Ca	317.933 r	0.001
As	189.042	0.006
Cd	214.441	0.0001
Cu	324.754	0.0001
Pb	220.353	0.001
Zn	206.200	0.0002
Ni	231.604	0.001
Co	228.615	0.001
Mn	257.610	0.00003

## Results

After igniting the plasma and allowing a 15-minute warm-up period, the Prodigy Plus was calibrated. Once the calibration was complete, a QC standard was analyzed with acceptance criteria of  $\pm 10\%$ .

Results from the sample analysis are shown in [Table VI](#), [Table VII](#) and [Table VIII](#). 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 VI Apple Juice Results					
Element	Wavelength (nm)	Dilution factor	Avg. Conc (ppm)	% Spike Recovery	% RSD
Na	589.592 r	5	7.13	103.4	0.4
Mg	279.553 r	5	40.5	103.5	0.3
K	766.491 r	200	1000	102.7	1.0
Ca	317.933 r	5	25.2	107.8	0.5
As	189.042	5	ND	109.4	1.2
Cd	214.441	5	ND	107.4	0.4
Cu	324.754	5	0.03	102.3	0.4
Pb	220.353	5	ND	101.7	0.3
Zn	206.200	5	0.11	110.6	0.5
Ni	231.604	5	ND	106.1	0.5
Co	228.615	5	ND	106.4	0.5
Mn	257.610	5	0.97	106.7	0.4

Table VII Sports Drink Results					
Element	Wavelength (nm)	Dilution Factor	Avg. Conc (ppm)	% Spike Recovery	% RSD
Na	589.592 r	100	430	84.3	0.1
Mg	279.553 r	5	4.67	98.2	0.1
K	766.491 r	100	140	87.9	0.5
Ca	317.933 r	5	12.3	98.5	0.1
As	189.042	5	ND	99.7	0.5
Cd	214.441	5	ND	102.9	0.5
Cu	324.754	5	ND	97.1	0.3
Pb	220.353	5	ND	99.3	0.1
Zn	206.200	5	ND	96.3	0.4
Ni	231.604	5	ND	97.6	0.4
Co	228.615	5	ND	97.1	0.3
Mn	257.610	5	ND	97.5	0.3

Table VIII Orange Juice Results					
Element	Wavelength (nm)	Dilution Factor	Avg. Conc (ppm)	% Spike Recovery	% RSD
Na	589.592 r	5	8.38	111.0	0.1
Mg	279.553 r	200	102	100.3	0.2
K	766.491 r	500	1822	99.2	0.3
Ca	317.933 r	200	106	100.2	0.3
As	189.042	5	0.31	116.3	0.3
Cd	214.441	5	0.05	115.0	0.2
Cu	324.754	5	0.42	112.3	0.1
Pb	220.353	5	0.04	108.7	0.2
Zn	206.200	5	0.36	117.4	0.2
Ni	231.604	5	0.01	113.7	0.2
Co	228.615	5	ND	114.6	0.2
Mn	257.610	5	0.33	114.9	0.7

## Conclusions

The analysis of beverages was successfully performed using the Teledyne Leeman Labs' Prodigy Plus ICP-OES. The spike recovery data shows that all the analytes were measured within  $\pm 20\%$  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 Prodigy Plus 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 Orange juice, 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/MCLsEPAsDWP-2014-07-01.pdf](http://www.waterboards.ca.gov/drinking_water/certlic/drinkingwater/documents/dwdocuments/MCLsEPAsDWP-2014-07-01.pdf) (accessed November 13, 2017)
2. World Health Organization. *Guidelines for Drinking-water Quality - 4<sup>th</sup> Ed.*; Geneva, Switzerland, 2011. [Online] [http://apps.who.int/iris/bitstream/10665/44584/1/9789241548151\\_eng.pdf](http://apps.who.int/iris/bitstream/10665/44584/1/9789241548151_eng.pdf) (accessed November 13, 2017)