# Determination of Heavy Metals in Reagent Chemicals Using the Teledyne Leeman Lab's Prodigy7 ICP-OES Following Procedures Developed by the ACS Committee

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## Introduction

The American Chemical Society (ACS) Committee mandates the specifications for most chemicals used in analytical testing, and is the only organization in the world that sets requirements and develops validated methods for determining the purity of reagent chemicals.

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 determine ultra-trace elemental levels in selected reagent-grade chemicals. The obtained results were compared with specifications set by ACS and presented in Table I. [1]

**Table I ACS Specifications**

| Reagent Chemical | Sample Weight/Final Volume (g/mL) | Maximum Allowable Concentration (ppm) of Heavy Metals[A] |
|---|---|---|
| KOH | 1.0/100 | 10 |
| HCl | 10.0/100 | 1 |
| $Na_2SO_4$ | 2.0/100 | 5 |
| $CaCO_3$ | 1.0/100 | 10 |
| NaCl | 2.0/100 | 5 |

A. Heavy Metals – The summation of Ag, As, Bi, Cd, Cu, Hg, Mo, Pb, Sb and Sn.

## 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

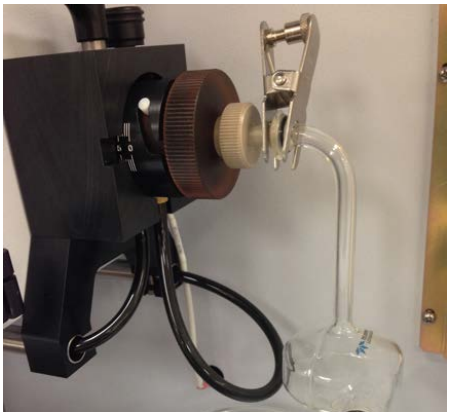
The sample introduction configuration used for this study is shown in Table II.

| Table II Sample Introduction Configuration | |
|---|---|
| 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) |

Yttrium (Y) (371.030 nm) 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 allowing 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 | 13 L/min |
| Auxiliary Flow | 0.5 L/min |
| Nebulizer Pressure | 0.9 L/min |
| Uptake Rate | 25 rpm |
| Integration Time:<br>Axial | <br>30 seconds |

## Calibration Standards

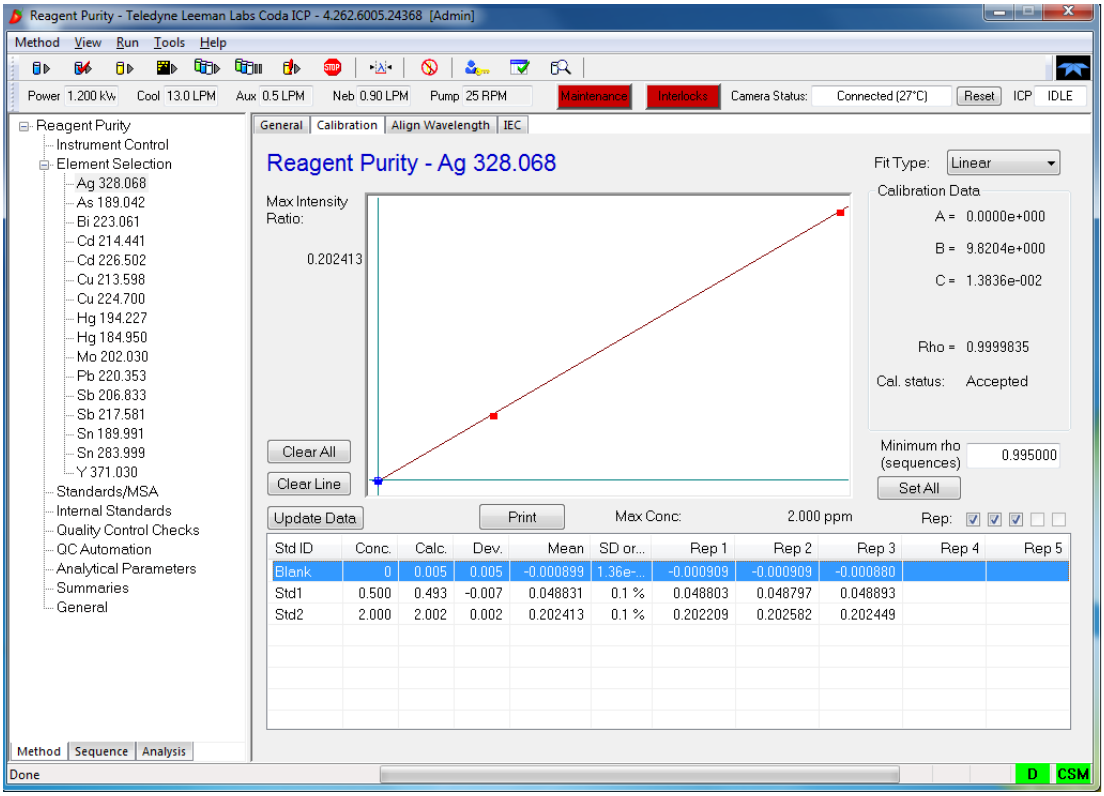
Calibration standards were prepared from single-element stock solutions (VHG Labs® Standards, Manchester, NH). Standards were prepared on the day of analysis in 5% nitric acid. The final concentrations are listed in Table IV.

**Table IV Calibration Standards, ppm**

| Element | Wavelength, nm | Blank | STD1 | STD2 | STD3 | STD4 |
|---|---|---|---|---|---|---|
| Ag | 328.068 | 0 | 0.5 | 2.0 | - | - |
| As | 189.042 | 0 | - | - | 0.5 | 2.0 |
| Bi | 223.061 | 0 | - | - | 0.5 | 2.0 |
| Cd | 214.441 | 0 | - | - | 0.5 | 2.0 |
| Cu | 213.598 | 0 | - | - | 0.5 | 2.0 |
| Hg | 194.227 | 0 | - | - | 0.5 | 2.0 |
| Mo | 202.030 | 0 | - | - | 0.5 | 2.0 |
| Pb | 220.353 | 0 | - | - | 0.5 | 2.0 |
| Sb | 217.581 | 0 | - | - | 0.5 | 2.0 |
| Sn | 189.991 | 0 | - | - | 0.5 | 2.0 |

An example calibration curve is shown in Figure 2. The curve is based on the calibration of Silver (Ag) at 328.068 nm in axial-view mode.

**Figure 2** Calibration Curve of Ag at 328.068 nm



## Sample Preparation

Five reagent chemicals were analyzed: KOH, HCl, $Na_2SO_4$, $CaCO_3$ and NaCl. In order to achieve the specifications, selected reagents were diluted with deionized water, acidified with 5% nitric acid, made up to volume, and aspirated directly into the instrument's sample introduction system. 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) for the elements of interest. Detection limits were determined as concentrations corresponding to three times the standard deviation of 10 consecutive measurements of the calibration blank.

**Table V Instrument Detection Limits (IDL)**

| Element | Wavelength (nm) | DL (ppm) |
|---|---|---|
| Ag | 328.068 | 0.0005 |
| As | 189.042 | 0.0055 |
| Bi | 223.061 | 0.0024 |
| Cd | 214.441 | 0.0001 |
| Cu | 213.598 | 0.0005 |
| Hg | 194.227 | 0.0017 |
| Mo | 202.030 | 0.0003 |
| Pb | 220.353 | 0.0015 |
| Sb | 217.581 | 0.0038 |
| Sn | 189.991 | 0.0019 |

## 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 an acceptance criteria of ±10%. QC standard results are presented 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 % |
|---|---|---|---|---|---|
| Ag | 328.068 | 0.5 | 0.49 | 99.1 | 0.0 |
| As | 189.042 | 0.5 | 0.47 | 94.4 | 0.3 |
| Bi | 223.061 | 0.5 | 0.48 | 96.5 | 0.5 |
| Cd | 214.441 | 0.5 | 0.48 | 96.9 | 0.2 |
| Cu | 213.598 | 0.5 | 0.48 | 96.2 | 0.9 |
| Hg | 194.227 | 0.5 | 0.48 | 96.4 | 0.2 |
| Mo | 202.030 | 0.5 | 0.48 | 96.8 | 0.5 |
| Pb | 220.353 | 0.5 | 0.48 | 96.0 | 1.5 |
| Sb | 217.581 | 0.5 | 0.47 | 95.1 | 0.5 |
| Sn | 189.991 | 0.5 | 0.48 | 95.8 | 0.6 |

Results from the analysis are shown in Table VII, Table VIII, Table IX, Table X and Table XI. Results for each reagent chemical are reported in units of parts per million (ppm) with dilution factors applied. Results are also presented for the recoveries of the 0.2 ppm 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 KOH**

| Element | Wavelength (nm) | Dilution Factor | Avg Conc (ppm) | Spike Recovery % | RSD % |
|---|---|---|---|---|---|
| Ag | 328.068 | 100 | 0.42 | 102.9 | 0.1 |
| As | 189.042 | 100 | 0.65 | 120.1 | 3.2 |
| Bi | 223.061 | 100 | 0.96 | 90.9 | 1.1 |
| Cd | 214.441 | 100 | 0.76 | 107.8 | 0.0 |
| Cu | 213.598 | 100 | 0.68 | 97.2 | 0.3 |
| Hg | 194.227 | 100 | 0.12 | 113.2 | 0.4 |
| Mo | 202.030 | 100 | 1.12 | 104.9 | 0.3 |
| Pb | 220.353 | 100 | 0.80 | 92.9 | 0.7 |
| Sb | 217.581 | 100 | 1.57 | 106.4 | 1.1 |
| Sn | 189.991 | 100 | 1.41 | 103.1 | 0.1 |
| Maximum Allowable = 10 ppm; The summation of all concentrations = 8.5 ppm | | | | | |

**Table VIII HCl**

| Element | Wavelength (nm) | Dilution Factor | Avg Conc (ppm) | Spike Recovery % | RSD % |
|---|---|---|---|---|---|
| Ag | 328.068 | 10 | 0.05 | 99.8 | 0.1 |
| As | 189.042 | 10 | 0.07 | 97.3 | 0.6 |
| Bi | 223.061 | 10 | 0.11 | 92.6 | 0.6 |
| Cd | 214.441 | 10 | 0.08 | 97.3 | 0.1 |
| Cu | 213.598 | 10 | 0.08 | 94.6 | 0.2 |
| Hg | 194.227 | 10 | 0.08 | 95.0 | 0.3 |
| Mo | 202.030 | 10 | 0.11 | 95.4 | 0.0 |
| Pb | 220.353 | 10 | 0.10 | 94.6 | 0.2 |
| Sb | 217.581 | 10 | 0.15 | 95.4 | 0.3 |
| Sn | 189.991 | 10 | 0.11 | 95.9 | 0.4 |
| Maximum Allowable = 1 ppm; The summation of all concentrations = 0.9 ppm | | | | | |

| Table IX $Na_2SO_4$ | | | | | |
|---|---|---|---|---|---|
| **Element** | **Wavelength (nm)** | **Dilution Factor** | **Avg Conc (ppm)** | **Spike Recovery %** | **RSD %** |
| Ag | 328.068 | 50 | 0.27 | 102.1 | 0.2 |
| As | 189.042 | 50 | ND | 117.1 | 2.8 |
| Bi | 223.061 | 50 | 0.55 | 89.7 | 0.6 |
| Cd | 214.441 | 50 | 0.37 | 103.1 | 0.2 |
| Cu | 213.598 | 50 | 0.29 | 93.3 | 0.3 |
| Hg | 194.227 | 50 | 0.26 | 111.4 | 0.5 |
| Mo | 202.030 | 50 | 0.56 | 103.0 | 0.2 |
| Pb | 220.353 | 50 | 0.48 | 88.6 | 0.4 |
| Sb | 217.581 | 50 | 0.44 | 108.6 | 1.0 |
| Sn | 189.991 | 50 | 0.57 | 99.8 | 0.2 |
| Maximum Allowable = 5 ppm; The summation of all concentrations = 3.8 ppm | | | | | |

| Table X $CaCO_3$ | | | | | |
|---|---|---|---|---|---|
| **Element** | **Wavelength (nm)** | **Dilution Factor** | **Avg Conc (ppm)** | **Spike Recovery %** | **RSD %** |
| Ag | 328.068 | 100 | 1.94 | 93.5 | 0.2 |
| As | 189.042 | 100 | ND | 120.0 | 2.5 |
| Bi | 223.061 | 100 | ND | 98.2 | 1.0 |
| Cd | 214.441 | 100 | 0.38 | 107.1 | 0.4 |
| Cu | 213.598 | 100 | 0.19 | 94.4 | 0.5 |
| Hg | 194.227 | 100 | ND | 122.0 | 1.0 |
| Mo | 202.030 | 100 | ND | 110.7 | 0.5 |
| Pb | 220.353 | 100 | 1.13 | 96.1 | 1.0 |
| Sb | 217.581 | 100 | ND | 118.3 | 1.8 |
| Sn | 189.991 | 100 | 1.72 | 106.6 | 0.7 |
| Maximum Allowable = 10 ppm; The summation of all concentrations = 5.4 ppm | | | | | |

| Table XI NaCl | | | | | |
|---|---|---|---|---|---|
| **Element** | **Wavelength (nm)** | **Dilution Factor** | **Avg Conc (ppm)** | **Spike Recovery %** | **RSD %** |
| Ag | 328.068 | 50 | 1.00 | 95.4 | 0.2 |
| As | 189.042 | 50 | ND | 119.6 | 1.6 |
| Bi | 223.061 | 50 | 0.35 | 96.6 | 0.6 |
| Cd | 214.441 | 50 | 0.44 | 106.0 | 0.1 |
| Cu | 213.598 | 50 | 0.37 | 95.3 | 0.0 |
| Hg | 194.227 | 50 | 0.54 | 108.0 | 0.5 |
| Mo | 202.030 | 50 | 0.56 | 107.9 | 0.3 |
| Pb | 220.353 | 50 | 0.46 | 92.3 | 0.7 |
| Sb | 217.581 | 50 | 0.49 | 112.9 | 0.6 |
| Sn | 189.991 | 50 | 0.84 | 102.7 | 0.1 |
| Maximum Allowable = 5 ppm; The summation of all concentrations = 5.0 ppm | | | | | |

## Conclusions

The analysis of reagent-grade chemicals was successfully performed using the Teledyne Leeman Labs Prodigy7 ICP-OES. The spike recovery data indicates that all analytes were measured within ±25% of the acceptable spike recovery limits. 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 the results and the simplicity of the sample treatment, the Prodigy7 ICP-OES quickly and accurately measured the ultra-trace elemental levels in selected reagent-grade chemicals.

The reported summation of all concentrations of targeted elements did not exceed the specifications given by the American Chemical Society (ACS) for heavy metals by ICP-OES.

## References

1. American Chemical Society, Committee on Analytical Reagents. Paul A. Bouis, Chair. *Reagent Chemicals: Specifications and Procedures for Reagents and Standard-Grade Reference Materials.* 11th Ed.; Washington, DC

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