

# The Study and Application of Hydrometallurgical Gold Leaching in the Analysis of Refractory Precious Metals

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**Abstract.** Three orthogonal tests are separately designed for each hydrometallurgical gold leaching process to finding the optimum reaction conditions of melting gold and palladium in each process. Under the optimum condition, the determination amount of gold and palladium in aqua regia—hydrofluoric acid, Sodium thiosulfate, and potassium iodide reaches 2.87g/kg and 8.34 g/kg, 2.39g/kg and 8.12 g/kg, 2.51g/kg and 7.84g/kg. From the result, the content of gold and palladium using the leaching process of combining Aqua regia, hydrofluoric acid and hydrogen peroxide is relatively higher than the other processes. In addition, the experiment procedure of aqua regia digestion operates easily, using less equipment, and its period is short.

## 1. Introduction

In recent years, with the development of technology and electronic industries, the pace of upgrading electronic devices is accelerating, and the electronic wastes from it are also increasing [1]. Printed circuit boards is an important part of every electronic devices, generally containing gold, silver, palladium and other precious metals, whose recycling value is high but quantity is low[2]. So the pretreatment method is very important for the recycling of waste circuit board.

There are many literature reports of hydrometallurgical treatment process [3-5]. But the report of the design of orthogonal tests on the process is few. In this article, three orthogonal tests are designed for each hydrometallurgical gold leaching process to finding the optimum reaction conditions of melting gold and palladium in each process and determined the content.

## 2. Experimental

### 2.1. Orthogonal tests on aqua regia system

**2.1.1. Ingredients.** The ingredients of the experiment are the powder of circuit board below 100 mesh.

**2.1.2. Materials, chemicals and apparatus.** Nitrate, hydrochloric acid, hydrofluoric acid, hydrogen peroxide (All the reagent is AR), 50ml Teflon crucible, separating funnel, heating plate, ICP-AES, electronic analytical balance.



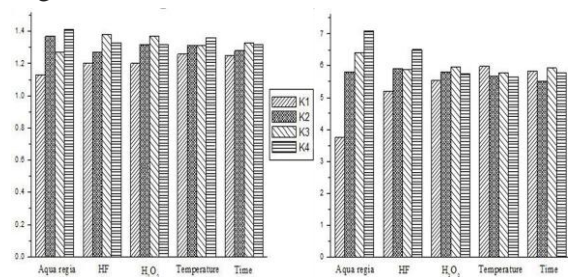
**2.1.3. Experimental procedure.** The sample (0.5000g) was taken from the crushed circuit board and put into Teflon crucible. A little deionized water was added. New aqua regia was made ( $V_{\text{HCl}}:V_{\text{HNO}_3}=3:1$ ) and slowly added. When the yellow smoke gradually disappeared, heated until the solution became transparently green, with stable level and no yellow smoke (otherwise aqua regia should be added). Cooled to room temperature, hydrofluoric acid was added and heated again. The lid was sealed when white smoke appeared. Cooled down when about 0.5ml solution remained in it. Hydrogen peroxide was added and heated until the solution is almost dry out, then nitrate (15ml, 10%) was added. The digested solution was filtered by separating funnel, diluted to volume by 50ml volumetric flask, and mixed to prepare for measurement.

**2.1.4. Result and analysis.** The amount of aqua regia, hydrofluoric acid and hydrogen peroxide, the reaction time and the reaction temperature are chosen as the factors. Each factor has four levels. A orthogonal test, shown in Table 1.

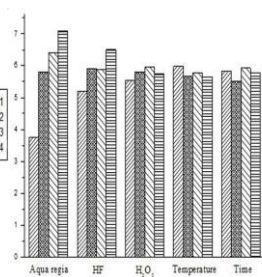
**Table 1.** The factors of orthogonal tests and their levels.

Factors Levels	Aqua regia /ml	HF /ml	H <sub>2</sub> O <sub>2</sub> /ml	Time /h	Temperature /°C
1	10	5	3	1.5	100
2	15	7	5	2	120
3	20	9	7	2.5	140
4	25	11	9	3	160

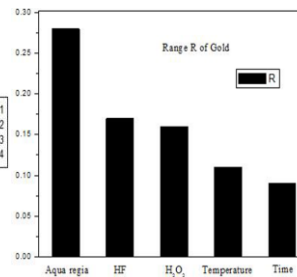
The value K and the range R gotten from analyzing the result are shown in the Figure 1. to Figure4.



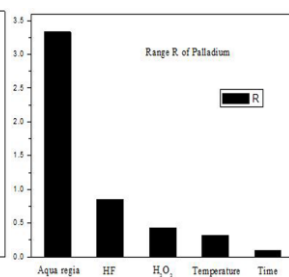
**Figure 1.** The analysis on the value K of leaching gold in aqua regia system.



**Figure 2.** The analysis on the value K of leaching palladium in aqua regia system.



**Figure 3.** The analysis on the range R of leaching gold in aqua regia system.



**Figure 4.** The analysis on the range R of leaching palladium in aqua regia system.

**2.1.5. The choice of best experimental condition.** From the analysis of the result, the value K and the range R of gold and palladium can be calculated. It can be concluded that the amount of aqua regia has the most significant influence on the result of leaching, followed by hydrofluoric acid; the reaction time has the smallest influence.

**2.1.6. The situation of gold and palladium leaching under the optimum condition.** Based on the orthogonal experiment, the optimum condition and the result is shown in the Table 2.

**Table 2.** The situation of gold and palladium leaching in aqua regia system.

Factors	Best condition of Au	Result	Best condition of Pd	Result
Aqua regia/ml	25	2.87g/kg	25	8.34 g/kg
HF/ ml	9		11	
H <sub>2</sub> O <sub>2</sub> / ml	7		7	
Temperature/°C	160		160	
Time/h	2.5		2.5	

Under this reaction condition, the content of gold and palladium dissolved reached 2.87g/kg and 8.34g/kg. It can indicate that the dissolve effect is great under such conditions and the result is reliable.

## 2.2. Orthogonal tests on sodium thiosulfate system

2.2.1. *Ingredient.* The ingredients of the experiment are the powder of circuit board below 100 mesh.

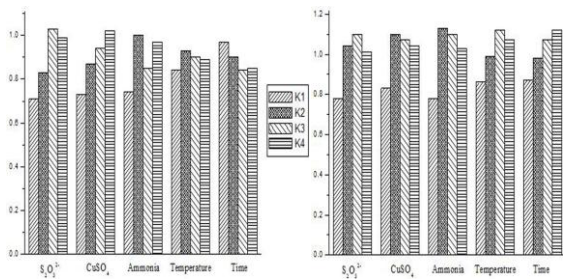
2.2.2. *Materials, chemicals and apparatus.* The reagent: sodium thiosulfate, sodium chloride, copper sulfate, ammonia (all AR). The apparatus used in this experiment was Magnetic stirrer, Electronic balance, Thermostatic bath (HH-1), ICP-AES.

2.2.3. *Experimental procedures.* The powdered sample was weighed and then put into 250ml beaker. Some sodium thiosulfate was slowly transferred into beaker by pipette. Then some copper sulfate and ammonia was transferred into beaker. Sodium thiosulfate concentration, copper sulfate concentration, ammonia concentration, reaction time and temperature were chosen as the factors of the experiment. Each factor has four levels. This orthogonal experiment is shown in the Table 3.

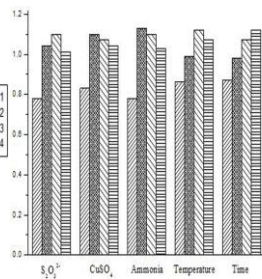
**Table 3.** The factors and levels of orthogonal tests on sodium thiosulfate system.

Factors Levels	S <sub>2</sub> O <sub>3</sub> <sup>2-</sup> /mol/L	CuSO <sub>4</sub> /mol/L	Ammonia /mol/L	Time /h	Temperature /°C
1	0.2	0.1	0.2	1.5	20
2	0.3	0.2	0.4	2	40
3	0.4	0.3	0.6	3	60
4	0.5	0.4	0.8	4	80

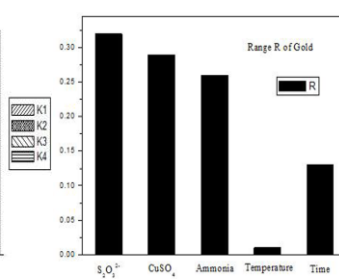
2.2.4. *Result and analysis.* The value K and the range R gotten from analyzing the result are shown in the Figure 5. to Figure 8.



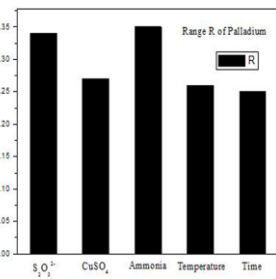
**Figure 5.** The analysis on the value K of leaching gold in sodium thiosulfate system.



**Figure 6.** The analysis on the value K of leaching palladium in sodium thiosulfate system.



**Figure 7.** The analysis on the range R of leaching gold in sodium thiosulfate system.



**Figure 8.** The analysis on the range R of leaching palladium in sodium thiosulfate system.

**2.2.5. The choice of best experimental condition.** From the value K and the range R, it can be shown that the amount of sodium thiosulfate, copper sulfate and ammonia has a significant influence on the result. When the concentration of  $S_2O_3^{2-}$  is increased, the compound  $[Cu(S_2O_3)_3]^{5-}$  will be synthesized in the solution, resulting the decrease of the concentration of  $S_4O_6^{2-}$ .  $S_2O_3^{2-}$  can be oxidized to  $S_4O_6^{2-}$  by  $Cu^{2+}$ . The higher concentration of  $Cu^{2+}$  leads to the larger consumption of  $S_2O_3^{2-}$ . The final gold leaching rate will be low. So these three factors should be strictly controlled. The influence of temperature and reaction time is small.

**2.2.6. The situation of gold and palladium leaching under the optimum condition.** Under the premise of sodium thiosulfate leaching experiments, the optimum condition and the result is shown in the Table 4.

**Table 4.** The situation of gold and palladium leaching in sodium thiosulfate system.

Factors	Best condition of Au	Result	Best condition of Pd	Result
$S_2O_3^{2-}$ / mol/L	0.4		0.4	
$CuSO_4$ / mol/L	0.2		0.2	
Ammonia/ mol/L	0.2	2.39g/kg	0.2	8.12 g/kg
Temperature / $^{\circ}C$	80	g	60	
Time/h	4		4	

Under the best dissolution condition, their content reaches 2.39g/kg and 8.12 g/kg. It shows that under this condition the dissolution effect of gold and palladium is well and the result is reliable.

### 2.3. Orthogonal tests on KI system

**2.3.1. Ingredients.** The ingredients of the experiment is the powder of circuit board below 100 mesh.

**2.3.2. Materials, chemicals and apparatus.** KI (AR), Sodium hypochlorite (effective chlorine content  $\geq 12\%$ ), deionized water. Digital homothermal magnetic stirrer, homothermal water bath

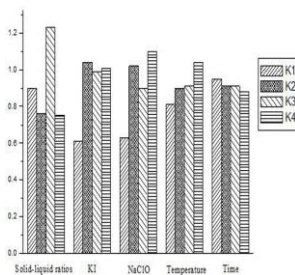
kettle, standard sieve set, ICP-AES.

**2.3.3. Experimental procedures.** The powdered sample was weighed and then put into 250 ml beaker. Some KI was slowly transferred into beaker by pipette. The orthogonal experiment is designed as what is shown in the Table 5.

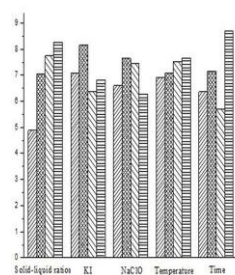
**Table 5.** The factors and levels of KI system.

Factors Levels	Solid-liquid ratios	KI /mol/L	NaClO /mol/L	Time /h	Temperature/ °C
1	1:6	0.05	6	2.5	25
2	1:8	0.1	8	3	30
3	1:10	0.2	10	4	40
4	1:12	0.3	12	5	50

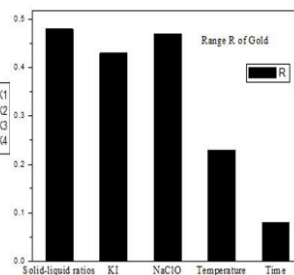
**2.3.4. Result and analysis.** The value K and the range R gotten from analyzing the result are shown in the Figure 9. to Figure 12.



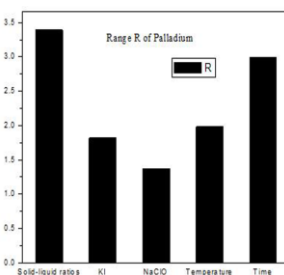
**Figure 9.** The analysis on the value K of leaching gold in KI system.



**Figure 10.** The analysis on the value K of leaching gold in KI system.



**Figure 11.** The analysis on the value K of leaching gold in KI system.



**Figure 12.** The analysis on the value K of leaching gold in KI system.

**2.3.5. The choice of best experimental condition.** The experiment of using KI method to leach precious metal from circuit board indicates that sodium hypochlorite, as an additional oxidant, can effectively increase the leaching rate of precious metal. In addition, the solid-liquid ratio and the concentration of KI both have significant influence on the leaching rate, so these two factors should be strictly controlled in practice application.

**2.3.6. The situation of gold and palladium leaching under the optimum condition.** Under the premise of KI leaching experiments, circuit boards are dissolved in the best reaction condition. Then the digestion solution is determined. The result is shown in Table 6.

**Table 6.** The situation of gold and palladium leaching in KI system.

Factors	Best condition of Au	Result	Best condition of Pd	Result
Solid-liquid ratio	0.1		0.08	
KI/ mol/L	0.1		0.1	

NaClO/ mol/L	12	2.51g/kg	8	7.84 g/kg
Temperature/°C	50		50	
Time/h	2.5		3	

Under the best dissolution condition, their content reaches 2.51g/kg and 7.84 g/kg. It shows that under this condition the dissolution effect of gold and palladium is well and the result is reliable.

### 3. Conclusion

Three orthogonal tests are separately designed for each hydrometallurgical gold leaching process to finding the optimum reaction conditions of melting gold and palladium. The result shows that: aqua regia—hydrofluoric acid, Sodium thiosulfate, and potassium iodide, under the optimum condition, the determination amount of gold and palladium reaches 2.87g/kg and 8.34 g/kg, 2.39g/kg and 8.12 g/kg, 2.51g/kg and 7.84g/kg respective. From the result, we can see that the content of gold and palladium determined in the digestion solution from digesting circuit board using the leaching process of combining Aqua regia—hydrofluoric acid is relatively higher than the others. In addition, the experiment procedure of aqua regia digestion operates easily, using less equipment, and its period is short. So there is reliable to choose aqua regia gold leaching process as the pretreatment method.

### 4. Acknowledgments

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### 5. References

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