

High Accuracy and High Precision Determinations of Precious Metals and Platinum Group Metals using Inductively Coupled Plasma Spectroscopy

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Introduction

The precious metal catalyst (PMC) market world wide is approximately 14 billion per annum and estimated to increase to 19 billion by 2022. Profitability of reclaiming these precious metals is very dependent on the accuracy of the determination. 0.5% or tighter is a typical requirement. Achieving this on an ICP is not trivial with typical uncertainties of 1% for the technique. The following work will highlight Pt, Pd, and Au stability and accuracy. Also, HCL and Nitric Acid need to be kept consistent and is typically not quantified using ICP. However, data is presented by analyzing both chlorine and nitrogen.

Experimental

The Agilent 5110 ICP was used for this study. A standard sea spray nebulizer and a single pass cyclonic spray chamber was used for all work. A study was done to find the best conditions for highest accuracy and precision. Table 1 below shows each of the 9 conditions tested. Flow rates are in L/Min, power in KW and the pump rate in rpm. Condition set 8 yielded accuracy of <0.3% and long term stability shown in Figures below.



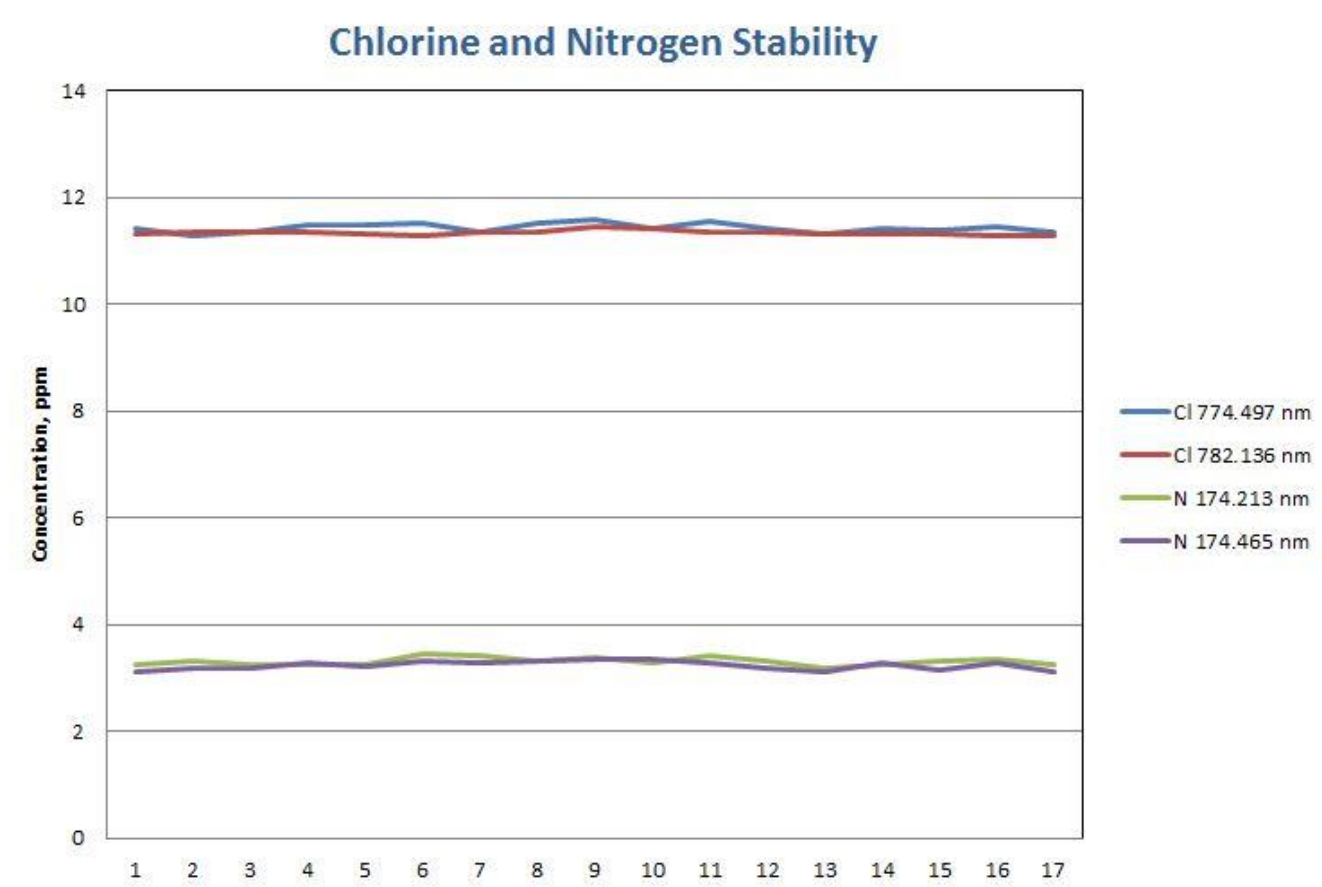
Set	Power	VH	Neb. Flow	Aux. Flow	Plasma Flow	Pump Rate
1	1.4	12	0.65	0.8	13	12
2	1.2	16	0.95	0.8	16	12
3	1.4	12	0.65	0.8	13	12
4	1.2	16	0.95	0.9	15	12
5	1.2	18	0.95	0.9	15	12
6	1.2	17	0.9	0.9	15	9
7	1.2	15	1	0.9	16	14
8	1.4	16	1.05	0.9	16	14
9	1.2	14	1	0.9	15	14

Experimental

Matrix matching of the acid concentration is important for recoveries and accuracy. HCL and HNO3 levels were quantitated using the acids as standards and DI water as the blank. The expected concentrations are 11% and 3% respectively. Tables below show this data as well as stability over time

HCL and HNO3 Quantitation by ICP analyzing Chlorine and Nitrogen

CI 774.497 nm	CI 782.136 nm	N 174.213 nm	N 174.465 nm
11.43	11.33	3.24	3.12
11.3	11.36	3.33	3.19
11.36	11.37	3.24	3.18
11.48	11.34	3.24	3.27
11.48	11.33	3.26	3.21
11.51	11.3	3.45	3.33
11.34	11.34	3.41	3.3
11.51	11.34	3.33	3.31
11.6	11.47	3.39	3.34
11.43	11.43	3.3	3.36
11.54	11.34	3.43	3.28
11.42	11.36	3.32	3.17
11.31	11.31	3.17	3.11
11.42	11.33	3.25	3.28
11.4	11.31	3.31	3.16
11.44	11.28	3.36	3.29
11.37	11.3	3.26	3.13
0.08	0.05	0.08	0.08
11.43	11.34	3.31	3.24
0.72	0.41	2.35	2.55



Results and Discussion

The results below show spike recoveries for the requested analytes in a Palladium matrix. The analysis was done in radial mode and the spike level was 100 ppb

Pd Sample Matrix, ppm: 100 ppb spikes and recoveries

Solution Label	Ag	Al	Al	As	As	Au
328.068	167.019	396.152	193.696	200.334	211.068	
Pt Blank	0.01	0	0.00 u	0.03 u	0.01 u	0.04
100ppbspk	0.1	0.1	0.1	0.14	0.11	0.13
Spike Rec. %	90	100	100	110	100	90

Solution Label	Bi	Ca	Ca	Ca	Cd	Cd
223.061	315.887	317.933	422.673	214.439	226.502	
Pt Blank	-0.01	0.01	0	0	0	0
100ppbspk	0.08	0.1	0.09	0.1	0.1	0.1
Spike Rec. %	90	90	90	100	100	100

Solution Label	Co	Co	Cr	Cr	Cu	Cu
237.863	238.892	206.158	283.563	213.598	223.009	
Pt Blank	0	0	-0.01	0	0	0
100ppbspk	0.09	0.1	0.1	0.1	0.09	0.1
Spike Rec. %	90	100	110	100	90	100

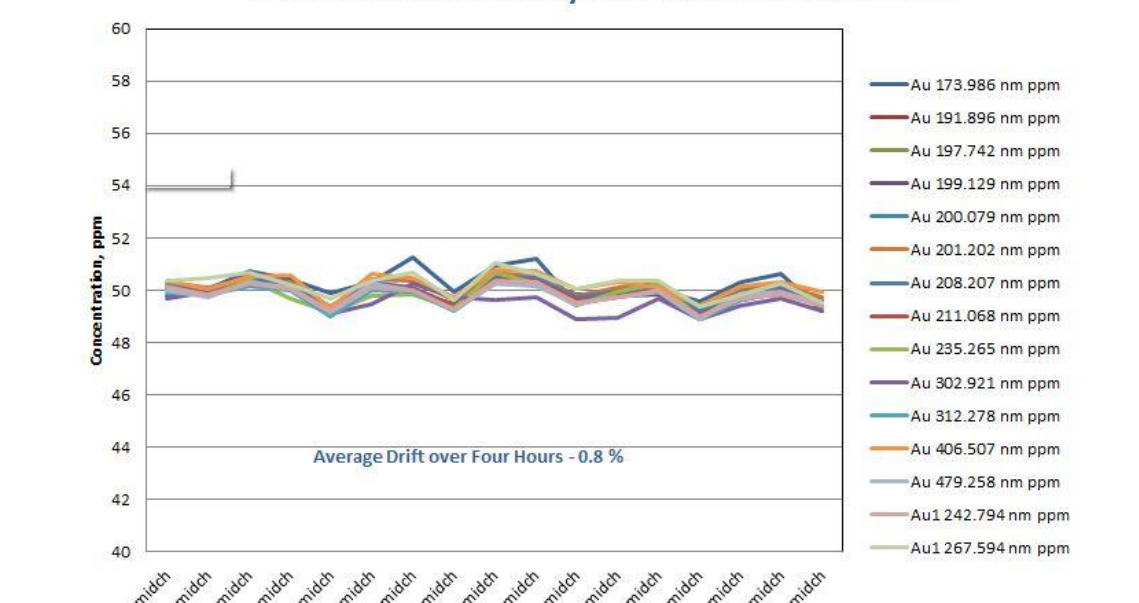
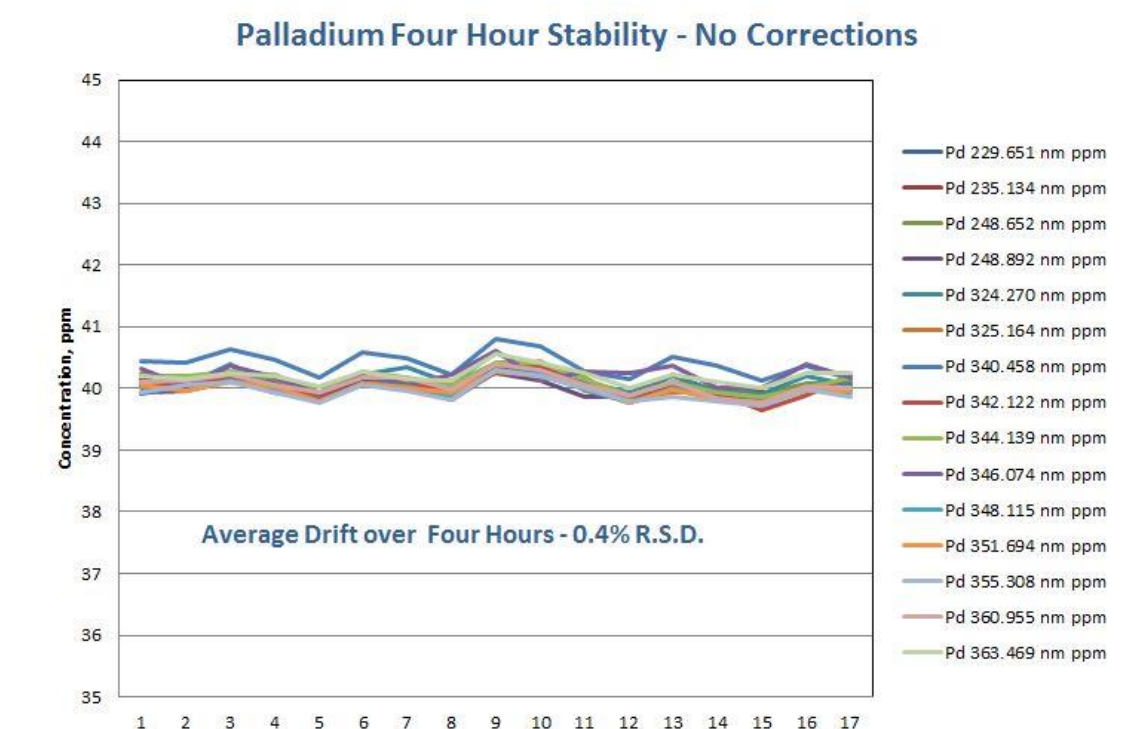
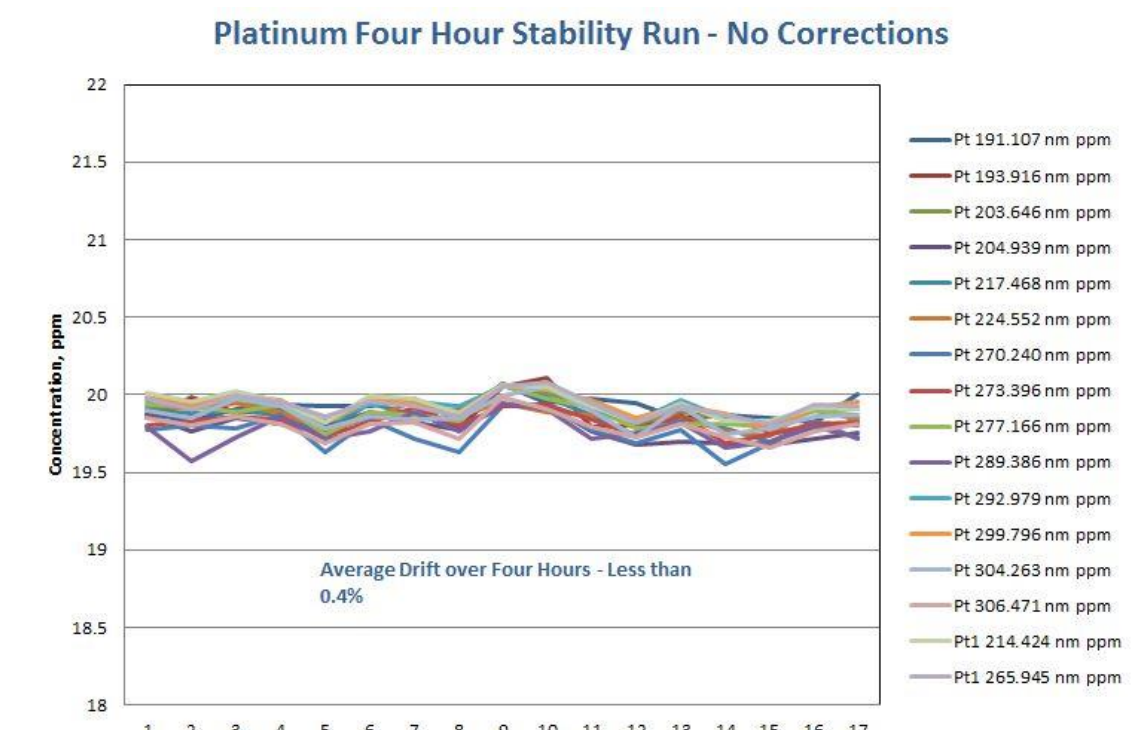
Solution Label	Mn	Mn	Mo	Mo	Mo	Ni
257.610	259.372	202.032	204.598	281.615	230.299	
Pt Blank	0	0	0	-0.01	0	0
100ppbspk	0.1	0.1	0.1	0.09	0.1	0.1
Spike Rec. %	100	100	100	100	100	100

Solution Label	Pb	Pb	Pb	Pt	Rh	Rh
217.000	220.353	283.305	214.424	233.477	343.488	
Pt Blank	0	0	0	0	0	0
100ppbspk	0.09	0.09	0.1	0.1	0.09	0.11
Spike Rec. %	90	90	100	100	90	110

Solution Label	Ru	S	S	S	Sb	Se
349.894	180.669	181.972	182.562	217.582	206.279	
Pt Blank	0	0.07	0.04	0	0	-0.01
100ppbspk	0.1	0.01	0.02	0.1	0.09	0.08
Spike Rec. %	100	100	100	100	90	90

Solution Label	Sn	Sn	Te	Ti	Ti	V
189.925	326.233	214.282	334.941	336.122	309.310	
Pt Blank	0	0	-0.01	0	0	0
100ppbspk	0.1	0.11	0.08	0.1	0.1	0.1
Spike Rec. %	100	110	90	100	100	100

Solution Label	W	Zn	Zn	Zr	Zr	Zr
239.708	206.200	213.857	339.198	343.823	349.619	
Pt Blank	0.01	0	0.01	0	0	0
100ppbspk	0.1	0.09	0.11	0.1	0.1	0.1
Spike Rec. %	90	90	100	100	100	100



Run	Sample	Uncorrected			Drift Corrected		
		Au ppm	Avg	RSD	Au ppm	Avg	RSD
Run #1	1	60.01	59.94	0.16%	60.03	60.04	0.02%
Run #1	2	59.87			60.04		
Run #2	1	60.02	60.17	0.36%	59.89	59.94	0.11%
Run #2	2	60.32			59.99		
Run #3	1	60.14	60.27	0.30%	60.04	60.09	0.11%
Run #3	2	60.40			60.14		
Run #4	1	60.24	60.03	0.49%	60.03	60.04	0.01%
Run #4	2	59.82			60.04		
Run #5	1	60.27	60.33	0.13%	60.00	59.96	0.09%
Run #5	2	60.39			59.92		
		Average Expected Difference	60.15	0.29%	Average Actual Difference	60.01	0.07%
			60.00	0.25%		60.00	0.02%

Run	Sample	Pd ppm			Pt ppm		
		Avg	RSD	Pd ppm	Avg	RSD	
Run #1	1	50.09	50.05	0.11%	50.01	50.04	0.08%
Run #1	2	50.01			50.07		
Run #2	1	50.01	50.12	0.31%	49.98	50.00	0.05%
Run #2	2	50.23			50.01		
Run #3	1	50.16	50.27	0.32%	49.98	50.02	0.09%
Run #3	2	50.38			50.05		
Run #4	1	50.23	50.08	0.42%	50.02	50.03	0.05%
Run #4	2	49.93			50.05		
Run #5	1	50.26	50.34	0.23%	50.00	49.99	0.04%
Run #5	2	50.43			49.97		
		Average Actual Difference	50.17	0.28%	Average Actual Difference	50.01	0.06%
			50.00	0.34%		50.00	0.03%

Run	Sample	Pt ppm			Pt ppm		
		Avg	RSD	Pt ppm	Avg	RSD	
Run #1	1	24.92	24.93	0.05%	25.02	25.05	0.19%
Run #1	2	24.94			25.09		
Run #2	1	24.90	24.97	0.38%	25.04	25.05	0.04%
Run #2	2	25.04			25.06		
Run #3	1	24.91	24.98	0.40%	25.01	25.03	0.13%
Run #3	2	25.05			25.06		
Run #4	1	24.99	24.89	0.61%	25.02	25.02	0.00%
Run #4	2	24.78			25.02		
Run #5	1	25.05	25.10	0.29%	25.07	25.06	0.06%
Run #5	2	25.15			25.05		
		Average Actual Difference	24.97	0.34%	Average Actual Difference	25.04	0.09%
			25.00	-0.10%		25.00	0.17%

To Summarize:		RSD Before Drift Corr	RSD After Drift Corr
		Au	0.29%
Pd	0.28%	0.06%	
Pt	0.34%	0.09%	

Conclusions

Customer application uses 16 wavelengths per analyte. Results are typically averaged and drift corrected. Precision is typically 0.3% and the accuracy has been reported as less than 0.5%