

# Electrochemical reactor Flexicone.



**User instructions**

## **Important safety information!**

**Please use reactor in well ventilated area.**

**Reagent supplied in container consist from few several different components not mixed properly, please mix properly in dry condition before add water.**

**Reagent classified as not dangerous and toxic (0/0/0) in dry and wet condition before use.**

**After applied electrolysis process , solution become corrosive and can produce toxic gases .**

**Keep rectifier current below level , when gases start generated.**

**Keep electronic and electric devices away from solution.**

**Use gloves and glasses , when working with chemicals.**

The reactor is designed to intensify chemical processes and more fully recover precious metals from refractory ores and concentrates, as well as sulphide and gold-silver tellurides of minerals at normal atmospheric pressure and temperature without the use of toxic reagents. It can be used for the quick extraction of precious metals from any rock including double-hardness ores, finishing finishing of the concentrate, as well as for quick assay analysis with a gold content of more than 0.1 g / ton of ore.

The reactor is a device 3 in one. Consists of units: directly from a reactor vessel for an electrochemical process with a power source and electrodes, a solution filtration unit and a solution receiving tank. In the receiving tank there is a drain pipe with a ball valve and a vacuum pipe. A diaphragm pump is used to create a vacuum for quick filtration and also used as air mixing of pulp in reactor .



## **Advantages of using the reactor and Flexicone sulphide oxidation and leaching technology:**

Highest percentage extraction of gold from ores compare to other methods.

Non-hazardous , non-flammable chemical reagent - classification (000)

Inexpensive reagent.

Leaching of noble metals takes place in less chemically aggressive conditions. Concentration and consumption of chemicals are reduced.

This is achieved due to the flow of the electrochemical process .

The possibility of stepwise leaching of metals.

Universality of technology and applicability for different types of ores and concentrates.

Simplicity of control and process control by voltage/current regulation on electrodes.

High wear resistance design, quick change and low cost of consumables.

**Specification :**

Reactor:	FLR14.	TR250
Volume of reactor, l	14	250
Size LxWxH, mm	400x500x350	650x650x1900
Weight, kg	7	40
Power supply rectifier		
input voltage ,VAC	220	220
output Voltage,VDC	7-15 (adjustable).	0-24 (adjustable).
output Current, A	0-20(adjustable).	0-20(adjustable)
Membrane pump:		
Voltage, VDC	7-15	24
Current , Amp	6	15
Power ,Watts.	72	360
Capacity, l/min	6	30
Pressure,mPa	0.9	0.9
Turbo drive, kW		0.25
Voltage ,VAC		220

**The process on FLR14 consists of 4 stages**

1. leaching-carried out in an active reactor
2. filtration - carried out on a suction filter
3. precipitation of gold from the filtered solution
4. reagent regeneration

A diaphragm pump is used to pump clean chemical solution.

**Reactor preparation:**

1. Place the flat cathode first on perforated bottom
2. Place the filter (orange)
3. Connect the flat cathode with - power
4. Connect the central anode with + power ( Anode must be inserted in pulp on half deep )
5. The pump is used for liquid and vacuum pumping to work suction filtration
6. The power supply has voltage and current regulation.

**Preparation of ore and iodine-iodide leaching process**

1. Grind ore to a size of 70 microns
2. To speed up the leaching process, it is recommended to pass the ore through a concentrator to isolate a large fraction of gold

3. The head of the concentrate for melting, tails for leaching
4. For complete extraction before leaching, sulfide and refractory ores are also opened by the electrochemical in the reactor, without oxidation of refractory ores, the extraction can be 50%-80%.
5. For the oxidation of ore, we prepare an oxidising solution: from the calculation, the ratio of ore to solution is 2 : 1 by weight. The oxidising process and reagent are selected according to the technological scheme based on the composition of the ore. (Information is provided additionally)
6. We prepare the leaching solution: from the calculation, the ratio of ore to solution is 1: 1 by weight, for this we prepare a new solution or use the last washing filtrate after leaching, reagents from 30 to 250 g per litre of pure water or the last washing filtrate (concentration of the reagent in the solution depends on the metal content in the rock).  
Reagents are mixed in the beginning in a small volume of water. Then the entire volume of the solution is added. The pH of solution is regulated by the addition of alkali,
7. The prepared solution is loaded into the reactor. The pulp is mixed by air bubbling supplied from air pump. Solution from receiver tank must be returned back to reactor during leaching process. The process lasts from 60 to 240 minutes (the leaching time is determined by trial and depends on the size of the gold particles and the type of ore. When concentrates with a high gold content are leached, the concentration of the solution increases.  
The pH of the solution is ???, The cell voltage is ??? V, the temperature is 20-40 degrees C  
If non-ferrous metals are present in the ore (copper, zinc, lead), an additional reagent may be required. To reduce leaching reagent consumption pre-oxidation of ore is highly recommended. Leaching process is controlled by the colour of the solution, which should be dark brown.  
Attention!!! With pH greater than ???, the gold precipitates from solution. It is necessary to add hydrochloric or sulfuric acid to lower the pH to ???.
8. The gold content in the solution is controlled by an indicator reagent (optional) according to the colour of the solution. The minimum gold content in the solution to start displaying 0.5 mg of gold per 1 litre of solution.
9. Upon reaching the maximum extraction of gold into the solution, the voltage from the electrodes is turned off, 50-100 mg of a 30% hydrogen peroxide solution is added in small portions, the pulp is mixed for 5 minutes, the pulp is filtered off (a minimum filtration time of 10 minutes is required to dissolve the settled gold at the cathode). The cake is washed with clean water.
10. The filtrate enters the regeneration in a separate two-chamber electrolyzer for the extraction of precious metals and regeneration of the reagent (optionally supplied).
11. If desired, when several precious metals are present, they can be deposited simultaneously or separately. A solution containing gold, silver, platinum, palladium and rhodium, dissolved in the electrolyzer and filtered from the base material, is alkalized (pH about 8); and all gold, silver and palladium in the filtrate are simultaneously precipitated by the addition of an alkaline reducing solution of sodium borohydride.
12. If separate extraction of metals is necessary, gold is first extracted, which precipitates from the solution by passing sulfur dioxide or sodium pyrosulfite until gold is completely precipitated and then filtered off. The acidic filtrate containing now dissolved silver, platinum, palladium and rhodium is made alkaline (pH about ????) and then treated with an alkaline hydroxylamine solution to precipitate silver, which is then filtered off. The remaining alkaline filtrate, now containing only platinum, palladium and rhodium, is then treated with an alkaline solution of sodium borohydride to precipitate palladium.
13. If there is only gold, the filtered solution is heated to boiling point and 3 g per liter of hydrogen peroxide is added. Then the gold settles for an hour. After drying, the gold precipitate is melted.
14. The solution is regenerated by the addition of hydrochloric acid to pH ????? and hydrogen peroxide or enters the regeneration in a separate two-chamber electrolyzer and enters the next cycle

## Cases studies

### 1. Rock processing

Quartz-sulfide arsenic ore with a gold content of 50 g / t is ground to a size of 0.1 mm.

Ground ore is processed through a Flexicone centrifugal concentrator to recover free gold and silver. A concentrate with a gold / silver content above 5% can be directly melted. For the best collector melting with a lower content, we recommend the Flexicone induction furnace with ultrasonic deposition in the crucible.

Concentrated tailings are treated using a FLR14 leach reactor

An aqueous 8% reagent solution and ground quartz ore are poured into the apparatus. The ratio of T: W is 1: 1. The resulting pulp is processed for 180 minutes in the electrolyzer mode to extract gold from the rock. To intensify the process, it is recommended to mix the pulp with a air bubbling . Gold and silver are leached from ore. Then the pulp is filtered. The cake is washed with water. The resulting clarified solution is mixed with the main filtrate for chemical precipitation of gold or sent to leach the ore of the following material. For chemical precipitation, the filtrate is poured into a stainless steel tank, brought to PH????? with the addition of caustic alkali. Heated to boiling point and added per liter of solution 3 g of hydrogen peroxide. Then the gold settles for an hour. The precipitate of gold after drying is melted

The degree of extraction of gold and silver is up to 99.8%

### 2. Analysis of the ore for gold content

A sample of finely ground ore with a gold content of 0.1 g / ton in an amount of 1 kg of is mixed with 0.2 litre of a 10% reagent solution. The resulting pulp is processed for 180 minutes in the electrolyzer in the mode for rock analysis and with electrodes for analysis. To intensify the process, it is recommended to mix the pulp . The gold content in the solution is controlled by an indicator reagent (optional) according to the colour of the solution. The minimum gold content in the solution to start displaying 0.5 mg of gold per 1 litre of solution.

Analysis accuracy + - 20%.

### 3. Removing precious metals from electronic waste

A material was received for processing, which is a product obtained by mechanical enrichment of electronic scrap through closed-loop crushing, screening, air classification followed by separation of the metal fraction by magnetic and electrostatic separation in an amount of 1 kg, containing (in %), Au 0.52 ; Ag 2.20; Sn 15.9; Pb 7.48; Zn 0.36; Cu 27.60; Ni 0.20; Fe 5.00.

The separation of tin, lead, aluminium, and part of zinc from scrap was carried out by electrochemical dissolution in 10–20% alkali solutions with anodic polarisation of scrap. Leaching was carried out in an electrolytic cell with a stainless steel anode into which scrap was loaded and a cylindrical cathode made of the same material with constant stirring and removal of excess heat. The process was constantly monitored by changing the weight of the scrap. At a current density of 300 A / m<sup>2</sup>, a current strength of 40 A, a ratio of solid to liquid (T: G) = 3 and (room) temperature of 50-70, the process ends in 6 hours, while the degree of extraction of tin, lead and aluminium and zinc into the solution respectively (c) 92, 80, 65, 55 (the concentration of elements in the solution is equal to 47.38; 20.2; 2.11; 0.67 g / l). At the same time, tin-lead powder was deposited on the cathode, gradually crumbling to the bottom of the cell. 18% current efficiency

After washing the leached scrap from alkali, its weight was 756 g. The electrolyte was filtered from tin-lead powder and reused. As aluminium and zinc accumulate in the electrolyte, part of the solution is removed from the process and purified from them by precipitation of sodium aluminate by cooling the solution to 16-18 degrees and subsequent cementation of zinc on iron chips. The purified electrolyte is returned to the process head.

The gold-containing electronic scrap was cleaned of copper and non-alkali insoluble non-ferrous metals by electrochemical leaching of these elements into a copper-ammonia etching solution or a composition based on monoethanolamine with the simultaneous separation of powdered copper at the cathode. The process was carried out in the previously described electrolyzer with constant monitoring of the loss in weight of the loaded scrap. At a current density of 200 A / m<sup>2</sup>, T: F 2, constant air purging and room temperature, the process ends in 6 hours, while the degree of extraction of copper, nickel and silver into the solution was (c) 98, 90, 73, respectively. The residual copper content in solution after electrolysis 10 g / l. After washing the leached scrap from a copper-ammonia pickling solution, its weight was 468 g.

The electrolyte was filtered from copper powder and reused. As silver and nickel accumulate in the electrolyte, part of the solution is removed from the process and after ammonia is distilled off, silver chloride is liberated from it and nickel is cemented. The solution is supplemented with ammonium salt, ammonia and returned to the head of the process.

Cleaning scrap from iron was carried out by the method of sulfuric acid leaching in the presence of an oxidising agent, followed by precipitation of iron hydroxide.

Thus prepared material (scrap) was processed in accordance with example 1.

### **The process of the electrochemical chlorine-hypochlorite method of gold extraction when the anode is located directly in the pulp.**

A low-hazard substance is used as a reagent for chlorine extraction of gold: sodium chloride (hazard class 5). The economy of the process is determined mainly by the cost of electricity and anodes.

#### **Description of technology**

The transfer of metals (Au, Ag, etc.) from gold-bearing raw materials into solution occurs under the action of sodium hypochlorite NaClO. Sodium hypochlorite is produced by electrolysis of a sodium chloride solution. The advantages of direct production of sodium hypochlorite solution from sodium chloride are relatively low costs, simplicity of the process, and the possibility of using technical unrefined table salt. This recovers about 50 mg of gold and up to 720 mg of silver with each liter of leach solution containing sodium hypochlorite. For a more complete extraction of gold, a gold extractor is introduced, which contributes to the wetting of the rock and accelerates the dissolution of gold, slows down the processes of re-deposition of gold on impurities of iron and sulfide minerals. The process is possible both in percolation and agitation modes. In alkaline solutions in an oxidizing environment, iron is passivated and remains in a pile or tray. Since sulfide ores are oxidized faster, for ores with a high content of sulfides and iron, the mode of chloride-free oxidation of oxidizing impurities (iron, sulfide ores) can be used by feeding a solution with preliminary aeration of the electrolyte during electrolysis. Due to the oxidizing environment, sulfides and iron scrap are oxidized. The specified environment ensures the oxidation of impurities without dissolving gold and silver. A solution with a gold concentration of up to about 50 mg / l, when passing through the cathode, is regenerated with the deposition of gold on the cathode and is drained by gravity into a receiving vessel. The filter also separates impurities in an alkaline medium. Under the action of an electric current at a negative potential of the cathode, metals are precipitated on the electrode in the form of deposits. The electrode potentials for the release of gold and silver at a concentration of 1 mol / L in the eluate are -1.089 V and -1.788 V, respectively. From the cathode, impurities are washed off with sulfuric acid, and precious metals are washed off with aqua regia. To carry out the process, a sealed regeneration module is used.

#### **Preparation of reagents**

##### **Preparation of gold extractor solution**

An aqueous 10% sodium chloride solution is prepared in a container for mixing reagents. The prepared solution is fed into the reactor by a metering pump in percolation mode.

##### **Chlorine disposal**

The alkaline catholyte formed as a result of electrolysis is fed into the upper part of the reactor. This eliminates the ingress of chlorine from the reactor into the air of the working area and into the environment.

The solution to the arsenic problem

Arsenic is predominantly concentrated in the sludge containing iron at the electrolyzer cathode. After the accumulation of the sludge, it is treated separately, with the leaching of the arsenic solution and the release of arsenic in the electrolytic cell in metallic form. The isolated arsenic is separated from the solution on a suction filter, then dried with a washing solution, and dried in a vacuum. The wash solution is in recycling, it is recovered by recuperation. Metallic arsenic, after extraction, is subject to protection from oxygen and moisture in a plastic vacuum package at the filling unit and is to be used to obtain alloys in the production of photovoltaic cells.

Rare earth metals

Rare earth metals are concentrated in the eluate (sorbent regeneration solution), they are released during the extraction of precious metals and for their extraction additional columns for sorption extraction are installed, tanks for precipitation, a suction filter for precipitation separation, a precipitate drying unit, an oven for calcining the precipitate to obtain a concentrate.

### **Thiourea leaching of gold and silver**

To dissolve metallic gold in aqueous solutions of thiourea, two conditions must be met:

the use of a sufficiently effective oxidizing agent capable of converting metallic gold into an ionic state and not oxidizing thiourea;

ensuring the acidity of the medium within the range of  $\text{pH} = 2-4$  in order to protect thiourea and the resulting complex compound of gold from decomposition.

These conditions are achieved by introducing the required amount of sulfuric acid and ferric salts  $\text{Fe}_2(\text{SO}_4)_3$  into the solution.

The normal redox potential for the gold dissolution reaction is 380 mV. The leaching rate depends on the pH of the solution, the concentration of thiourea and the oxidizing agent. The main reaction of dissolution of gold and silver in thiocarbamide is expressed by the equation:  $\text{Au}^0 + 2\text{CS}(\text{NH}_2)_2 + \text{Fe}^{3+} = \text{Au}[\text{CS}(\text{NH}_2)_2]_2^+ + \text{Fe}^{2+} + (1)$ .

As can be seen from the equation, sulfuric acid present in solutions does not participate in the reaction and does not affect the rate of dissolution of gold in thiourea. Its use is associated with the need to preserve the thiourea complex of gold, stable only at  $\text{pH} = 4$ , as well as to prevent the hydrolysis of ferrous sulfate.

A twofold increase in the concentration of thiourea in solution from 0.4 M to 0.8 M does not significantly affect the dependence of the degree of gold recovery on the reaction time. So, for example, if within 5 hours from the beginning of the process 63.4% is extracted with solvent 1, then only 41% is extracted with solvent 2. For 10 hours of leaching, 93.4 and 83.3%, respectively, while by the end of the process (for 14–16 hours), the degree of recovery becomes equally high - 96%.

The degree of gold extraction from ammonia-thiosulfate solutions was 55–65%, and even less from hypochlorite solutions. Some samples were practically not opened with hypochlorite.

Based on the results obtained, the following technology for processing gold-bearing concentrates can be proposed, which includes several main stages:

- agitational leaching of concentrates;
- washing of leach products with circulating de-gold solution and rinsing water;
- electrochemical separation of noble metals from productive solutions;
- drying and calcining of the cathode deposit;
- sorption capture of precious metals from gold-free electrolyte;
- neutralization of acidic water-tail solutions with  $\text{Ca}(\text{OH})_2$  milk of lime;
- storage of tailings in a bulk-type tailing dump and full or partially closed water circulation from the tailing dump. The finished product is a Dore alloy.

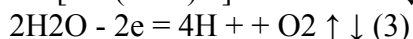
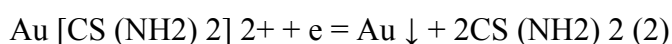
The original gravity concentrate enters the leaching stage with a thiourea solution containing thiourea, sulfuric acid, ferric salt, the ratio S: L = 1: 2. The pulp is fed to the suction filter for filtration. The solid precipitate is washed with a leaching solution, then with water. The washing solutions go to the stage of preparation of the leaching solution. The leaching cake is stored in a dump. The productive solution containing at least 100 mg / l of gold from the filtration stage enters the cathode space of the electrolyzer. ... The electrolyzer works to deplete the solution with or without electrolyte circulation, depending on the temperature regime. The spent catholyte containing 10-30 mg / l of gold from the electrolyzer enters the stage of preparation of the leaching solution, having previously passed the stage of sorption of residual gold on activated carbon. Gold is periodically stripped from coal, after which the coal is regenerated. Part of the spent solution is periodically withdrawn for cleaning from accumulating impurities. Cleaning is carried out with a solution of milk of lime, while ions of impurity metals form insoluble hydroxides, and calcium ions pass into insoluble sulfate. After filtration, a mixture of metal hydroxides and calcium sulfate enters the dump, and the filtrate containing thiourea enters the solvent preparation stage. A 0.1-0.5% solution of sulfuric acid enters the anode space, circulating in a closed loop. If necessary, a refrigerator is installed to reduce the temperature of the anolyte (not shown in the diagram). The black cathode deposit, consisting of finely dispersed gold, oxides and sulfides of impurity metals, is removed periodically with the electrolyzer turned off. The precipitate goes for filtration to the suction filter, on which the precipitate is removed from the cathodes. The filtrate is combined with the catholyte.

The filtered cathode precipitate is fed to the drying and calcination stage at 900 ° C and then to the melting stage in an induction furnace. The melt is poured into a cast iron mold, and the slags formed by fluxes (Na<sub>2</sub>CO<sub>3</sub>, Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>) and impurities consisting of oxides, and under them there is a Dore alloy (70–98% gold).

At the stages of drying and smelting, sulfides are oxidized, and the resulting gases, mainly SO<sub>2</sub>, CO<sub>2</sub>, NO<sub>2</sub>, NH<sub>3</sub>, are absorbed in the milk of lime in the absorber. Periodically, the sediment from the bottom of the absorber is removed to the dump.

In the proposed technological scheme, the thiourea solution is used in a closed cycle, which significantly reduces the operating costs for leaching and disposal of waste.

During the electrolysis of thiourea gold-containing solutions, the main reaction of gold deposition occurs at the cathode (reaction 2). At the anode, in addition to the main water oxidation reaction (reaction 3), thiourea is oxidized with the formation of dithiosulfide forms, which further decomposes, giving thiourea, cyanamide CNNH<sub>2</sub> and elemental sulfur (reaction 4):



Thus, there is a decrease in the content of the complexing agent, contamination of the metal precipitate with elemental sulfur, which, during drying, calcination and smelting, releases gaseous sulfur dioxide, which imposes certain requirements on the purification of process gases. In addition, reaction 4 consumes a certain amount of electricity. To eliminate these disadvantages, it is necessary to separate the cathode and anode spaces using membranes. Required circulation of the productive solution through the common cathode space with the deposition of gold on the cathodes, and circulation of the anolyte with the release of oxygen at the anode. Sulfuric acid solutions with good electrical conductivity are used as anolyte. We have developed several types of electrolyzers and installations based on them with different current loads and different degrees of automation of the electrolysis process.

All information is confidential. Provided only to a specific user and is not allowed to transfer to third parties without endorsement.

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