Precious Metal Precipitation in Eco-Goldex E Series Pregnant Solution with Zinc Cementation

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Declarations and Notes

- Gold precipitation and purification involve several chemicals that are not part of Eco-goldex package and technology, but are common methods and procedures in gold precipitation and purification practices. Since its unique feature of effective gold stripping capability, Eco-goldex has developed and identified several methods that proven to be effective for gold precipitation from Eco-goldex E derived preg solution.

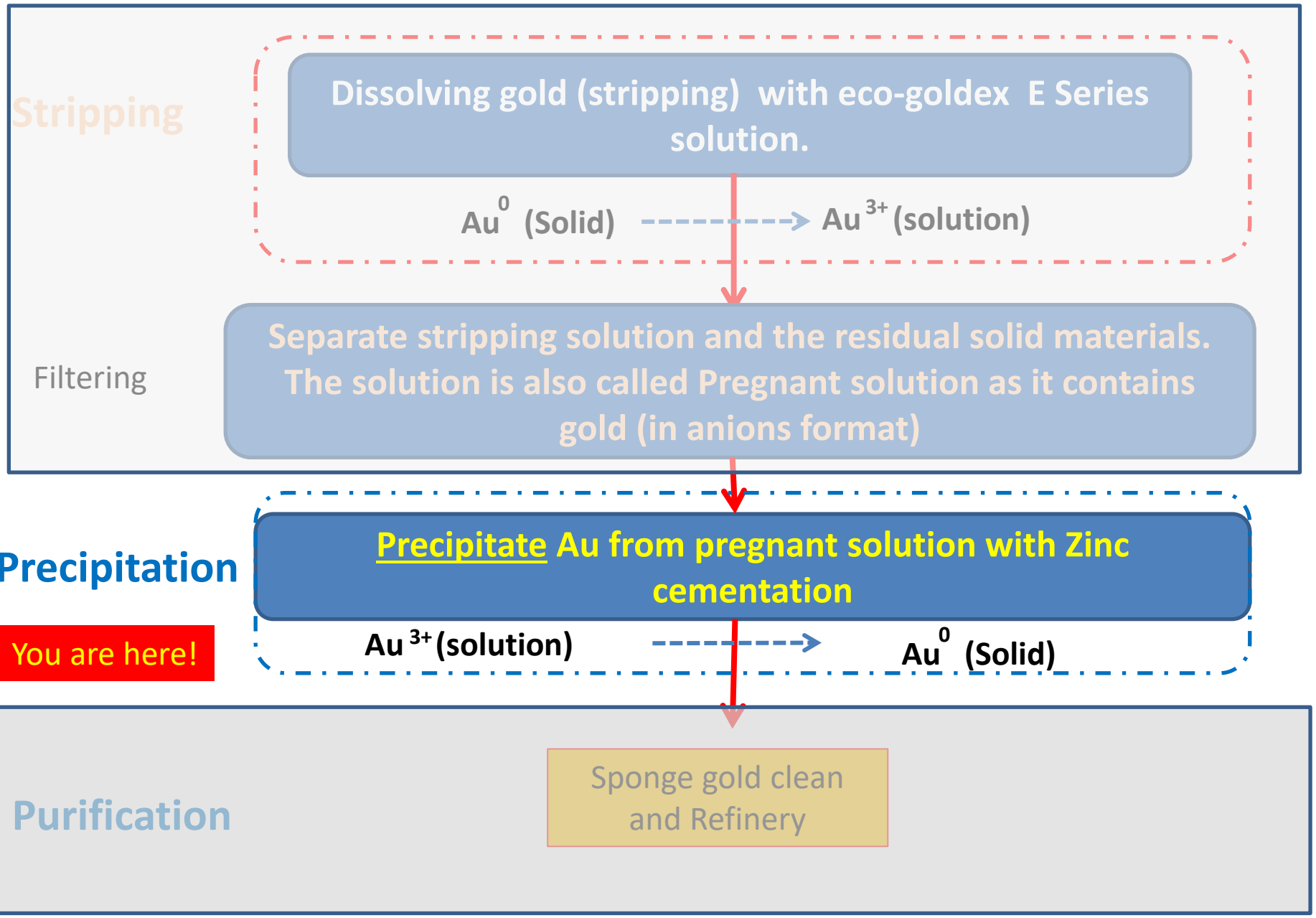
- All precautions and safety protections at operation site should be exercised and respected!

- Reagents should be stored at safe and secured place; reagent solution should not be intake. Leaching/stripping/precipitation/refinery operations should be conducted at open or well ventilated space.

- This instruction is prepared for gold precipitation dummies who don't have much background of gold precipitation and procedures.

- Considering the variety of e-scrap materials, solution colors (changes) observed in preg solution precipitation with zinc powder/dust/strips are not necessarily the same.

Major Gold Stripping/Precipitation/Recovery Steps



Zinc Cementation method and procedures

Chemicals and material used in this method

- 99.9% purity metallic Zinc strips / powder
  (purchased from eco-goldex or market, for gold precipitation. Galvanized zinc doesn't work!)
- Pregnant solution pH must be converted from base to acid condition for effective zinc cementation;

  (!!zinc cementation doesn't work if pregnant solution pH >7.0!!)
- Chemicals for pH adjust: diluted HCl or H_2SO_4, (15-20%)
- Agent C (Coagulation/flocculent agent for accelerating fine particle precipitation agent, optional)

Note 1:

1. *Preg-solution must be filtered and its pH must be adjusted to range **1.0 - 4.0** before adding zinc strips/powder. Without pH adjust, gold won't precipitate with zinc strips/powder.*

2. *We recommend using HCl /H2SO4 for pregnant solution pH adjust for better cementation result.*

3. *In the first step pH adjust, pH range should be around **1.0 - 4.0** with diluted HCl or H2SO4;*

   (!!Do pH adjusting in open space or well ventilated area!!)

Zinc powder has better cementation efficiency than strips but zinc strips provide simpler cementation process and skip the filtration step, so eco-goldex recommend both of them, users may decide which one to use based on their preference.

General zinc cementation processes

Eco-goldex has identified that multiple zinc cementation processes must be practiced to ensure satisfying gold recovery(>90% recovery).

- ✓ **The first step cementation usually drop about 45-55% gold from preg solution. After the first zinc cementation, pregnant solution pH will be raised to 5-6 in the process.**
- ✓ **The second step cementation drops about 40-45% gold from preg solution;**
- ✓ **We recommend 2-3 steps gold precipitation with zinc cementation till gold concentration is considerably low (<3 g/t) in the solution.**
- ✓ **More importantly, Do Not try to start with direct SMB precipitation as it won't work.**

1st step

Filtered preg solution → pH adjust with HCl or H_2SO_4 to 1.0-4.0→add zinc powder/strips (stirring) → let solution settle down → filtration (separate sludge and solution, zinc strips don't need filtration)

2nd step

Filtered solution from step 1 → pH adjust with HCl to 2-4.5 (after first step zinc cementation, preg solution pH will raise up to 6) →add zinc powder/strips → settle down → filtration (separate sludge and solution)

3rd step…

Use stannous chloride ($SnCl_2$) solution doing gold coloring test, if color test is positive, Repeat step 2 till solution becomes clear and zinc strips color not change after 8 hours.

- Cementation time usually overnight for zinc strips. 2-4 hours for zinc powder.
- After 2 steps zinc cementation, gold concentration in solution should be lower than 3 ppm.

Preg-solution pH Adjust using HCl

1. Slowly add diluted HCl into preg solution while stirring, make sure do this step at open space or in well ventilation area.
2. Make sure taking frequent measurements of preg-solution pH using pH tester/paper stick to avoid overdrawn pH.

pH test sticker
Indicates original preg-solution pH>9 (pH test paper showing violet or dark purple color)

Use diluted HCl/H2SO4 solution to drop Preg solution pH down to range 1-4.0

Stirring preg-solution while adding HCl / H2SO4 till Preg-solution pH down to 1.0-4.0

Zinc strips cementation

Once preg-solution pH regulated to the desired range (~2-4.5), put zinc strips in the preg-solution.
Make sure all zinc strips are submerged in the solution.

Adding zinc strips into it and slowly stirring solution.

Keeping zinc strips submerged and let the whole process for 6-8 hours. If zinc strips breaks into small pieces, it indicates they were set in the solution too long, take them out and replace with new zinc strips.

Zinc powder cementation process

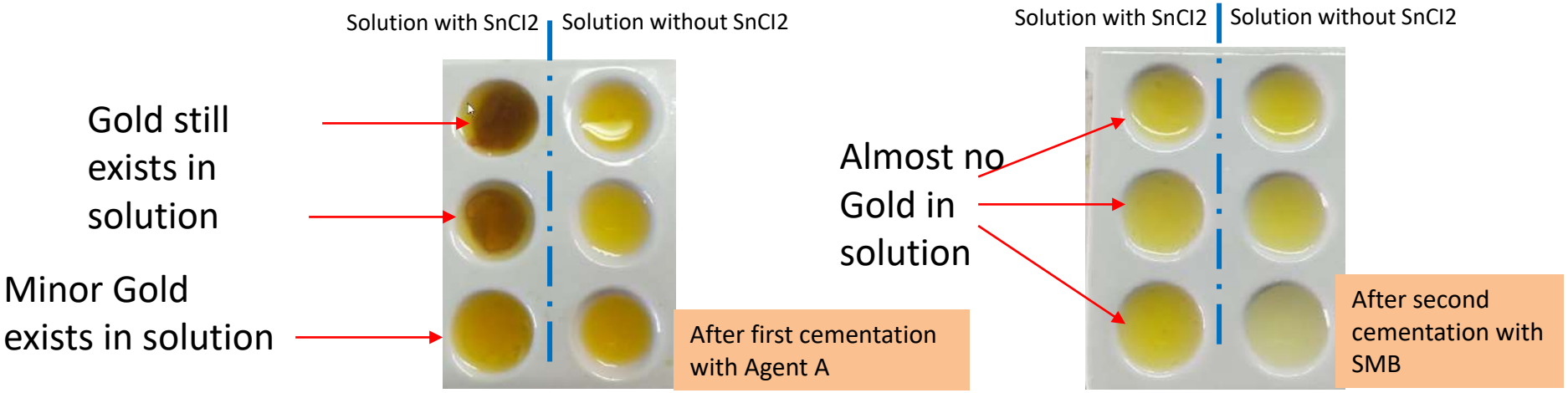
Filtered solution for the 2nd step process

Second stage precipitation

In the second cementation stage, solution pH is still need to be adjusted before adding zinc cementation due to the fact that the first zinc cementation process will bring the preg solution pH back to ~6.0 .

the second stage precipitation process just the same procedure as the first step. Usually, solution color shall be cleared out after the second step cementation.

Stannous chloride solution coloring test is a good indicator whether gold precipitation is complete or not as shown below.



Multiple stage zinc strip precipitations are recommended to complete gold precipitations

Tips/comments:
For small scale and time sensitive operators, zinc powder is recommended. For large scale and time none-sensitive operators, zinc strips cementation is recommended.

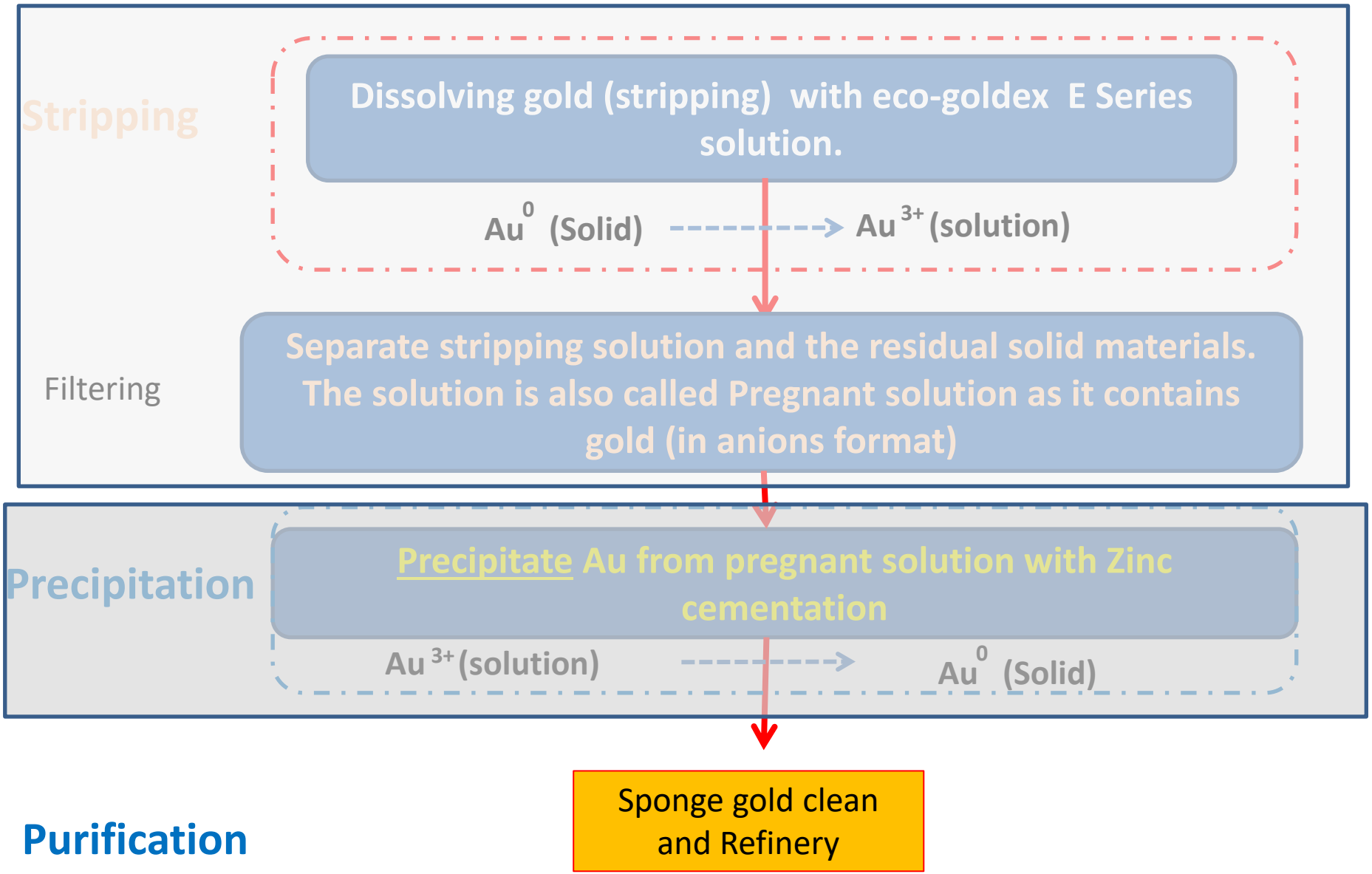
Zinc strips cementation usually much slower than the zinc powder, but zinc strips doesn't need filtration. gold will precipitate on the zinc strips (darkening). If there is no gold in the treated preg solution, you will see zinc strips won't change color no matter how long zinc strips are in the solution.

Muraitic Acid
adjusting PH

Color change of preg solution and zinc strips in the cycle of gold precipitation.

Basically, when zinc strips color won't change in over 20 hours, it indicates not much gold left in solution, and at the same time, solution should become clean and cleared out.
If solution pH raise over 5 during cementation, continue using HCl to lower pH to maintain effective cementation

Major Gold Stripping/Precipitation/Recovery Steps



Purification

You are here!

Loaded zinc strips/powder process

The purpose of this step is to remove residual (extra) zinc not completely reacted in the preg-solution and other base metals such as Cu, Ni that might be also precipitated in the cementation process. Zinc powder precipitation can be processed the same way. This step will improve gold purity accordingly. Here are the steps conducting zinc strip dissolving and cleaning:

1. Gently rinsing loaded zinc strips using clean tap water to remove residual stripping solutions;
2. Dissolve loaded zinc strips or filtered sludge (from zinc powder) using diluted HCl (~20%) or H_2SO_4 (20-30%). (be aware of possible Ag losing if you use HCl!, if your material contain considerable Ag and be cautious AgCl sediment at the bottom of the container!)

Slowly add diluted HCl or H_2SO_4 solution prepared into the pail to dissolve loaded zinc strips

Gentle stirring the solution till all Zinc strips are dissolved and become small tiny particles

!! Make sure this dissolve process long enough to dissolve all the extra zinc!!
If extra zinc is not removed completely and carried over to next step, the zinc sludge in cooking stage will be very difficult!

Add sufficient clean water (10-20 times) to dilute sulfuric acid to prepare filtering. Rinse several times before final filtering. Strong sulfuric acid will damage the filter paper if not diluted properly

Filtration of the primary gold dust

These tiny black dusts are primary gold with impurity

Make sure drying this dark sludge as much as you can to remove water from it.

Treatment of Black Gold Sludge

Till then, you have recovered most of your gold together with some impurities such as excessively stripped copper or undissolved zinc in the black sludge.
While military E-scrap materials or very high grade Materials may form reddish and coarse sponge gold, Most E-scrap materials stripping will form black gold sludge. These black color particles indicate impure gold in the sludge.
From here, you have two options to get your gold from the black gold:

Option 1:

Dissolve the black sludge cake in aqua regia, and recover gold with the conventional SMB method.

Option 2:

Using diluted HNO_3 to wash the black sludge cake in a heated container (glass Becker or ceramic) for a few minutes to remove extra impurities. Filter the solution and wash the residual part (gold) with hot water several times. Then melt the purified gold to get your gold.

Difference between black gold sludge with/out diluted acid washing(with elevated temperature)

Note 2:

Both HCl, H2SO4 are capable for extra(residual) zinc removal after gold precipitation process is completed. Here is the ratio to make diluted acid solution to wash loaded zinc strips:

- ✓ *Diluted HCl solution:*
  HCl : water = 1:1~1.5 (muriatic acid, most commercial HCl concentration is 30%).

- ✓ *Diluted H2SO4 solution:*
  H2SO4 : water = 1: 6~8. (most commercial H2SO4 concentration 96% or 98%)

Once extra zinc strips/powder are completely dissolved (no more gaseous bubbling and reactions), make sure using plenty of clean water to wash and dilute solution and then let the solution settling for separation.
In many cases, there are too much tiny black particles suspending in the solution and hard to settle, a few drops of coagulation agent solution (agent C) can help a quick and complete separation of solution and the fine particles .

Appendix

Au-Ag-Pt-Pd-Rh Separation

Au-Ag-Pt-Pd-Rh Separation

In the E-waste recycling, it is common, the electronic components contain certain amounts of multiple precious metals such as Au, Ag, Pd, Pt. in the automobile catalyst converter, Pd, Pt Rh are the major component metals. These metals are stripped off from the e-waste . Catalyst converter powder together and precipitate together.

So an effective method of how to separate them is important.

Below this table shows the precious metals precipitated with the zinc cementation method and chemical precipitation method.

Precipitation method	Preg solution (liter)	Precipitated gold ingot (grams)	Ingot composition (%)							
			Au	Pt	Pd	Ag	In	Ir	Cu	Zn
Zinc cementation	2.2	10.40	93.75	0.00	0.34	0.98	0.00	0.00	4.26	0.37
Chemical Precipitation	2.2	10.03	94.75	0.00	0.00	1.34	0.03	0.00	0.17	0.40
Zinc cementation	1.0	1.39	87.59	1.21	1.19	2.40	0.04	0.15	6.36	0.79
Chemical Precipitation	1.4	6.34	94.67	0.00	0.00	1.22	0.00	0.00	0.27	0.00

Many users don't know how to separate these metals to get single metal powder for futher smelting. The next page shows the methods and steps how to separate these metals from the metallic alloys/sludge.

Au-Ag-PGM Alloy

(ideally to make tiny metal beads or do this separation directly with the precipitation sludge for better dissolving)

Dissolving sludge /metal beads using HNO3

Ag/Pd in solution

Au/Pt /Rh in solid

Add NaCl/HCl

Add Aqua Regia

Filter solution

Ag in solid

Get Ag

Au/Pt in solution

Rh in solid

Get Rh

Add Formic Acid

Add SMB

Pd in solid

Get Pd

Pt in solution

Au in solid

Get Au

Add $NaHB_3$

Pt in solid

Get Pt