# Study on Preparation of Solid Ingot Calibration Standards for Trace Impurities in Palladium and Platinum

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**Abstract:** This work details, on a laboratory scale, a method to prepare small solid ingot calibration standards of palladium and platinum for use in solid sampling instrumentation such as arc/spark OES, spark ablation OES and GD-MS. Standards were prepared by first precipitating and co-precipitating spiked solutions of palladium and platinum along with the desired analytes then melting the "stock" powder with a bulk amount of 99.999% pure metal to attempt to achieve a final concentration in the metal of (5~10) ×10<sup>-6</sup> for 33 elements. Losses occurred in both the precipitation step and the thermal step with a final of 25 elements for Pd being quantitatively incorporated into the final ingot and 26 elements in Pt. Several contamination issues arose with Au, B, Fe, and Si.

**Key words:** calibration standards; instrumental analysis; trace impurities; contamination **CLC number:** TQ421.3<sup>+</sup>1, O657.31 **Document code:** A **Article ID:** 1004-0676(2016)04-0001-08

# 钯和铂中痕量杂质元素锭状固体标准样品制备的研究

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摘 要:详细介绍了制备钯和铂锭状固体标准样品的实验方法。该固体标样可用于测定固体样品的 电弧/火花发射光谱、火花烧蚀发射光谱和辉光放电质谱(GD-MS)等仪器分析方法。在标准样品的制 备过程中,首先将含有 33 种待测杂质元素的钯或铂溶液进行沉淀和共沉淀得到"贮备"粉末,然 后将粉末与 99.999%纯金属钯或铂进行熔炼,从而获得杂质元素含量在(5~10)×10<sup>-6</sup>的固体标准样品。 部分杂质元素在沉淀和共沉淀过程、热处理环节会发生损失,在最终得到的锭中,钯标准样品有 25 个元素、铂标准样品有 26 个元素得到定量回收,发生污染的元素主要包括金、硼、铁和硅。 关键词:标准样品;仪器分析;痕量杂质;污染

With the continued miniaturization of electronic components and sensors, the purity of the metals used in the fabrication of these devices becomes ever important. Smaller and smaller amounts of impurities can be problematic therefore the accurate quantification of these impurities at low levels is a matter of great concern. The precious metal industry has been taken to task to not only produce the metal of proper purity but to also be able to reliably quantify that purity. Analytical chemists have several options available to them in regards to instrumentation. Wet instrumental methods such as ICP-OES and ICP-MS, while they have the benefit of easily accessible certified reference materials, the preparation of the sample is time consuming, decreases the sensitivity by dilution, and increases the risk of contamination and matrix issues. Solid sampling, as in arc/spark OES, spark ablation OES and GD-MS, offer appealing alternatives to these

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problems. The only downfall, with the exception of gold<sup>[1]</sup>, is there are no certified reference materials available for trace elements in solid purity palladium or platinum. The last dispensed reference materials by the National Bureau of Standards were SRM 680 and 681, in the year 1967, for high purity platinum and doped platinum. The use of accurate certified reference materials as calibration standards will not only increase the precision and accuracy of these methods but it will also allow for a more direct comparison of these methods side by side. For those elements where SRM's or CRM's are not available, laboratories are faced with preparing in-house calibration standards or synthetic standards or calibration by indirect means such as solution standards<sup>[2]</sup>. This paper is the beginning of an investigation into the preparation of a set of calibration standards for trace impurities in a palladium and platinum matrix.

Solid standards can be prepared by adding impurities in the solid form to the matrix metal and then cast in a furnace or by adding aliquots of multi-element stock solutions, dried, reduced and pressed into a pellet<sup>[3-4]</sup>. The approach used in this investigation will be to utilize vendor prepared multi-element aqueous stock solutions which will be added to a solution of the dissolved matrix element and then reduce or co-precipitate as many of the impurities out of solution along with the matrix element. It is this precipitate that that will then be melted with the proper amount of bulk metal to achieve the desired concentration and cast into plates using an induction furnace.

#### **1** Experiment

#### 1.1 Preparation of calibration standards

1.1.1 Palladium and platinum standard stock powder

1) Run #1:  $5 \times 10^{-6}$  impurities addition

99.999% palladium or platinum (Sabin Metal Corp.) was dissolved in a 3:1 mixture of concentrated hydrochloric acid and concentrated nitric acid, both purity grade. The solution was heated until the entirety of the metal was dissolved. The impurities were added using vendor purchased, NIST traceable, custom made

multi-element calibration standards. There are two standards, one in HCl matrix and one in nitric acid matrix for a total of 34 elements (Tab.1). Proper aliquots are made and added to the dissolved Pd or Pt solution with the goal of achieving a final concentration of  $5 \times 10^{-6}$  impurities in the plate. The solution was brought to a pH of  $4\sim5$  with ammonium hydroxide. Once the proper pH is achieved the impurities and matrix are then reduced by adding 75 mL of 55% hydrazine hydrate. The palladium solution will finish with a final pH of 8. The platinum solution once again becomes acidic and must be adjusted up to 8. The solution was decanted off and the precipitated residue was vacuum filtered, dried and weighed.

#### Tab.1 Multi element stock solutions

表 1 多元素储备溶液	
Solution	Element
	Ag, Al, As, B, Bi, Ca, Cd, Co, Cr,
Stock A (HNO <sub>3</sub> )	Cu, Fe, Mg, Mo, Mn, Na, Ni, Pb, S,
	Sb, Se, Si, Sn, Te, Ti, V, Zn, Zr, W
Stock B (HCl)	Au, Pd, Pt, Ir, Rh, Ru

## 2) Run #2: $10 \times 10^{-6}$ impurities addition

In the second run the dissolution and reduction steps were the same as Run #1. The impurities added were increased for a final goal of  $10 \times 10^{-6}$  in the final plate. In addition to increasing the general concentrations, the concentrations of some of the problem elements seen in Run #1 were increased another 100 fold. For palladium there was added additional concentrations for aluminum, cadmium, antimony, selenium and titanium. In the initial run aluminum, antimony and titanium saw losses in the hydrazine drop and cadmium, selenium and titanium were lost during the melting. For platinum there was added additional concentrations of aluminum and ruthenium, lost in the hydrazine step, and bismuth, cadmium and titanium, which were lost in the melting step.

The effectiveness of the hydrazine reduction on each run was then determined by analyzing the spiked sponge by ICP after the hydrazine reduction and before melting.

# 1.1.2 Melting and casting the palladium and platinum plate

The spiked palladium and platinum powders were melted and cast using a vacuum induction furnace (Aseg Galloni, Pressovac Max). This programmable furnace has the ability to draw a vacuum, purge the system with an inert gas (argon) to prevent oxidation and to cast the melt under vacuum automatically.

The appropriate amount of spiked powder was added to a bulk amount of the pure powder and mixed. The target concentration in the final plate for Run #1 was  $5 \times 10^{-6}$  and Run #2,  $10 \times 10^{-6}$ . In order for the metal to properly couple in the furnace it was necessary to press the powder into disks using a hydraulic pellet press. The pressed disks were then placed in crucible and the crucible placed in the furnace.

A graphite casting mold was fabricated in-house to meet the plate specifications. In order for the melt to flow smoothly into the graphite casting mold (Fig.1), the mold must be coated with a releasing agent. In the case of palladium the releasing agent used was boron nitride and for platinum it was zirconium oxide. The mold is then heated to 500°C for palladium and 700°C for platinum.



Fig.1 Casting mold 图 1 铸模

After the mold is at temperature it is clamped shut and placed in the furnace in the proper position. Since palladium oxidizes at temperatures well below the melting point of palladium it is necessary to purge the system a minimum of 4 times with argon. With platinum the argon purge was not necessary.

Once the furnace has been purged sufficiently and reaches a temperature of 1600°C for palladium and 1850°C for platinum the melt is cast. The programmed cast sequence automatically tilts the crucible and mold assembly to pour the melt into the mold and returns to its original position. The system is being charged with 3 atmospheres of argon while this is happening. Once the sequence is done the mold can be removed and opened to reveal the cast plate (Fig.2).



Fig.2 Cast palladium plate 图 2 熔炼所得钯锭

The plate is then removed and cleaned in an ultrasonic bath for 3 minutes. The plate is now surfaced to remove any inclusions on the surface. This is done using a milling machine. After the surface is milled the plate is cut into individual pieces (Fig.3). The samples are now ready to prepared for analysis.



Fig.3 Final palladium plates 图 3 最终产品钯锭

#### 1.1.3 Analysis

The resulting plates were analyzed by ICP-OES (Agilent, 725-ES). For the ICP analysis pieces were clipped off each of 4 disks for each metal in order to

weigh 1.25 g each. The palladium samples were placed into Teflon beakers and dissolved in 8 mL of nitric acid and 20 mL of hydrochloric acid and heated until fully dissolved, approximately 2 hrs. The platinum samples were dissolved with 24 mL of nitric acid and 60 mL of hydrochloric acid. The platinum samples took 24 hrs to fully dissolve. When dissolved, the samples were transferred to a 50 mL Teflon volumetric flask and brought to volume with plasma grade water. The ICP was calibrated with matrix matched standards prepared from the same stock solutions used in the spiking of the solid standards. The instrument was calibrated with a blank and a  $2 \times 10^{-6}$  standard.

### 2 Results and discussions

# 2.1 Effectiveness of the hydrazine reduction

2.1.1 Run #1: Effectiveness of  $5 \times 10^{-6}$  impurities hydrazine reduction

The first step was to detail the efficiency of the reduction and coprecipitation for Run #1. Tab.2 shows the efficiencies of the hydrazine precipitation. Hydrazine has long been used as a reducing agent in many fields<sup>[5]</sup>. The pH of the solution becomes important in reducing as many of the impurities because of the different standard electrode potentials of hydrazine in acidic and alkali solutions:

 $N_2H_2 \leftrightarrow N_2 + 4H^+ + 4e^-$  (acidic:  $E^0 = -0.23V$ ) (1)  $N_2H_2 + 4OH^- \leftrightarrow N_2 + 4H_2O + 4e^-$  (alkaline:  $E^0 = -1.15V$ ) (2)

It can be seen here that the more alkaline solution is favored. The issue arises in the fact that many of the impurities as well as palladium and platinum will start to form volatile salts as the solution becomes more alkaline. More studies are necessary to determine the optimum pH range.

The palladium results (Tab.2) show that 12 (Al, B, Ca, Cr, Mg, Na, S, Si, Ti, V, W) out of the 33 elements did not get effectively precipitated by the hydrazine and showed losses over 50% (shaded in italics). The platinum results indicate that 8 (Al, B, Ca, Mg, Na, Ru, S, Si) out of the 33 elements showed losses over 50% (shaded in italics). This was expected for the alkali

metals and the alkaline earth metals because of their standard reduction potentials.

Tab.2 Impurities losses in palladium and platinum during Run#1 during reduction step

表っ	Run#1	过程钾和铂中杂质元素在还原过程中的损失
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Ŧ	Spiked spo	nge in p	alladium	Spiked spo	onge in p	latinum
Im- purity	Expected	Actual	Logt	Expected	Actual	Lost
1 5	/10 <sup>-6</sup>	/10 <sup>-6</sup>	Losi	/10 <sup>-6</sup>	/10 <sup>-6</sup>	Losi
Ag	50	49	-2%	125	137	10%
Al	50	0	-100%	125	43	-66%
As	50	46	-8%	125	127	2%
Au	50	56	12%	125	139	11%
В	50	0	-100%	125	0	-100%
Bi	50	55	10%	125	139	11%
Ca	50	3	-94%	127	7	-94%
Cd	50	54	8%	125	137	10%
Co	50	42	-16%	125	127	2%
Cr	50	12	-76%	125	126	1%
Cu	50	52	4%	125	142	14%
Fe	57	55	-4%	131	138	5%
Ir	50	52	4%	125	106	-15%
Mg	50	0	-100%	125	0	-100%
Mn	50	9	-82%	125	128	2%
Mo	50	40	-20%	125	87	-30%
Na	50	0	-100%	125	0	-100%
Ni	51	31	-39%	126	128	2%
Pb	50	49	-2%	125	138	10%
Pd	_	-	-	125	136	9%
Pt	50	50	0%	-	-	-
Rh	50	49	-2%	125	122	-2%
Ru	50	31	-38%	125	42	-66%
S	50	4	-92%	138	5	-96%
Sb	50	37	-26%	125	80	-36%
Se	50	47	-6%	125	130	4%
Si	50	3	-94%	125	39	-69%
Sn	50	55	10%	126	134	6%
Те	50	58	16%	125	114	-9%
Ti	50	10	-80%	127	130	2%
V	50	8	-84%	125	119	-5%
W	50	23	-54%	125	77	-38%
Zn	50	52	4%	126	134	6%
Zr	50	30	-40%	125	122	-2%

# 2.1.2 Run #2: Effectiveness of $10 \times 10^{-6}$ impurities hydrazine reduction

Following the same methods as Run #1 but with increased concentrations yielded the following results. Here we focus on the elements that showed losses through the hydrazine drop and that we increased the initial concentration (Tab.3). For the loss during reduction we found that on palladium the aluminum and antimony showed similar losses (shaded in italics) but still retained a decent amount going into the melting step. Titanium on the other hand had minimal losses compared to an 80% loss on run #1. As for platinum aluminum, was not so successful with a larger 96% loss compared to a 66% loss in run #1 and ruthenium showed similar losses as before but still retained  $133 \times 10^{-6}$  which is what we expected.

#### 2.2 Melt losses and contaminants

Once the spiked palladium and platinum powder was obtained and characterized the next step was to determine what impurities were lost in the melting process and what contaminants may have been introduced.

#### 2.2.1 Run #1 melt results

The melt losses and contaminants introduced for the palladium and platinum plate are detailed as follows (Tab.4 in next page). A total of 11 elements in the palladium showed melt losses of 50% or more (shaded in italics). The losses of selenium, arsenic, cadmium, tellurium and lead were expected to some extent because of their temperatures of volatility falling within the melting point of palladium. It is interesting to note that arsenic, molybdenum, manganese, lead, vanadium, zirconium and tungsten were not lost on the platinum, and this most likely is due to the fact that these elements form stable alloys with platinum and not in palladium. In the other direction it is seen that bismuth and antimony alloy better with palladium than platinum. For the melt losses palladium lost 10 elements, platinum had a better recovery and only lost 8 elements.

While Tab.2 detailing the hydrazine reduction did not show any appreciable contamination issues Tab.3 for palladium showed several apparent contaminants with gold, boron, calcium and silica and for platinum showed only silica. The boron in the palladium was from the boron nitride releasing agent that is applied to the casting mold. In past trials this was not seen as

# Tab.3 Impurities losses in palladium and platinum during Run #2 during reduction step

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I	Spiked spor	nge in pa	alladium	Spiked spo	nge in p	latinum
rity	Expected /10 <sup>-6</sup>	Actual /10 <sup>-6</sup>	Lost	Expected /10 <sup>-6</sup>	Actual /10 <sup>-6</sup>	Lost
Ag	100	97	-3%	100	104	4%
Al	1100	104	-91%	228	8	-96%
As	100	59	-41%	100	99	-1%
Au	100	93	-7%	100	97	-3%
В	100	0	-100%	100	0	-100%
Bi	100	96	-4%	1133	1130	0%
Ca	100	0	-100%	100	11	-89%
Cd	1100	1105	0%	1100	1110	1%
Co	100	99	-1%	100	103	3%
Cr	100	67	-33%	100	37	-63%
Cu	100	95	-5%	100	97	-3%
Fe	109	96	-12%	100	107	7%
Ir	100	101	1%	100	100	0%
Mg	100	2	-98%	100	0	-100%
Mn	100	99	-1%	100	104	4%
Mo	100	98	-2%	100	94	-6%
Na	100	0	-100%	100	0	-100%
Ni	100	96	-4%	100	100	0%
Pb	100	103	3%	100	99	-1%
Pd	_	-	-	100	99	-1%
Pt	100	95	-5%	-	-	-
Rh	100	98	-2%	100	103	3%
Ru	100	88	-12%	228	133	-42%
S	100	0	-100%	100	0	-100%
Sb	1100	181	-84%	100	28	-72%
Se	1100	1268	15%	100	100	0%
Si	100	27	-73%	100	11	-89%
Sn	100	99	-1%	100	99	-1%
Те	100	97	-3%	100	101	1%
Ti	1100	1051	-4%	1100	387	-65%
V	100	59	-41%	100	50	-50%
W	100	13	-87%	100	93	-7%
Zn	100	100	0%	100	107	7%
Zr	100	26	-74%	100	28	-72%

Tab.4	Impurities	losses/contaminant	in	palladium	and
I	olatinum dui	ring Run #1 melting s	tep		

衣 4 Kun #1 熔烁过程钯和钼甲尔应兀系旳顶大/汅	表 4	Run #1	熔炼过程钯和铂中杂质元素的损失/污染
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	Final plat	te of pall	ladium	Final pla	te of pla	tinum
Impurity	Expected	Actual	T	Expected	Actual	Lent
	/10 <sup>-6</sup>	/10 <sup>-6</sup>	Lost	/10 <sup>-6</sup>	/10 <sup>-6</sup>	Lost
Ag	5	4	-20%	5	4	-20%
Al	0	0		2	0	-100%
As	5	0	-100%	5	4	-20%
Au	6	10	67%	6	6	0%
В	0	29		0	0	
Bi	6	4	-33%	6	1	-83%
Ca	0	5		2	3	50%
Cd	5	2	-60%	5	2	-60%
Co	4	3	-25%	5	5	0%
Cr	1	3	200%	5	5	0%
Cu	5	6	20%	6	5	-17%
Fe	6	15	150%	12	9	-25%
Ir	5	5	0%	4	6	50%
Mg	0	0		0	0	
Mn	1	0	-100%	5	5	0%
Mo	4	0	-100%	3	4	33%
Na	0	0		0	0	
Ni	3	4	33%	6	5	-17%
Pb	5	0	-100%	6	5	-17%
Pd	_	_	-	5	8	60%
Pt	5	7	40%	_	-	-
Rh	5	4	-20%	5	6	20%
Ru	3	4	33%	2	2	0%
S	0	0		13	6	-54%
Sb	4	3	-25%	3	0	-100%
Se	5	1	-80%	5	0	-100%
Si	0	36		2	8	300%
Sn	6	5	-17%	6	9	50%
Те	6	0	-100%	5	0	-100%
Ti	1	0	-100%	7	1	-86%
V	1	0	-100%	5	4	-20%
W	2	0	-100%	3	3	0%
Zn	5	4	-20%	6	4	-33%
Zr	3	0	-100%	5	4	-20%

boron should not alloy with palladium. It could be attributed to insufficient cleaning of the plates. The silica in both the palladium and the platinum is most likely from the crucible that the metals were melted in. The origin of the iron in the palladium is most likely from the cutting devices.

2.2.2 Run #2 melt results

Now looking at melt losses (Tab.5, shaded in italics), and focusing again on those elements that were lost during this step in Run #1 and which we increased the initial concentration. It was shown that with palladium, the cadmium, selenium and titanium all showed appreciable losses at 45%, 44%, and 88% but still retained enough or more than enough than the goal concentration of  $10 \times 10^{-6}$  in the plate. The same was true for platinum with the bismuth, cadmium and titanium.

# Tab.5 Impurities losses/contaminant in palladium and platinum during Run #2 melting step

表 5	Run #2 熔炼过程钯和铂中杂质元素的损失/污染

	Final plat	te of pal	ladium	Final pla	te of pla	ıtinum
Impurity	Expected	Actual	Loct	Expected	Actual	Lost
	/10 <sup>-6</sup>	/10 <sup>-6</sup>	Losi	/10 <sup>-6</sup>	/10 <sup>-6</sup>	Losi
Ag	10	10	0%	10	9	-10%
Al	10	3	-70%	1	1	0%
As	6	6	0%	10	8	-20%
Au	9	6	-33%	10	8	-20%
В	0	47		0	0	
Bi	10	7	-30%	111	73	-34%
Ca	0	6		1	11	
Cd	110	61	-45%	109	26	-76%
Co	10	9	-10%	10	9	-10%
Cr	7	5	-29%	4	4	0%
Cu	9	10	11%	10	10	0%
Fe	10	18	80%	11	15	36%
Ir	10	10	0%	10	11	10%
Mg	0	0		0	0	
Mn	10	7	-30%	10	10	0%
Мо	10	0	-100%	9	7	-22%
Na	0	0		0	0	
Ni	10	9	-10%	10	9	-10%
Pb	10	8	-20%	10	9	-10%
Pd	_	_	-	10	10	0%
Pt	10	12	20%	_	-	-
Rh	10	9	-10%	10	8	-20%
Ru	9	7	-22%	13	12	-8%

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	Tab.5	continued	(续表	5)
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	Final plate of palladium			Final plate of platinum		
Impurity	Expected	Actual	T t	Expected	Actual	T
	/10 <sup>-6</sup>	/10 <sup>-6</sup>	Lost	/10 <sup>-6</sup>	/10 <sup>-6</sup>	Lost
S	0	0		0	0	
Sb	18	18	0%	3	0	-100%
Se	127	71	-44%	10	0	-100%
Si	3	43	1333%	1	25	2400%
Sn	10	6	-40%	10	15	50%
Те	10	10	0%	10	13	30%
Ti	105	13	-88%	38	18	-53%
V	6	2	-67%	5	4	-20%
W	1	0	-100%	9	6	-33%
Zn	10	8	-20%	11	10	-9%
Zr	3	0	-100%	3	5	67%

#### 2.3 Summary

#### 2.3.1 Problem element

Comparing the problem elements Run #1 to Run #2 (Tab.6 and 7), it shows that considerable amounts were retained in the final plate by increasing the initial concentration.

## Tab.6 Comparison of Run #1 vs. #2 with problem elements, impurities retained palladium final plate

田小灰百里心	/10	
Element	Run #1	Run #2
Al	0	3
Cd	2	61
Sb	3	18
Se	1	71
Ti	0	13

Tab.7 Comparison of Run #1 vs. #2 with problem elements, impurities retained platinum final plate

表 7	Run #1 和 Run	#2 流程最终所得铂锭中问题元素和	残
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留杂质含量	对比	/10 <sup>-6</sup>	
Element	Run #1	Run #2	
Al	0	1	
Bi	1	73	
Cd	2	26	
Ru	2	12	
Ti	1	18	

#### 2.3.2 Final recovery

In summary (Tab.8), through the hydrazine reduction and the melting, palladium retained a measurable amount of 18 out of the 33 elements added giving a recovery of 54.5%. Platinum had a much better recovery at 22 out of the 33 elements added giving a recovery of 66.7%.

Tab.8 Final recovery comparison (33 elements added) 表 8 最终回收对比(添加元素数量为 33 个)

	Run #1		Run #2	
Matrix	Elements in	Final	Elements in	Final
	final plate	recovery	final plate	recovery
Pd	18	54.5%	25	75.8%
Pt	22	66.7%	26	78.8%

In comparing the run summaries side by side in Tab.8, it can be seen that there was an increase in recoveries on the run with increased and added concentrations with palladium going from 18 to 25 elements in the final plate and platinum from 22 to 26 elements in the final plate.

#### 3 Conclusion

In conclusion it has been shown that it is possible to create calibration standards using commercially available multi-element stock solutions. There are several options to yet explore to solve some of the issues that arose such as low recoveries in the final plates. Adding impurities as the solid element or stable salts has yet to be attempted in our laboratory. Minimizing contamination should be easily overcome through diligent cleaning and varying the melting times and the general optimizing of the method through varying the final pH, digestion times and temperatures. The most important aspect, homogeneity, still needs to be thoroughly investigated. Before any sample can be used for comparison purposes, it must be proven to be completely homogeneous.

This short experiment is hoped to be the beginning of an in depth study into solving a problem that has plagued all laboratories that use these instruments, vendors that want to sell these instruments and customers who rely on the results of these instruments to be accurate.

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