Chemical Precipitation Method and Procedures of gold precipitation from Eco-Goldex E series Preg solution

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Chemical precipitation is one of the three methods that Eco-goldex has developed for gold precipitation in E series derived preg solution.

We recommend the chemical precipitation method to gold hobbyist, occasional operators as it is simple and quick to finish.

Chemicals are used in this method to precipitate Au(Ag) in E series preg-solution.
1. Agent A (caustic soda flakes or Potassium hydroxide (KOH) flakes): used to adjust preg solution pH to desired pH range;
2. Agent B: used to precipitate gold/silver from the pH regulated preg solution;
3. Agent C: flocculate used to accelerate precipitate gold/silver in preg solution.
4. Stove heater

For the chemical precipitation package user. The agent B in the package is for onetime small quantity test purpose only.

!!!DO NOT expect to drop gold in more than 5 liters of pregnant solution with the amount of agent B given in the package, or you will be ended up with gold partially dropped while the agent B is run out halfway!!!

Too many users have dropping problem just because they run out of agent B in this method.
A few key notes for chemical precipitation method:

- In chemical precipitation method, the higher gold concentration in the preg solution is, the better precipitation result will be. So try to maximum gold concentration in the stripping solution before stripping is ended.
- Chemical precipitation should be conducted in raised temperature in which preg solution temperature should be maintained at around 70-85°C.
- Preg solution pH should be adjusted using the agent A with the ratio recommended in the procedures described after (20 grams agent A in 1 liter of preg solution to be treated);
Chemical Precipitation Procedures

1. Preg-solution filtration (to remove solid impurities (residuals) formed in stripping process). This step is also critical. Preg solution must be filtered, clean and clear before entering in chemical precipitation process!!!

2. Raising preg solution temperature to 70-85 °C range.

3. While solution temperature is brought up, Slowly adding agent A into preg solution to adjust preg solution pH with the ratio recommended below; make sure stirring solution while adding agent A to avoid overflow.
  ➢ 20 grams of agent A per liter of pregnant solution to be used for preg solution conditioning

4. Slowly add agent B while stirring solution; solution color should be changed from dark red (orange) to dark greenish and form foam/bubbling at certain point. Keep adding agent B till no bubbling is formed when Agent B is added. By then, the solution should be turning clear out at top. Stop add Agent B in solution.
   ➢ 20 grams of Agent B per liter of pregnant solution to be used for gold precipitation. (this ratio can be changed if preg solution is very high/low in gold concentration, use reaction color to justify when to stop as described in this document)

5. Add a few drops of agent C solution to accelerate precipitation; settle down solution for a few hours before filtration.

6. Filtration of settled solution, retain the solid sludge for further cleaning with hot water 1-2 times.

7. Combine all solid sludge and smelt in furnace to get gold dore.
Step 1: Raising Preg solution temperature

- Sit preg solution in a beaker or a ceramic container on a stove and raising solution temperature to around 70-85°C;
- If you consider gold concentration in preg solution is not very high, you may raise solution temperature further higher to boiling point to evaporate part of water to bring up gold concentration in solution in another way.
- maintain preg solution temperature around 80 °C.

One more time reminding:
Use ~5 liters of pregnant solution do the test with chemical method as the quantity of agent B is limited.
Step 2: preg solution pH adjusting

- While maintaining preg solution temperature around 60-85°C,
- Add agent A into the preg solution (slowly) while stirring to make sure agent A is completely dissolved quickly to avoid overflow (too much agent A add in one shot will cause overflow).
- The amount of agent A added in the pregnant solution is 20 grams per liter of solution (i.e. for 5 liters of pregnant solution, you would need to add $5 \times 20 = 100$ grams Agent A to be added).

When starting, Preg solution usually has pH at around 10.5

(notes: pH measurement is NOT critical in the chemical precipitation method, the RATIO of agent A to the volume of solution is important)
**Step 3: Adding Agent B**

- Once the pregnant solution is conditioned with Agent A, slowly adding agent B to the pregnant solution while keeping vigilant stirring in solution. **Amount of agent B should be added in small quantity to avoid bubbling and overflow.** For first time test, it is recommended to fill beaker less than half depth to avoid possible overflow.

- With more agent B is added, Preg solution color will start change to dark or dark greenish and black particles start to form in solution. The black particles are gold.

- Keep adding Agent B till no bubbling is formed when agent B added and solution color cleared out to light greenish on top of the container.

- The total amount of Agent B to be added is about the same ratio as agent A, which is 20 grams Agent B in 1 L pregnant solution in normal situation, when gold concentration is low in the preg solution, this amount to be used can be less and agent B reaction with the solution can be used as indicator to justify when to stop adding Agent B as described below.

- It is ok to boil the preg solution for about 1-2 minutes after Agent B is added.

- Add a few drops of flocculate solution (Agent C) to accelerate precipitation. Let the solution settle for about 1-2 hours to complete precipitation.
Preg solution color change sequence after Agent B is added

Add agent B

solution temperature while agent B is added
Tips of how to judge Agent B adding in solution to complete gold precipitation

When agent B is added, strong golden color foaming formed indicates a lot of gold in solution.

When agent B is added, golden color foaming formed at the edge indicates still some gold in solution.

When agent B is added, only white grey color foaming formed or no(weak) foaming is formed at the top indicates no gold in solution. At this point, you may stop adding Agent B, and let the solution settle down.

!! This visual checking is a back up of the 20 grams Agent B per liter solution ratio role.!!
Adding Agent C (optional)

- Agent C is a strong flocculate and can help accelerating precipitation of fine gold particles in the preg-solution and should accordingly shorting gold precipitation time.
- 1 gram of Agent C adding into 1 liter of distilled warn water and set for 2 hours, then it is ready to use.
- Quantity of agent C solution can’t put too much, only about 1-2‰ (volume) of the preg solution. If too much, the precipitation stuff will be too clamminess to clean later on.

As long as agent B precipitation result is good (obvious big black gold particles are formed), Agent C step can be skipped. If not, then Agent C solution is recommended.

Before Agent C solution added in preg solution

The preg-solution should be settled down quickly.

Precipitation Complete settle down
Original preg-solution

After agent B added in preg-solution

Complete settlement of precipitation in preg-solution

After PAC added in preg-solution
Difficulty situation treatment

When gold concentration in the pregnant solution is too low or the pregnant solution was not filtered properly (not clean), agent B doesn’t create good (black) precipitation in the solution, what you see is that the solution becomes dark, but no black particle precipitation, the following method can be used to help recuperating the fine gold in the pregnant solution:

Use citric acid to adjust the dark solution while stirring to adjust solution pH to 5-6, then you should be able to see black fine particles to drop from the solution, add Agent C solution to help this process.
Key Notes for Chemical Precipitation method

• **Preg solution temperature is KEY. It should be raised to about 70-80 °C;**

• **The higher gold concentration in the preg solution, the better performance of the chemical precipitation method. This is why we recommend to evaporate water from preg solution by heating if it is possible (as much as you can);**

• **Both agent A, B should be slowly and small amount (multiple times) added in the preg solution to avoid bubbling in the container (beaker) and overflow.**

• **The agent weight ratios of Agent A, B to be used in the preg solution is the same: 20 grams per liter solution.**

• **Agent C solution can help fine particle precipitation.**
Precipitation Cleaning

- After complete settled down, either filter the precipitation or just pour out the top clean solution and leave the precipitation (sludge) in the beaker;
- Add HOT WATER to (cook) wash the precipitates in the beaker, and let it settle down and repeat the filtering process (2-3 times are recommended);
- Filtering the solution and retain solid sludge.
- To remove the impurity in the black gold, put the solid sludge gold in a beaker and ten add proper amount of Sulfuric acid (H₂SO₄) (the acid should merge the sludge) and bring the solution to boiling once. Pour out the solution and wash the sponge gold with clean water twice then switch to gold smelting.
- It can also be cleaned using diluted HCl/HNO₃ to remove other metals such as Cu that might been precipitated by agent B.
Au-Ag-Pt-Pd-Rh-Ir 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.

<table>
<thead>
<tr>
<th>Precipitation method</th>
<th>Preg solution (liter)</th>
<th>Precipitated gold ingot (grams)</th>
<th>Ingot composition (%)</th>
<th>Cu</th>
<th>Zn</th>
</tr>
</thead>
<tbody>
<tr>
<td>Zinc cementation</td>
<td>2.2</td>
<td>10.40</td>
<td>Au 93.75</td>
<td>0.00</td>
<td>0.34</td>
</tr>
<tr>
<td>Chemical Precipitation</td>
<td>2.2</td>
<td>10.03</td>
<td>Pt 94.75</td>
<td>0.00</td>
<td>0.00</td>
</tr>
<tr>
<td>Zinc cementation</td>
<td>1.0</td>
<td>1.39</td>
<td>Pd 87.59</td>
<td>1.21</td>
<td>1.19</td>
</tr>
<tr>
<td>Chemical Precipitation</td>
<td>1.4</td>
<td>6.34</td>
<td>Au 94.67</td>
<td>0.00</td>
<td>0.00</td>
</tr>
</tbody>
</table>

Many users don’t know how to separate these metals to get single metal powder for further smelting. The next page shows the methods and steps how to separate these metals from the metallic alloys/sludge.
Au-Ag-PGM Alloy

Dissolving sludge/metal beads using HNO3

Ag/Pd in solution

Filter solution

Add NaCl/HCl

Ag in solid

Add Formic Acid

Pd in solid

Au/Pt in solution

Au/Pt /Rh in solid

Add Aqua Regia

Rh in solid

Add SMB

Pt in solution

Add NaHB₃

Au in solid

Pt in solid

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