

# Determination of the Degree of Regeneration of Fluoride Ammonium

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**ABSTRACT.** The invention relates to the field of hydrometallurgy of precious metals and can be used to extract gold from the tails of gold mining factories. The objective of the invention is the additional extraction of gold from the tailings of ores and beneficiation products into commercial products by reducing the sorption activity of the solid phase by increasing the temperature of the pulp.

**KEY WORDS:** tail, fractional composition, processing, slag, crushing, cyanide

## I. INTRODUCTION

Currently, studies are underway in the world to extract useful components from off-balance gold and copper ores, as well as industrial waste in a number of priority areas, including: heap leaching for balance gold and copper ores; biotechnological method for processing dumps of sulfide ores; processing of zinc clinker by electrochemical method; processing of tailings of hydrometallurgical plants; processing of slag from pyrometallurgical plants; increasing the number of output as a result of ore beneficiation and processing of waste from gold mining factories.

To determine the degree of regeneration of ammonium fluoride formed during the separation of hexafluoride ammonium silicate (HFSA), the material balance of the separation process [1] is presented in Table 1

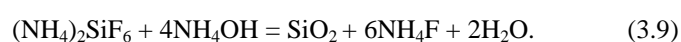
**Table 1**The material balance of the process of separation of GFSA

Name, Kg	Arrival, kg	Name	Consumption, Kg
Tails	100	(NH <sub>4</sub> ) <sub>2</sub> SiF <sub>6</sub> ,	187,8
Quartz	63,3	NH <sub>3</sub>	71,7
Water	2	water	38 +2
NH <sub>4</sub> F	234,2		
Other	34,7	other	34,7
Total:	334,2	Total:	334,2

In the table. 2 shows the molecular weights of the used and forming substances in the course of the reaction.

**Table 2**Molecular Weights of Used and Formative Substances

Molecular Weight	Molecular Weight gr
(NH <sub>4</sub> ) <sub>2</sub> SiF <sub>6</sub>	178
NH <sub>4</sub> OH	35
NH <sub>4</sub> F	37
SiO <sub>2</sub>	60
NH <sub>3</sub>	17
H <sub>2</sub> O	18
NH <sub>4</sub> HF	57
NH <sub>4</sub> HF• H <sub>2</sub> O	75





Determine the amount of water needed to dissolve  $\text{NH}_4\text{F}$ . (85 g of  $\text{NH}_4\text{F}$ . are dissolved in 100 g of water at  $25^\circ\text{C}$  [2].  
 $261.17 + 222 = 483.17 \text{ ml}$ .

Amount of ammonia required for the reaction.  $X_1 = 184 \times 140 / 178 = 144.7 \text{ ml}$  at 100% concentration.  
At 15% ammonia is necessary.

$$X_{12} = 100 \times 144.7 / 15 = 964.6 \text{ ml (gr.)}$$

We took 100 gr. weighed HFSA, the concentration of commercial ammonia corresponds to 24%, a 24% ammonia solution is required for the reaction.

$$\begin{aligned} 140 - X \\ 24 - 100; \\ X_3 = 583.3 \text{ ml} \end{aligned}$$

In our case, a 15% ammonia solution is used. Amount of ammonia needed for 178 gr. equal to  $X = 933.4 \text{ ml}$ , for 100 g. necessary.

$$\begin{aligned} 178 - 933.3 \text{ ml.} \\ 100 - X; \\ X = 524.3 \text{ ml.} \end{aligned}$$

For a complete reaction to 100 gr.  $(\text{NH}_4)_2\text{SiF}_6$  requires 24% ammonia solution

$$\begin{aligned} 178 - 583.3 \text{ ml.} \\ 100 - X; \\ X = 325.88 \text{ ml.} \end{aligned}$$

So, for the complete carrying out of reactions (3), 325.8 ml are necessary. 24% ammonia solution, using a 15% solution of 524.3 ml. ammonia.

In this case, the  $\text{NH}_4\text{F}$  content in the solution is equal.

$$X_2 = 184 \times 222 / 179 = 228.2 \text{ gr.}$$

According to reaction 3, water is formed in an amount.

$$X_3 = 37.2 \text{ g.}$$

The total amount of water is.

$$\begin{aligned} 964.6 - 144.7 = 819.9 \text{ ml.} \\ 819.9 + 37.2 = 857.1 \text{ ml.} \\ \text{In this case, SiO}_2 \text{ is formed.} \\ X_4 = 62.02 \text{ gr.} \end{aligned}$$

In the table. 3 shows the material balance of obtaining silicon dioxide

**Table 3 The material balance of obtaining silicon dioxide**

Name	Coming,kg	Name	Consumption,Kg
15% $\text{NH}_4\text{OH}$	964,6	$\text{H}_2\text{O}$	857,1
$(\text{NH}_4)_2\text{SiF}_6$	184	$\text{NH}_4\text{F}$	228,2
		$\text{SiO}_2$	62,2
Total	1148,6	Total	1147,32

When filtering the formed silicon oxide, we get a precipitate with 10% moisture  $62.02 + 6.202 = 68.222 \text{ gr.}$  wet sediment.

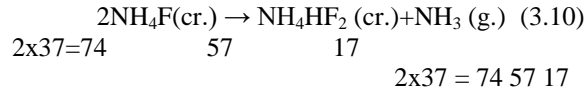
The concentration of the aqueous  $\text{NH}_4\text{F}$  solution will be  $228.2 + 857.1 = 1085.3$ .

$$[c] = 100 \times 228.2 / 1085.3 = 21.147 = 21.2\%$$

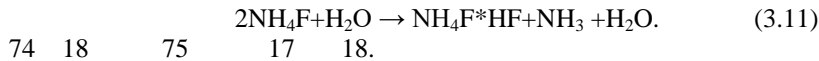
Evaporation of aqueous solutions of  $\text{NH}_4\text{F}$  leads to its decomposition. The boiling point and composition of the solutions are constantly changing. Even at the end of evaporation, the concentrate, in addition to  $\text{NH}_4\text{HF}_2$ , contains  $\text{NH}_4\text{F}$  and  $\text{H}_2\text{O}$ . The complete conversion and dehydration of  $\text{NH}_4\text{F}$  corresponds to the complete boiling-off of  $\text{NH}_4\text{HF}_2$ , that is, cannot be achieved during ordinary evaporation.

Under normal conditions, the density of  $\text{NH}_4\text{F}$  is  $1.002 \text{ g/cm}^3$ .

Melting of  $\text{NH}_4\text{F}$  is only possible under pressure (at low pressures, the melting point is close to  $240^\circ\text{C}$ ). When heated under ordinary conditions, it decomposes according to the equation:



Upon evaporation of the solution, we obtain ammonium bifluoride in the form of a monohydrate.



Since the content of NH<sub>4</sub>F in our solution is 228.2 g, we get 231.28 salt in the form of a monohydrate from it. The amount of water in the resulting salt = 55.5 ml.

The required amount of evaporated water.

$$850.9 - 55.5 = 795.4 \text{ ml.}$$

Amount of ammonia which was formed upon receipt of salt = 52.42 ml.

The material balance of the production of ammonium bifluoride is presented in table. 4

**Table 4 The material balance of obtaining ammonium bifluoride**

Name	Arrival, kg	Name	Consumption, kg
NH <sub>4</sub> F	228,2	NH <sub>4</sub> F*HF*H <sub>2</sub> O	231,28
H <sub>2</sub> O	850,9	H <sub>2</sub> O	795,4
		NH <sub>3</sub>	52,42
Total:	1079,1	Total:	1079,1

The obtained ammonium bifluoride is used for repeated fluorination. Further fluorination occurs according to equation (3.2).

## II. SIGNIFICANCE OF THE SYSTEM

The objective of the invention is the additional extraction of gold from the tailings of ores and beneficiation products into commercial products by reducing the sorption activity of the solid phase by increasing the temperature of the pulp.

**Determination of the yield of silicon dioxide.** In doing so, we use the above equation. As a result of the interaction of a solution of ammonium hexafluorosilicate with ammonia water, silicon dioxide is formed. For a more complete process, mixing is necessary.

During the experiments, a 15% ammonia solution with a density of 0.9390 g / cm<sup>3</sup> was used, the flow rate of which is 524.3 ml of a 15% ammonia solution per 100 g. ammonium hexafluorosilicate. Stirred for 1 hour, then filtered, the precipitate was washed three times with 100 ml and the washings were mixed with the filtrate. The precipitate is dried at 150<sup>0</sup>C and calcined at 700<sup>0</sup>C after cooling, weighed. The results are presented in table. 5

**Table 5  
Experimental determination of the yield of silicon dioxide**

Name	Experiences				
	1	2	3	4	5
(NH <sub>4</sub> ) <sub>2</sub> SiF <sub>6</sub> , g	100	100	100	100	100
SiO <sub>2</sub> , g	34	35	33,7	34	34

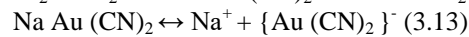
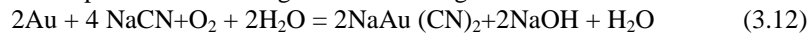
## III. METHODOLOGY

In carrying out the work, complex research methods were used, including scientific generalizations, experimental studies in laboratory and industrial conditions for the processing of technogenic formations using inorganic acids and an oxidizing agent, as well as methods of mathematical statistics and correlation analysis of test results using modern computer technology.

**Cyanide secondary cakes.** After desiliconization, this product underwent cyanidation in order to isolate precious metals from it.



The essence of the cyanidation process lies in the fact that the crushed ore material containing gold is brought into contact with a solution of sodium cyanide, under the action of which gold passes from the ore into the solution. In the presence of oxygen, dissolution proceeds according to the following reaction:



Cyanidation tests of secondary cakes were carried out in the laboratory of the Chadaksky Mill, in bottles with a closed-type mixer with a capacity of 0.75 liters. The concentration of sodium cyanide and protective alkali was determined by titration with a solution of silver nitrate in the presence of indicators. Mixing was carried out on a mechanical mixer with air from the compressor.

**The conditions for cyanide cakes.**

- Sample 100 g
- ratio T; W 1: 2,
- cyanide concentration of 0.4%,
- residual concentration of lime (CaOH) 0.02%,
- Cyanidation time is 18 hours.

**IV. THE EXPERIMENTAL RESULTS**

- Reagents and equipment The hardware scheme for processing gold-containing tailings consists of the following processes:

- 1 - ore opening process:
- 1- NH<sub>4</sub>F loading unit;
- 1a - tail loading unit;
- 2- charge loading unit;
- 3-3a - fluorination apparatus (furnace);
- 3b - desublimation apparatus
- 4- dust collecting device (filter);
- 5- apparatus for capturing NH<sub>3</sub>, H<sub>2</sub>O, HF (absorber);
- 2 - the process of purification of ammonia water and the regeneration of ammonium fluoride:
- 6 - apparatus for mixing the resulting products;
- 7 - filter;
- 8 - mold;
- 5 - apparatus for capturing NH<sub>3</sub>, H<sub>2</sub>O;
- 3 - deposition and filtration of silicon dioxide:
- 7 - filtering apparatus and deposition;
- 4 process of drying and calcining silicon oxide:
- 9 - drying apparatus;
- 10 - calcinations apparatus.

- Samples were taken from gold-bearing stale tails of the gold recovery area. The distribution of gold over sections of the tailings is given in table. 1.1. The number of samples taken and analyzed - 1000 pieces, including:

- 400 samples (210 points) from a square (40x40) m;
- 600 samples (300 points) from a square (10x10) m.
- assay method - assay; sampling depth 1-3 m., for some points 5-20 m; the average gold content in the tailings is 0.9 g / t; gold reserves - 3.3 tons; fractional composition of 85% - 0.074 mm.

**Table 6 Rational composition of tailings tailing**

Forms of finding gold	Content Au g / t	Distribution Au,%
Gold recovered by cyanidation (free and intergrowth)	0,49	35,3
Gold plated with alkaline destructible films (arsenic minerals) (hard to cyanide gold)	0,33	23,7
Gold plated with films of oxidized iron minerals (hard to cyanide gold)	0,07	5,0
Gold contained in pyrite and arsenopyrite (not recoverable by cyanidation)	0,26	18,7
Gold finely interspersed in rock-forming minerals (not recoverable by cyanidation)	0,24	17,3
Total	1,39	100,0

A pilot plant was designed and manufactured with a capacity of 10,000 tons of ash-containing tailings per day. The installation was tested on gold-containing tailings and positive results were obtained, which made it possible to recommend the developed fluoride technology for processing gold-containing tailings for industrial use. In the table. Figures 7 and 8 show the results of a chemical and complete chemical analysis of the tailings of gold mining plants before and after processing. The content of valuable components according to the results of analytical control over the processing of gold-containing tailings increased.

The temperature for intensive fluorination of gold-containing tailings was maintained at the level of 370-400 0C. Temperature control was carried out using sensors installed in the control panel with other control devices.

The specific consumption of ammonium bifluoride was determined by stoichiometry compared to the silica content in gold-containing tails. At a charge heating rate of 10 ° C / min, desilicization and separation by 85% in 1 hour. and at a speed of 26 ° C / min, desilicization and separation into separate components of the processed mass was 98% per hour.

**Table 7 Results of chemical analysis of stale tail samples  
Gold mining factories**

Content%									
Au,g/t	Ag, g/t	As	Fe <sub>all</sub>	Fe <sup>2+</sup>	Fe <sup>3+</sup>	S <sub>all</sub>	S <sub>s</sub>	CO <sub>2</sub>	CO <sub>oorg</sub>
0,36	1,7	0,19	4,46	2,05	2,41	0,71	0,35	2,02	0,32

**Table8 Complete chemical analysis of stale tail samples  
gold mining factories**

Content, %										
SiO <sub>2</sub>	Fe <sub>2</sub> O <sub>3</sub>	FeO	TiO <sub>2</sub>	Na <sub>2</sub> O	Al <sub>2</sub> O <sub>3</sub>	CaO	MgO	K <sub>2</sub> O	MnO	P <sub>2</sub> O <sub>5</sub>
60,0	3,4	2,6	1,0	2,0	13,7	3,3	1,65	2,6	0,52	0,16

**V.CONCLUSION AND FUTURE WORK**

The obtained results of industrial tests of the developed method for extracting gold from gold-containing tailings. Gold from the extraction factories, allowed us to determine the main technological parameters of the process before extraction. The test results indicate a high degree of readiness of the developed fluoride technology for the integrated processing of the tailings of gold mining plants.

Due to the lack of lamps, there is no data on silver in solutions designed to determine the silver content. The results of cyanidation of secondary cakes are given in table. 9.

**Table 9 The results of cyanidation of secondary cakes.**

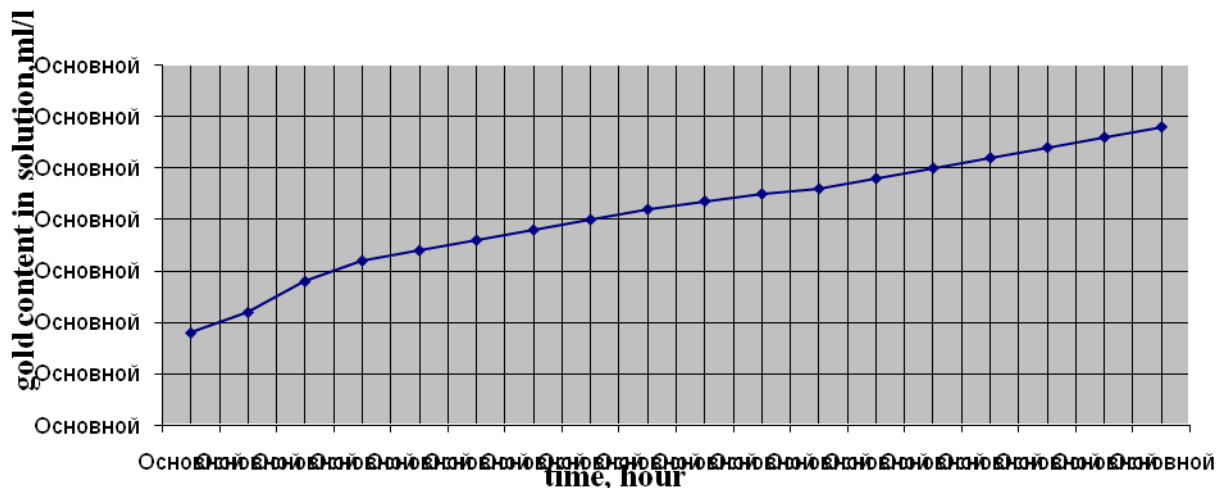
**Sample No. 1 Lab. No. 947 Content**

Names product	Weight, g/ml	Content Au, g/t	Content Ag, g/t	Extraction Au, %	Extraction Ag, %
Source	100	1,2	13,0	99,9	99,9
cake, g / t	100	сл	сл	99	99
solution, mg / l		0,29			

**Sample No. 2 Lab. No. 948**

Names product	Weight ,g ml	Content Au, g/t	Content Au, g/t	Extraction Au, %	Extraction Ag, %
Source	100	1,2	17,0	99,9	99,9
cake, g / t	100	сл	Сл	99	99
solution, mg / l		0,26			

After analyzing every hour by the liquid phase for gold, we determined the kinetics of gold dissolution from secondary cakes, which is shown in Fig. 1



**Figure 1. Kinetics of dissolution of gold from secondary cakes**

Thus, to determine the degree of regeneration of ammonium fluoride formed during the separation of hexafluoride ammonium silicate, a material balance of the process is drawn up on the basis of which a technological scheme for producing silicon dioxide is proposed.

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