TELEDYNE LEEMAN LABS
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APPLICATION NOTE:	AN1805
TECHNOLOGY:	ICP
INDUSTRY:	Mining

Analysis of Impurities in 24 Karat (99.9%) Gold Using the Teledyne Leeman Labs' Prodigy Plus ICP-OES

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Introduction

Gold is a bright, reddish-yellow, dense, soft and malleable metal. Normally found pure in nuggets or as grains in rocks and alluvial deposits, it can also be found alloyed with silver, copper or palladium and occasionally as gold compounds in minerals. While gold is resistant to most acids, it can be dissolved in aqua regia, as well as alkaline solutions of cyanide, both of which are used in mining and electroplating industries.

Due to gold's resistance to corrosion and electrical conductivity, gold's primary industrial application is for electrical connectors, but it can also be used for infrared shielding, colored glass production and dental applications. Approximately 10% of the total gold mined is used for industrial purposes, while the remainder is used for investment and jewelry.

This application note will demonstrate the ability of the Teledyne Leeman Labs' Prodigy Plus ICP spectrometer to analyze 24 karat (99.9%) gold for impurities.

Instrument

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

Figure 1 Prodigy Plus ICP-OES

Figure 2 Teledyne CETAC ASX-280 Autosampler

The Prodigy Plus is a compact benchtop simultaneous ICP-OES system featuring an 800 mm focal length Echelle optical system coupled with a mega-pixel Large Format CMOS (L-CMOS) detector. At 28 x 28 mm, the active area of the L-CMOS is significantly larger than any other solid-state detector currently used for ICP-OES. This combination allows the Prodigy Plus 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 Prodigy Plus uses a 40.68 MHz rugged, free-running RF Generator, allowing it to handle the most difficult sample matrices, as well as common organic solvents.

Sample Introduction

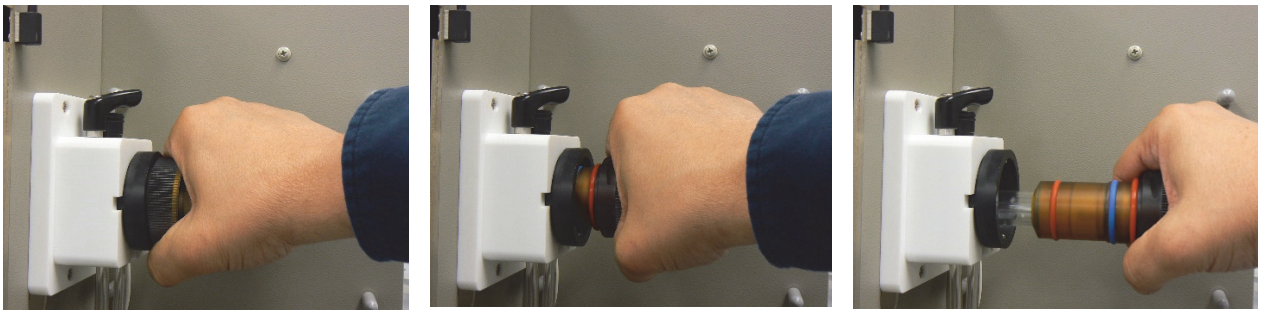
A high-sensitivity sample introduction system ensures that sufficient and steady emission signals are transmitted to the spectrometer. The sample introduction configuration used for this application note is shown in Table I.

Table I Sample Introduction

Component	Description	Part Number
Nebulizer	Glass Conikal	PN 120-00463-1
Spray Chamber	Glass Cyclonic with No Center Knockout Tube	PN 120-00461-2
Sample Uptake Tubing	0.76 mm	PN 309-00069-7
Sample Drain Tubing	1.14 mm	PN 309-00069-4

The Prodigy Plus's torch is mounted using an innovative twist-n-lock cassette system shown in Figure 3. The design permits operators to remove and replace the torch to the exact same position, providing day-to-day reproducibility and simplified training. Additionally, the twist-lock design automatically connects the coolant and auxiliary gas flows, eliminating potential errors.

Figure 3 Twist-n-Lock Sample Introduction



Method

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 II.

Table II Instrument Operating Conditions

Parameter	Value	Part Number
RF Power	1.20 kW	
Coolant Flow	16.0 LPM	
Auxiliary Flow	0.2 LPM	
Nebulizer Pressure	31 PSI	
Pump Rate	25 RPM	
Torch	Quartz Demountable	PN 318-00167-1
Injector	2.5 mm	PN 318-00161-AQ1
Integration Time	Axial 60 sec	

Instrument Detection Limits

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

Table III Instrument Detection Limits

Element	Wavelength (nm)	DL (ppm)
Ag	328.068	0.0004
As	189.042	0.0076
Bi	223.061	0.0060
Cd	226.502	0.0005
Co	228.615	0.0009
Cr	206.149	0.0008
Cu	224.700	0.0011
Fe	239.563	0.0003
Mn	257.610	0.0001
Ni	221.648	0.0006
Pb	220.353	0.0020
Pd	340.458	0.0008
Pt	265.945	0.0012
Rh	343.489	0.0016
Sb	206.833	0.0077
Se	196.090	0.0082
Sn	189.991	0.0030
Te	214.281	0.0070
Ti	337.280	0.0004
Zn	202.548	0.0003

Sample Preparation

Three gold reference samples were obtained from the Rand Refinery, South Africa for analysis. The samples were dissolved in 10% aqua regia and brought to 100 ml in volumetric flasks.

Calibration Standards

Calibration Standards were prepared from single-element stock solutions (VHG Labs® Standards, Manchester, NH). The final concentrations are listed in Table IV. The standards were not matrix matched to the samples.

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Table IV Calibration Standards, ppm					
Element	**Wavelength, nm**	**Blank**	**Std1**	**Std2**	**Std3**
Ag	328.068	0	0.5	1	2
As	189.042	0	0.5	1	2
Bi	223.061	0	0.5	1	2
Cd	226.502	0	0.5	1	2
Co	228.615	0	0.5	1	2
Cr	206.149	0	0.5	1	2
Cu	224.700	0	0.5	1	2
Fe	239.563	0	0.5	1	2
Mn	257.610	0	0.5	1	2
Ni	221.648	0	0.5	1	2
Pb	220.353	0	0.5	1	2
Pd	340.458	0	0.5	1	2
Pt	265.945	0	0.5	1	2
Rh	343.489	0	0.5	1	2
Sb	206.833	0	0.5	1	2
Se	196.090	0	0.5	1	2
Sn	189.991	0	0.5	1	2
Te	214.281	0	0.5	1	2
Ti	337.280	0	0.5	1	2
Zn	202.548	0	0.5	1	2

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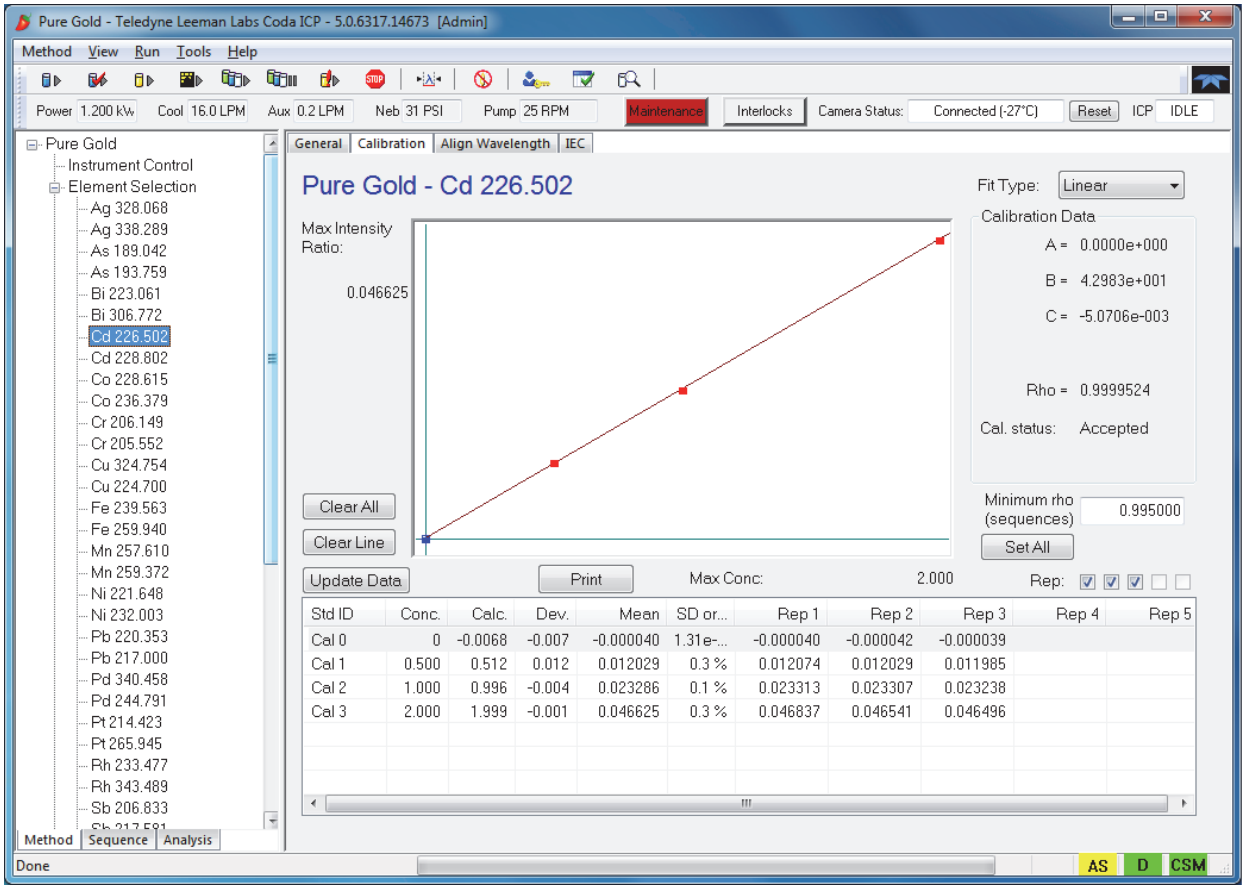
An example calibration curve is shown in Figure 4. The curve was based on the calibration of Cd at 226.502 nm in axial-view mode.

Figure 4 Calibration Curve of Cd at 226.502nm



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 ±10%.

Results from the sample analysis are shown in Table V, Table IV and Table VII. Results for each sample are reported in units of parts per million (mg/Kg), along with standard deviation of the analysis, the certified value and % recovery. Results are listed as Not Detected (ND), if the measured concentration was at or below the IDL.

Table V RAuHP1					
Element	Wavelength (nm)	Result (mg/Kg)	Std Dev	Certified Value (mg/Kg)	% Recovery
Ag	328.068	125.7	5.98	116.6	107.8
As	189.042	104.3	0.67	99.2	105.1
Bi	223.061	83.7	0.74	81.4	102.8
Cd	226.502	96.5	0.06	99.2	97.2
Co	228.615	101.6	0.31	103.7	98.0
Cr	206.149	107.6	0.06	100.6	106.9
Cu	224.700	126.3	0.15	112.0	112.8
Fe	239.563	126.4	0.20	121.7	103.8
Mn	257.610	110.9	1.21	103.3	107.4
Ni	221.648	97.7	0.15	92.2	105.9
Pb	220.353	107.6	0.50	116.3	92.5
Pd	340.458	103.3	0.82	95.5	108.2
Pt	265.945	98.9	0.10	89.7	110.3
Rh	343.489	97.0	0.87	91.4	106.1
Sb	206.833	92.4	1.07	87.7	105.4
Se	196.090	91.3	1.65	90.2	101.3
Sn	189.991	92.8	0.15	85.7	108.2
Te	214.281	152.7	0.85	161.7	94.4
Ti	337.280	81.6	0.06	75.1	108.7
Zn	202.548	92.3	0.31	87.2	105.9

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Table VI RAuP2					
Element	Wavelength (nm)	Result (mg/Kg)	Std Dev	Certified Value (mg/Kg)	% Recovery
Ag	328.068	30.5	0.25	30.4	100.4
As	189.042	12.4	0.12	11.4	109.0
Bi	223.061	12.2	0.40	10.7	114.0
Cd	226.502	10.9	0.06	11.4	95.9
Co	228.615	12.9	0.10	12.4	104.0
Cr	206.149	12.4	0.00	10.9	113.6
Cu	224.700	36.7	0.10	34.7	105.8
Fe	239.563	29.8	0.06	27.2	109.4
Mn	257.610	13.0	0.25	12.5	104.2
Ni	221.648	7.7	0.10	6.4	120.3
Pb	220.353	15.7	0.15	15.3	102.4
Pd	340.458	12.2	0.12	11.2	108.6
Pt	265.945	12.0	0.06	11.2	106.8
Rh	343.489	12.5	0.12	11.0	113.9
Sb	206.833	11.3	0.40	12.5	90.8
Se	196.090	10.3	0.78	9.5	108.7
Sn	189.991	11.7	0.06	10.8	108.6
Te	214.281	22.3	1.16	21.9	102.0
Ti	337.280	7.9	0.06	7.5	105.8
Zn	202.548	18.2	0.10	16.2	112.3

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Table VII RAuP5					
Element	Wavelength (nm)	Result (mg/Kg)	Std Dev	Certified Value (mg/Kg)	% Recovery
Ag	328.068	16.8	0.02	16.5	101.9
As	189.042	ND	NA	<1	
Bi	223.061	3.9	0.30	<1	
Cd	226.502	0.2	0.02	<1	
Co	228.615	0.6	0.05	<1	
Cr	206.149	ND	NA	<1	
Cu	224.700	21.0	0.05	20	104.9
Fe	239.563	9.6	0.002	8.9	107.9
Mn	257.610	1.1	0.001	<1	
Ni	221.648	0.5	0.04	<1	
Pb	220.353	3.1	0.10	<1	
Pd	340.458	ND	NA	<1	
Pt	265.945	ND	NA	<1	
Rh	343.489	ND	NA	<1	
Sb	206.833	ND	NA	<1	
Se	196.090	ND	NA	<2	
Sn	189.991	ND	NA	<1	
Te	214.281	2.6	0.01	<1	
Ti	337.280	ND	NA	<1	
Zn	202.548	3.5	0.11	3.3	106.1

Conclusions

The analysis of pure gold for 20 elements was successful using a Teledyne Leeman Labs' axial-view Prodigy Plus High-Dispersion ICP. Accurate results were obtained for all samples. The image stabilized plasma and the simultaneous data collection of both peak and background data combine to provide exceptionally precise and stable results.

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