RADOM™

Reimagine Plasma

MICAP-OES 1000

DETERMINATION OF GOLD AND PLATINUM GROUP METALS IN GEOLOGICAL CERTIFIED REFERENCE MATERIALS



- Lowest Operating Cost
- Simultaneous Measurement
- Lowest Carbon Footprint
- Smallest Laboratory Footprint

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Introduction

The mining industry utilizes Certified Reference Materials as Quality Control samples to verify real geological sample analysis in both preparation and instrumental analysis. The laboratory can confidently report results of mining samples knowing that the correct values are used for mining and processing decisions.

This application brief will outline the process of preparing and analyzing gold prill samples generated by the Fire Assay process for gold (Au), palladium (Pd) and platinum (Pt) in several CRMs made from real geological samples. CRMs are displayed in Table 1.

CRM	Company	Source
CDN-GS-1AB	CDN RESOURCE LABORATORIES, 102 Avenue, Langley, BC, Canada	Standard CDN-GS-1AB was prepared from material that became available to CDN Resource Laboratories from Nevada Gold Mines, Turquoise Ridge, Nevada.
G319-9	GEOSTATS PTY LTD, 20 Hines Road, O'Connor, Western Australia 6163	This material is described as a Very high-grade low sulfide ore.
OREAS L12	OREAS 37A Hosie Street, Bayswater North, VIC 3153, AUSTRALIA	OREAS L12 was prepared from a blend of high-grade gold-bearing ores and barren metasediments and basalt.
OxC168	ROCK LABS 630, Kaikorai Valley Road, Private Bag 1960, Dunedin 9054 New Zealand	Basalt and feldspar minerals with minor quantities of finely divided gold-containing minerals that have been screened to ensure there is no gold nugget effect.
OxQ115	ROCK LABS 630, Kaikorai Valley Road, Private Bag 1960, Dunedin 9054 New Zealand	Basalt and feldspar minerals with minor quantities of finely divided gold-containing minerals that have been screened to ensure there is no gold nugget effect.
PD05	ROCK LABS 630, Kaikorai Valley Road, Private Bag 1960, Dunedin 9054 New Zealand	Concentrates containing platinum, palladium and gold that have been screened to ensure there is no gold nugget effect and then blended with an appropriate matrix of barren minerals
PK03	ROCK LABS 630, Kaikorai Valley Road, Private Bag 1960, Dunedin 9054 New Zealand	Concentrates containing platinum, palladium and gold that have been screened to ensure there is no gold nugget effect and then blended with an appropriate matrix of barren minerals.

Table 1: CRMs and source.

MICAP-OES

MICAP-OES 1000 is a Microwave Indicatively Coupled Atmospheric Plasma – Optical Emission Spectrometer with 1000W power (MICAP-OES 1000). This innovative nitrogen-based plasma source replaces the traditional argon generated plasma technology. This is only possible by incorporating Radom's CERAWAVE™ technology which replaces the metallic water-cooled coils found in commercially available ICP-OES instruments today.



The plasma source is coupled to an echelle based, research-grade spectrometer via a fiber optic connection. The plasma is viewed axially with auto-optimized plasma tail removal. The optimized viewing position is ensured with the torch alignment system. The entire echelle spectrum (194nm to 625nm + 766nm) is simultaneously captured with each replicate measurement.

MICAP-OES 1000 is a powerful instrumental technique for metal analysis. This instrumentation provides fast, simultaneous sample measurements eliminating multiple preparation steps. In addition, the analysis is performed using instrumentation with low carbon footprint by eliminating the need for combustible gas, sustained usage of argon gas and chillers. The benefits are a low- cost, ease of use analysis while reducing chemical waste and carbon emissions.

Experimental Design

CRM prill samples were received from a commercial mining laboratory consisting of 10-30 mg ingots for each sample, the result of the fire assay starting with ~30g of sample. 1 mL of 50% nitric acid was added to the vials and heated in an SCP Science DigiPrep Jr Hotblock set to 90°C for 15 minutes. The samples were then cooled, 2 mL of concentrated hydrochloric acid were added, and subsequently heated for an additional 15 minutes and then cooled.. The digested test solutions were allowed to cool to room temperature and diluted to 10mL with Type I DI water.

Standard Preparation

The rinse solution and the standard diluent were prepared as 20% HCl and 5% HNO3. The working standards containing gold (Au), palladium (Pd) and platinum (Pt) were prepared with the concentrations shown in Table 2.

MICAP-OES 1000 Sample Introduction Assembly utilized in this work is summarized in Table 3.

Blank	Standard 1
0.050	Standard 2
0.500	Standard 3
5.00	Standard 4
50.0	Standard 5

Table 2. Working Standards.

Component	Description
Torch	Radom 1.5 mm invjector
Spray Chamber	Single pass cyclonic
Nebulizer	High efficiency polymeric nebulizer
Sample Tubing	White/White PVC Flex (1.02mm ID)
Drain Tubing	Blue/Yellow PVC Flex (1.52mm ID)

Table 3. MICAP-OES 1000 Sample Introduction Assembly.

The Plasma and Measurement Conditions are presented in Figure 1. The total sample measurement time is 2 minutes and 45 seconds.

Instrument Parameters	Sampling Parameters		
Coolant:	14	L/min	(8 - 20)
Auxiliary:	0.3	L/min	(0.2 - 2)
Nebulizer:	0.65	L/min	(0 - 2)
Power:	1000	W	(750 - 1000)
Exposure:	3000	ms	(40 - 10000)
Replicates			
# of Exposures:	10		(1 - 100)
# of Repeats:	3		(1 - 15)

Figure 1. Plasma and Measurement Conditions

The sample introduction assembly is pictured in Figure 2. The torch is inserted upward until the pegs hit the base of the torch holder. The plasma will not ignite if it is not inserted completely. An air knife removes the plasma tail resulting in a consistent position for axial viewing of the plasma, reproducible from operator to operator.



Figure 2. Sample Introduction Assembly.

RESULTS AND DISCUSSION

Representative calibration curves with figures of merit for each wavelength are presented in Figure 3. The graphs represent the appropriate concentration range per wavelength. The prepared samples were analyzed on two different MICAPs at two different locations.

Figure 3. Representative Calibration Curves.

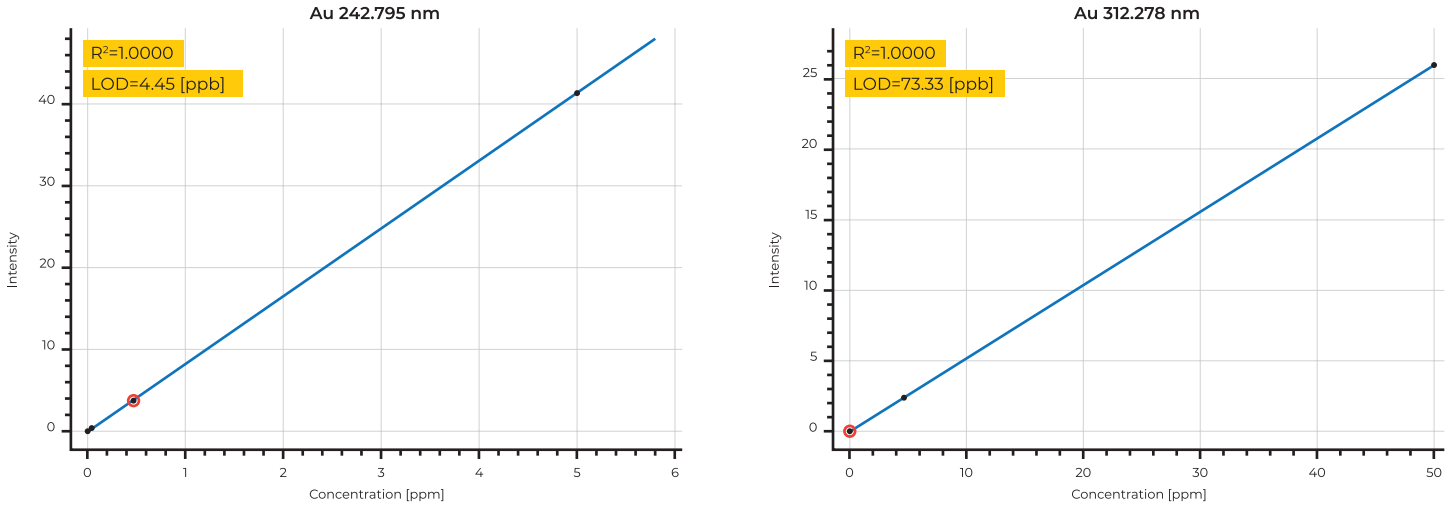
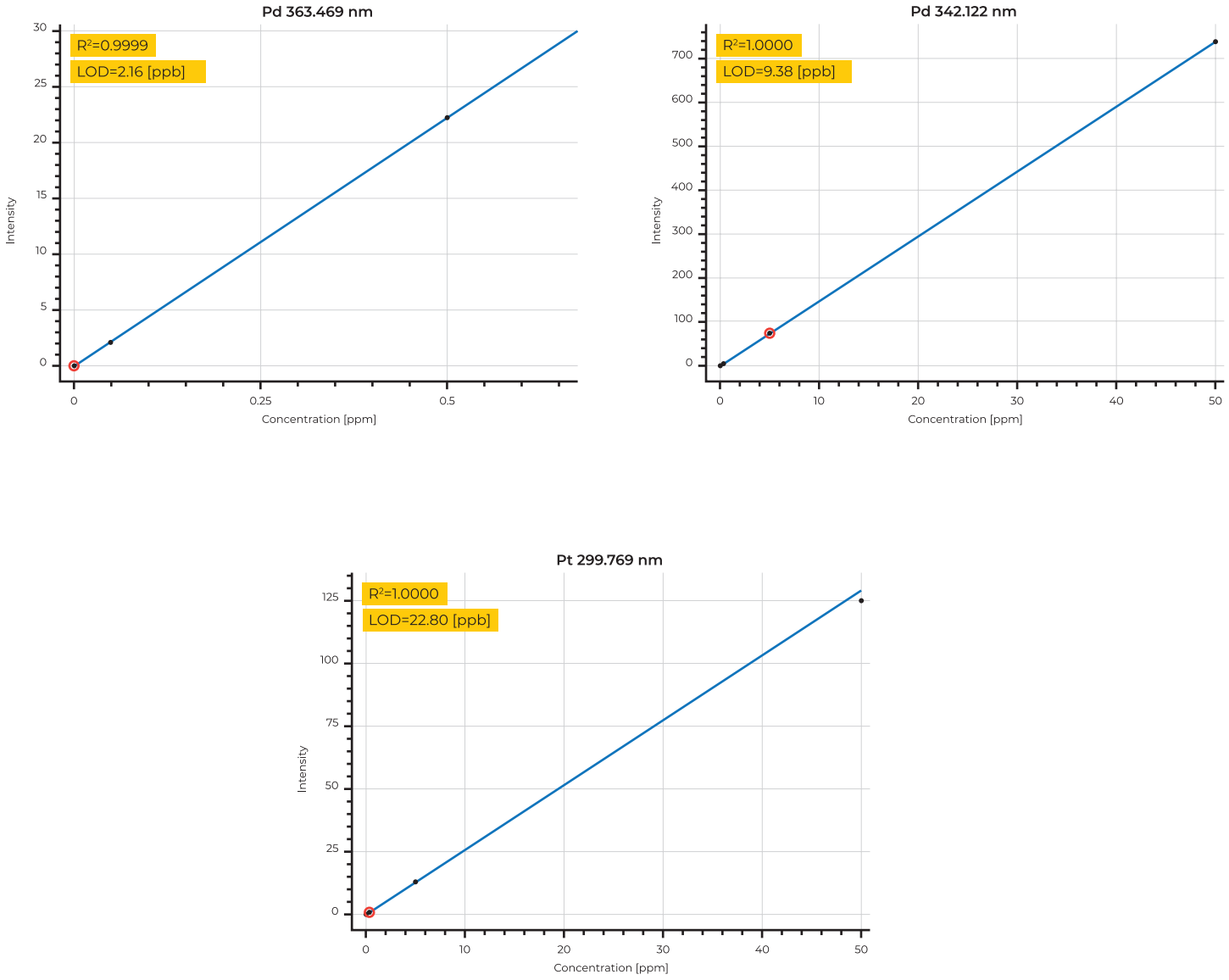


Figure 3. Representative Calibration Curves.



LOD	Au 242.495	Au 312.278	Pd 363.469	Pd 342.122	Pt 299.769
MICAP 1	3.4	73	2.2	9.4	2.3
MICAP 2	4.6	69	7.5	9.7	27

Table 3. LODs calculated by the RIS software on the two instruments.

The prill sample weight and final volume weight were entered into the software. MICAP-OES 1000 is a simultaneous, echelle polychromator with CMOS detector. As a result, multiple wavelengths can be selected with no disadvantage to sample volume consumed or total time of analysis. Two wavelengths were selected for gold and platinum. Cross calibration was used where the more sensitive line is used for low levels and a less sensitive line for higher levels. The results determined for gold and platinum were reported from the primary and secondary wavelengths as an example. Sample results are listed in Tables 3 and 4.

LOD	Au ppm		Certified Value	% Recovery Au		Relative Percent Difference
	MICAP 1	MICAP 2	Au	MICAP 1	MICAP 2	Au
CDN-GS-1AB 1	1,553	1,594	1,477	105%	108%	3%
CDN-GS-1AB 2	1,605	1,638	1,477	109%	111%	2%
CDN-GS-1AB 3	1,636	1,624	1,477	111%	110%	1%
CDN-GS-1AB 4	1,482	1,511	1,477	100%	102%	2%
CDN-GS-1AB 5	1,572	1,535	1,477	106%	104%	2%
CDN-GS-1AB 6	1,581	1,595	1,477	107%	108%	1%
G319-9 1	95,215	95,348	97,320	98%	98%	0%
OREAS L12 1	0,682	0,714	0,615	111%	116%	4%
OREAS L12 2	0,665	0,685	0,615	108%	111%	3%
OREAS L12 3	0,656	0,704	0,615	107%	114%	7%
OREAS L12 4	0,649	0,691	0,615	106%	112%	6%
OREAS L12 5	0,673	0,700	0,615	109%	114%	4%
OxC168 1	0,228	0,249	0,213	107%	117%	9%
OxC168 2	0,221	0,245	0,213	104%	115%	10%
OxC168 3	0,221	0,245	0,213	104%	115%	10%
OxC168 4	0,224	0,245	0,213	105%	115%	9%
OxC168 5	0,232	0,245	0,213	109%	115%	5%
OxQ115 6	24,904	26,078	25,220	99%	103%	5%
PD05	0,547	0,588	0,519	105%	113%	7%
PK03 1	4,976	5,166	5,038	99%	103%	4%
PK03 2	4,952	4,961	5,038	98%	98%	0%
PK03 3	5,121	5,097	5,038	102%	101%	0%
PK03 4	4,737	4,674	5,038	94%	93%	1%
PK03 5	4,756	4,709	5,038	94%	93%	1%
PK03 6	4,646	4,624	5,038	92%	92%	0%

Table 3. Au CRM results.

KEY
% Recovery
90-110%
80-90 and 110-120

CRM	Pd ppm		Pt ppm		Certified Value		% Recovery Pd		% Recovery Pt		Relative Percent Difference	
	MICAP 1	MICAP 2	MICAP 1	MICAP 2	Pd	Pt	MICAP 1	MICAP 2	MICAP 1	MICAP 2	Pd	Pt
PD05	0,651	0,692	0,435	0,470	0,596	0,430	109%	116%	101%	109%	6%	8%
PK03 1	6,382	6,394	4,407	4,513	6,028	4,291	106%	106%	103%	105%	0%	2%
PK03 2	6,444	6,211	4,491	4,452	6,028	4,291	107%	103%	105%	104%	4%	1%
PK03 3	6,682	6,333	4,596	4,499	6,028	4,291	111%	105%	107%	105%	5%	2%
PK03 4	6,187	5,747	4,252	4,099	6,028	4,291	103%	95%	99%	96%	7%	4%
PK03 5	6,263	5,752	4,378	4,213	6,028	4,291	104%	95%	102%	98%	9%	4%
PK03 6	6,069	5,641	4,253	4,128	6,028	4,291	101%	94%	99%	96%	7%	3%

Table 4. Pd and Pt CRM results.

KEY
% Recovery
90-110%
80-90 and 110-120

The standard readback recovery is presented in Table 5 with all recoveries within ± 10% for all check standards in the linear range.

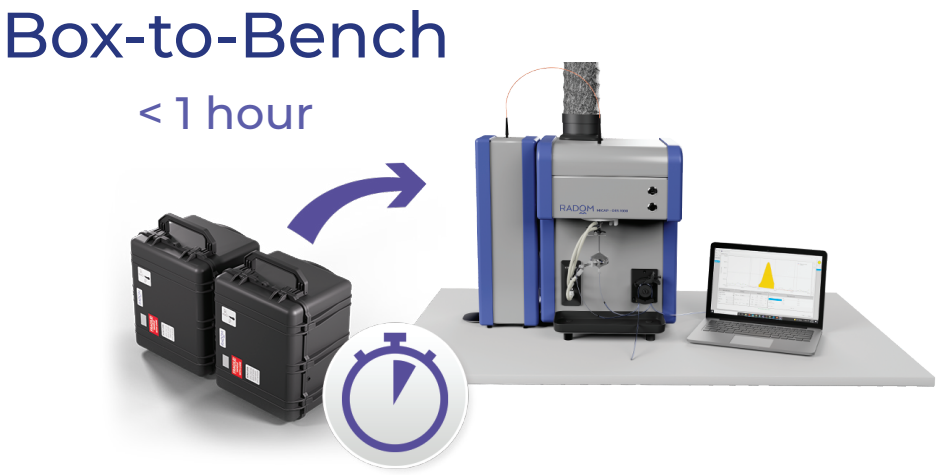
Check Standard	Au 242.495	Au 312.278	Pd363.469	Pd 342.122	Pt 299.769
CCV 50	93%	101%	NA	102%	98%
CCV 5	101%	96%	96%	105%	106%
CCV 0.5	107%	86%	104%	101%	105%
CCV 0.05	114%	NA	104%	NA	112%
CCB	<DL	<DL	<DL	<DL	<DL
Linear Range	0-5	5-50	0-0.5	0.5-50	0-50

Table 5. Standard Readback Recovery.

KEY
90-110%
80-90, 110-120%
NA=OUTSID CAL

Conclusions

The MICAP-OES 1000 system features a robust nitrogen plasma that is customer installable with it's "Box to Bench" design. With its Cerawave™ technology, the cost-effective nitrogen plasma emission spectometer has demonstrated excellent performance for Au, Pt, and Pd in this high-matrix fire assay application.



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