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A procedure for preparing carbonaceous cyanidation tailings for gold fire assay

Alexander V. Byvaltsev[1], **Grigoriy I. Voiloshnikov**[2]✉, **Olga D. Khmelnitskaya**[3]

[1-3]*Irkutsk Research Institute of Precious and Rare Metals and Diamonds, Irkutsk, Russia*

Abstract. The aim is to develop a procedure for gold analysis of carbonaceous ore cyanidation tailings bearing gold in an adsorbed form. The proposed procedure includes high-temperature washing of the solid phase of tailings using a filter followed by transferring the adsorbed gold to the washing solution. The filtrate is analyzed separately, the solid phase undergoes standard fire assay, and the total gold grade is calculated by summing the gold in the solution and that in the solid phase. Using the proposed procedure, the proportion of adsorbed gold not detected by standard fire assay decreased by 4.1 times, from 38.2% to 9.3%. The studies were conducted using additions of gold solutions with a gold balance estimation. This additionally allowed gold adsorption isotherms to be studied by natural carbonaceous matter under cyanidation conditions. It was shown that at a gold concentration of 0.01–0.05 mg/dm^3 typical of industrial cyanidation tailings, the adsorbed gold value was significant at the level of 0.1–1.1 g/t. These gold losses are directly determined by the preg-robbing activity of the raw material. The K constant of Freundlich isotherms plotted as the Au loading of organic carbon, g/t vs Au concentration, mg/dm^3 was 303–3037 g/t, which is only an order of magnitude lower than that for commercial activated carbons. However, the greater mass of natural carbonaceous matter and its extended surface result in substantial gold losses with cyanidation tailings. The developed analytical procedure can be used to refine the actual gold losses with cyanidation tailings of carbonaceous raw materials.

Keywords: gold, cyanidation tailings, preg-robbing activity, carbonaceous matter, fire assay

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МЕТАЛЛУРГИЯ

Научная статья
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Методика подготовки углистосодержащих хвостов цианирования к пробирному анализу золота

А.В. Бывальцев[1], **Г.И. Войлошников**[2]✉, **О.Д. Хмельницкая**[3]

[1-3]*Иркутский научно-исследовательский институт благородных и редких металлов и алмазов, Иркутск, Россия*

Резюме. Цель – разработка методики анализа золота в хвостах цианирования углистосодержащих руд, содержащих золото в сорбированной форме. В предлагаемой методике перевод сорбированного золота в раствор осуществлялся путем высокотемпературной отмывки твердой фазы хвостов на фильтре. При этом фильтрат анализируется отдельно, твердая фаза подвергается стандартному пробирному анализу, а расчет содержания золота в исходном образце ведется по сумме золота в растворе и твердой фазе. По испытанной методике, доля сорбированного золота, неопределяемого пробирным анализом, в среднем снизилась с 38,2 до 9,3% – в 4,1 раза. Исследования проведены методом добавок известного количества золота, что, кроме основной цели, позволило изучить изотермы сорбции золота природным углистым веществом в условиях цианирования. Показано, что при концентрации золота в жидкой фазе пульпы на уровне 0,01–0,05 мг/дм^3, характерной для хвостов цианирования промышленных предприятий, доля сорбированного золота составила 0,1–1,1 г/т. Данные потери Au напрямую определяются сорбционной активностью сырья. Коэффициент K изотерм Фрейндлиха, построенных в виде зависимости содержания Au в углистом веществе (в г/т) от концентрации Au в жидкой фазе (в мг/дм^3), составил 303–3037, что лишь на порядок ниже, чем для товарных образцов активных углей. Однако превосходящее количество природного углистого вещества и его развитая поверхность предопределяют высокие потери золота с хвостами цианирования. Разработанная методика

анализа может быть использована для уточнения реальных потерь золота с хвостами цианирования углистосодержащего сырья.

Ключевые слова: золото, хвосты цианирования, сорбционная активность, углистое вещество, пробирный анализ

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INTRODUCTION

Studies show that preg-robbing ores and ore processing products cannot be easily processed by cyanidation and require additional treatment to increase gold recovery[4] [1–13]. The chemical analysis of preg-robbing ores and ore processing products can be challenging since the results of gold fire assay are often lower than actual [14–17]. The authors explain the difference by the volatility of carbonaceous gold species at a temperature above 300 °C. An improved procedure was developed to determine gold in the samples of preg-robbing ores and ore processing products by fire assay [18]. The procedure includes the pretreatment of samples by roasting with the addition of calcium and barium oxides and peroxides which minimize the formation of gaseous carbon and sulphur oxides and the losses of gold species with gases during fire assay.

Irgiredmet has been studying preg-robbing ores and ore processing products for many years. The institute discovered that the results of the gold fire assay are lower than actual when the tailings of preg-robbing ore cyanidation are analyzed [19]. Fire assay often cannot determine up to 50% of gold adsorbed by the solid phase during cyanidation. The authors explain the difference between fire assay results and actual gold content by the presence of gold in the solid phase in the adsorbed form, i.e. nonmetallic, atomized state. A fraction of atomized gold does not form any large enough metallic particles during the analysis. Also, it does not get wet by lead and remains encapsulated by borosilicate slags in the form of a real solution or nanoscale inclusion causing the erroneous results of fire assay. Similarly, this assumption is confirmed by lower fire assay results when the evaporated samples of cyanidation tailings are analyzed that consist of non-preg-robbing ores without organic carbon and carbonaceous gold species but contain some gold in a nonmetallic form before fire assay.

The studies were carried out to improve the accuracy of fire assay when it comes to the cyanidation tailings of preg-robbing ores and ore processing products. The method involves the recovery of hard-to-determine detect adsorbed gold from the solid phase into solution. The solution is analyzed for gold separately. Then, the solid phase is analyzed by standard fire assay. The gold content in the feed sample is calculated by summing gold in the solution and the solid phase. The high-temperature washing of the solid phase was tested to recover adsorbed gold from the samples before fire assay. The solid phase is washed using a filter. The slurry is heated to 90 °C and retained for a certain time followed by the filtration and washing of cake with heated water or a specific solution using a filter. Increasing water temperature reduces the preg-robbing of the solid phase. The equilibrium distribution of gold also decreases and shifts towards the solution while absorbed gold transfers to the liquid phase of the slurry. In addition to temperature, the method involves the washing of cake with heated water which contains no gold. As a result, the equilibrium further shifts towards the solution and the extra-elution of adsorbed gold takes place. The method has been already patented by the authors for commercial application [20] but it can be also practical for analytical purposes.

OBJECTIVE OF STUDY

The objective of the study was to investigate gold adsorption from cyanide solutions by natural carbonaceous matter and develop a procedure for the analysis of gold in carbonaceous cyanidation tailings.

MATERIALS AND METHODS

The materials of the study were the cyanidation tailings of preg-robbing ores and ore

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[4] Suzdal: Launch of HiTeCC Technology. Available from: https://minedocs.com/20/Suzdal_Presentation_06282016.pdf [Accessed 25th September 2024].

processing products from four mines in Russia. The chemical composition and size distribution of the products are summarized in Table 1. The experimental procedure included the “contamination” of samples with a specified quantity of gold. Then, a fraction of each sample was divided into two parts. One part was analyzed by direct fire assay with atomic absorption spectrometry (AAS) finish while another part was subjected to high-temperature washing followed by the analysis of a resulting solution and the fire assay of a washed sample with AAS finish.

Preparations. All cyanidation tailings had been produced during “standard” static laboratory experiments under the following conditions: each subsample mass of 1.00 kg; temperature of 22–24 °C; pH (slurry) = 10.8–11.2 (adjusted by the addition of CaO); NaCN concentration of 1–2 g/dm^3; liquid-to-solid ratio (L/S) = 2:1; cyanidation time of 48 h; fresh activated carbon addition of 10 vol. %.

After “standard” cyanidation at room temperature, activated carbon had been replaced by fresh adsorbent followed by high-temperature treatment under the same conditions except temperature (85–90 °C) and time (3 hours). Then, activated carbon was again replaced by fresh one followed by the second stage of high-temperature treatment. The purpose of these manipulations was to maintain the integrity of the experiments, i.e. to maximize the recovery of adsorbed gold from cyanidation tailings. The tailings of high-temperature treatment contained 0.33–2.76 g/t of gold. It was presumed that this gold was finely disseminated in minerals and could not be leached with cyanide.

After the separation of activated carbon, the slurry was cooled and sampled (~10% of the slurry volume) for the analysis of gold content in solid and liquid phases (3 replicates), see Table 1. The gold concentration in the liquid phase was below the detection limit (<0.005 mg/dm^3) and assumed as 0 mg/dm^3 in calculations.

Main experiments. The cake of each cyanidation tailings slurry sample was divided into 5 equal parts (0.18 kg of solids each) and mixed with the various quantities of synthetic $Na[Au(CN)_2]$ solution containing 86.2 mg/dm^3 of gold. Then, the samples were agitated at room temperature for 48 hours to adsorb gold by the solid phase. After agitation, 1/2 (0.09 kg of solids) of each slurry sample was collected and filtered using a Buchner filter funnel. The filtrate was subjected to AAS for gold analysis. The wet cake was weighted, dried to a constant mass at 105 °C, and analyzed for initial moisture content calculated by weight loss. The dry

Table 1. Characteristics of the studied carbonaceous preg-robbing cyanidation tailings
Таблица 1. Характеристики исследованных углистых сорбционно-активных хвостов цианирования

Product characteristics		**Value**			
Product		Ore cyanidation tailings – Mine 1	Ore cyanidation tailings – Mine 2	Tailings of gravity tailings cyanidation – Mine 3	Tailings of flotation concentrate cyanidation – Mine 4
Name		Product 1	Product 2	Product 3	Product 4
Component	SiO_2, %	67.8	76.4	61.9	41,5
	Al_2O_3, %	14.2	12.4	18.4	11,2
	Fe_{tot}, %	3.40	4.18	1.16	15,4
	S_{tot}, %	0.40	0.55	0.27	11,9
	C_{org}, %	0.34	0.60	0.52	2,8
	Au*, g/t	0.33	0.56	0.45	2,76
Mass fraction of -0,071 mm size, %		89	94	95	98
Mass fraction of -0,16 mm size, %		100	100	100	100
Slurry L/S		2:1	2:1	2:1	2:1
Slurry pH		10,9	11.0	11.2	11.0
NaCN concentration in liquid phase, g/dm^3		1,2	1.8	1.5	1.1
Au concentration in liquid phase, mg/dm^3		<0.005	<0.005	<0.005	<0.005

Note: Au content after two stages of cyanidation tailings treatment by high-temperature adsorption process.

cake was subjected to gold fire assay with AAS finish (3 replicates); the results were averaged.

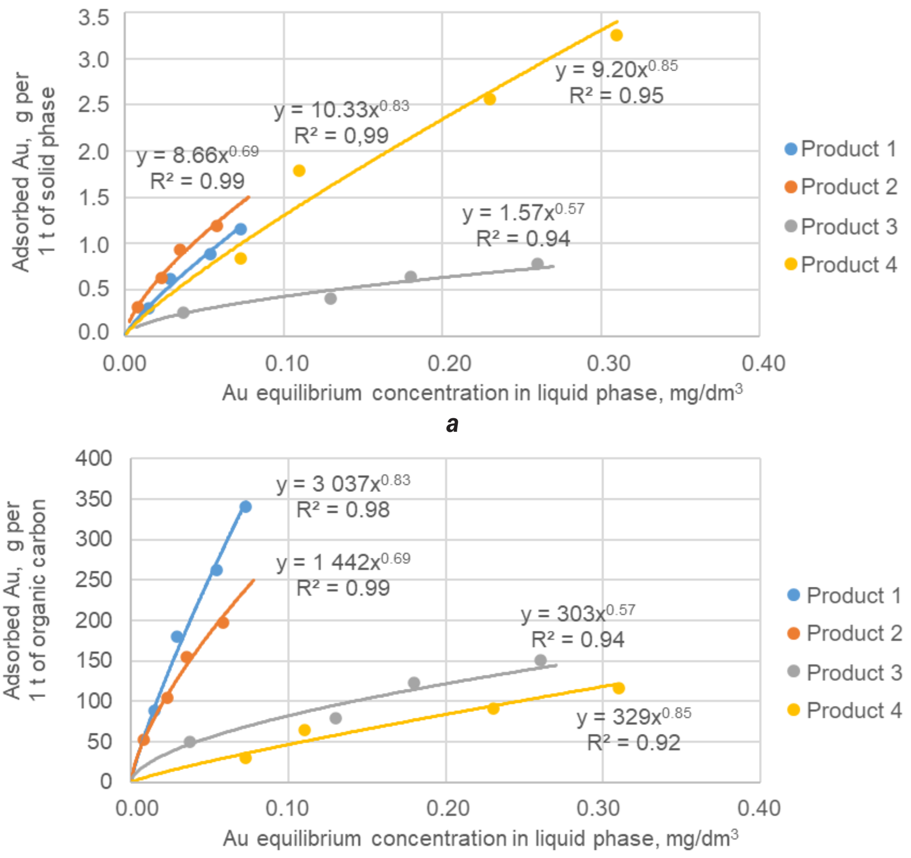
The second part of each “gold-contaminated” sample (0.09 kg of solids) was washed at high temperature to recover adsorbed gold into solution and calculate the quantity of recovered Au. For this purpose, the slurry was heated to 90°C and retained at this temperature for 10 min. Then, the slurry was collected and filtered using a heated Buchner filter funnel and washed with 0.20 L of water heated to 90°C combining barren and washing filtrates. After measuring the volume of each combined filtrate, it was subjected to AAS for gold analysis. The washed cake was dried to a constant mass at 105°C and subjected to gold fire assay with AAS finish (3 replicates); the results were averaged. The gold content in the solid phase before washing was calculated summing precious metal contained in filtrate and washed cake and normalized to the initial sample mass. Table 2 summarizes the results of gold washing from the solid phase.

The time of bullion smelting was 45 min at 1080°C. Flux composition was as follows (per 25 g of a sample): 40 g of lead oxide, 120 g of soda, 30 g of borax, 3 g of metallic iron, and 3 g of flour. The time of cupellation was 45 minutes at 905°C. The resulting gold beads were dissolved in aqua regia followed by AAS of solutions.

RESULTS AND DISCUSSION

Table 2 presents the results of sample “contamination” with gold. Adding a specified amount of $Na[Au(CN)_2]$ was used to determine the correct isotherm of gold adsorption by the solid phase and organic carbon directly under cyanidation conditions, i.e. without the preliminary removal of carbonaceous matter and at a characteristic composition of salt in the slurry liquid phase. The isotherms are shown in Fig. 1 and Fig. 2.

Fig. 1 *a* indicates that the fraction of adsorbed gold was 0.1–0.4 g/t, 0.2–0.8 g/t,



a

b

Fig. 1. *Isotherms of gold adsorption by the solid phase of cyanidation tailings (a) and natural organic carbon in cyanidation tailings (b)*

Рис. 1. *Изотермы сорбции золота твердой фазой хвостов цианирования (a) и природным органическим углеродом, входящим в их состав (b)*

Table 2. Results of experiments on carbonaceous cyanidation tailings "contamination" with sodium dicyanoaurate
Таблица 2. Результаты опытов по «заражению» углистосодержащих хвостов цианирования дицианоауратом натрия

Experiment No.	**Experimental**				**Design**				
	Added Au for solid phase, g/t	Produced by filtration after an experiment			Au in cake (by Au loss in liquid phase and residual cake moisture), g/t	Adsorbed Au		Added Au undetermined by fire assay	
		Au concentration in liquid phase, mg/dm^3	Filter cake moisture content,%	Au in cake by fire assay with AAS, g/t		g per 1 t of solid phase	g per 1 t of organic carbon	g/t	%
Product 1									
1 (blank)	0	0	16.2	0.33	0.33	0	0	0	0
2	0.33	0.015	17.5	0.54	0.63	0.30	88	0.09	31.1
3	0.67	0.029	15.5	0.71	0.95	0.61	180	0.24	39.1
4	1.00	0.054	18.9	0.93	1.23	0.89	262	0.30	33.3
5	1.30	0.073	17.0	1.14	1.50	1.16	340	0.36	30.8
Product 2									
6 (blank)	0	0	14.3	0.56	0.56	0	0	0	0
7	0.33	0.008	14.7	0.70	0.88	0.31	52	0.18	56.6
8	0.67	0.023	18.4	0.90	1.19	0.62	104	0.29	45.5
9	1.00	0.035	15.0	1.08	1.50	0.93	155	0.42	44.5
10	1.30	0.058	15.5	1.37	1.76	1.19	198	0.39	32.3
Product 3									
11 (blank)	0	0	14.3	0.46	0.45	0	0	0	0
12	0.33	0.037	15.5	0.62	0.71	0.26	49	0.09	35.3
13	0.67	0.13	17.2	0.73	0.89	0.41	79	0.15	35.2
14	1.00	0.18	16.9	0.94	1.13	0.64	123	0.18	26.8
15	1.30	0.26	16.8	1.04	1.28	0.78	150	0.24	29.3
Product 4									
16 (blank)	0	0	19.1	2.75	2.78	0	0	0	0
17	0.99	0.073	20.8	3.19	3.62	0.84	30	0.43	50.2
18	2.01	0.11	17.6	3.74	4.57	1.79	64	0.83	45.9
19	3.02	0.23	17.9	4.17	5.37	2.56	91	1.20	45.9
20	3.88	0.31	18.3	5.13	6.09	3.26	116	0.96	28.8

and 0.3–1.1 g/t at an Au concentration in the liquid phase of 0.01 mg/dm^3 (typical for cyanidation tailings from existing mines), 0.03 mg/dm^3, and 0.05 mg/dm^3, respectively. It should be noted that this fraction is quite significant and represents direct gold losses with cyanidation tailings caused by preg-robbing. In other words, this gold would have been recovered without preg-robbing.

As Fig. 1 *b* illustrates, the K factor of Freundlich isotherms (Au concentration (mg/dm^3) of the liquid phase vs. Au content (g/t) in carbonaceous matter) for organic carbon was 303–3037. K value for activated carbon typically varies from 3,000 to 30,000. It means that preg-robbing is only one order of magnitude lower for natural organic carbon than activated carbon. However, a larger quantity of natural carbonaceous matter in the feed (proportional to the process streams of activated carbon and natural carbonaceous matter in a cyanidation plant) resulted in significant adsorbed gold losses with cyanidation tailings.

From 30.8% to 56.5% of adsorbed gold (38.2% on average) was not determined in "contaminated" samples by direct fire assay with AAS finish.

Table 3 summarizes the results of the experiments on the high-temperature washing of adsorbed gold from cyanidation tailings to improve the accuracy of fire assay. The combined filtrates from washing contained 0.073–0.82 mg/dm^3 of gold. It is higher than the gold concentration in the initial liquid phase (0.015–0.31 mg/dm^3), even at the ~1.8-times dilution of solutions using washing water, and confirms the recovery of gold from carbonaceous matter. The washed solid phase contained less gold (0.34–3.04 g/t) than the solid phase without washing (0.54–5.13 g/t, see Table 2)

Table 3. Results of experiments on high-temperature washing of adsorbed gold from carbonaceous cyanidation tailings

Таблица 3. Результаты опытов по высокотемпературной отмывке сорбированного золота из углистосодержащих хвостов цианирования

Experiment No.	**Experimental**					**Design**		
	Added before each experiment		Produced after each experiment by slurry filtration			Au content in initial solid phase by balance (washed cake + filtrate), g/t	Added Au undetermined by washing and washed cake and solution analysis	
	Initial Au content in solid phase, g/t	Initial Au concentration in liquid phase, mg/dm^3	Combined filtrate volume, L	Final Au concentration in liquid phase, mg/dm^3	Au content in washed cake by fire assay with AAS, g/t			
							g/t	%
Product 1								
21 (blank)	0.33	0	0.364	0	0.34	0.34	0	0
22	0.63	0.015	0.359	0.073	0.34	0.60	0.03	10.6
23	0.95	0.029	0.360	0.15	0.37	0.91	0.04	5.7
24	1.23	0.054	0.352	0.21	0.41	1.12	0.11	12.3
25	1.50	0.073	0.371	0.25	0.45	1.33	0.17	14.2
Product 2								
26 (blank)	0.56	0	0.367	0	0.54	0.54	0	0
27	0.88	0.008	0.353	0.071	0.58	0.84	0.03	10.5
28	1.19	0.023	0.355	0.15	0.59	1.14	0.05	8.5
29	1.50	0.035	0.371	0.20	0.62	1.37	0.12	13.0
30	1.76	0.058	0.351	0.28	0.68	1.66	0.10	8.4
Product 3								
31 (blank)	0.45	0	0.363	0	0.45	0.45	0	0
32	0.71	0.037	0.363	0.079	0.45	0.69	0.02	7.0
33	0.89	0.13	0.374	0.16	0.47	0.87	0.01	2.7
34	1.13	0.18	0.370	0.22	0.51	1.05	0.07	10.7
35	1.28	0.26	0.354	0.31	0.50	1.20	0.08	10.2
Product 4								
36 (blank)	2.76	0	0.363	0	2.80	2.80	0	0
37	3.62	0.073	0.362	0.20	2.85	3.51	0.12	13.3
38	4.57	0.11	0.350	0.44	2.90	4.39	0.18	10.0
39	5.37	0.23	0.345	0.72	2.97	5.27	0.10	3.7
40	6.09	0.31	0.373	0.82	3.04	5.82	0.27	8.1

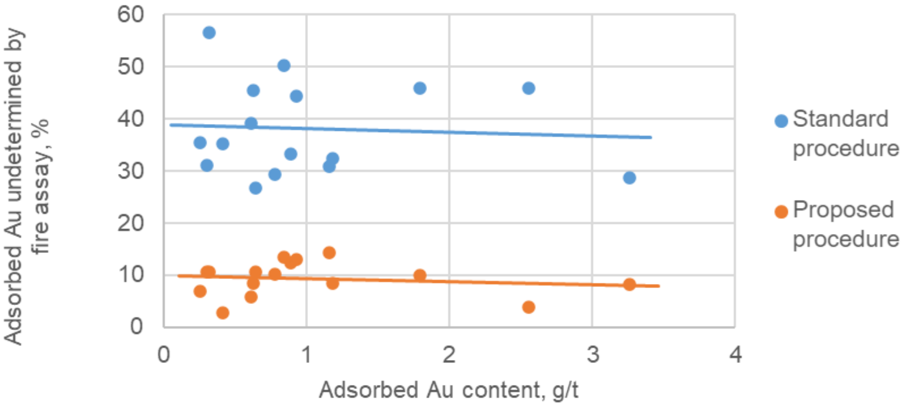


Fig. 2. *Fraction of adsorbed gold undetermined by direct fire assay according to the standard procedure vs fraction of adsorbed gold analyzed using high-temperature washing*

Рис. 2. *Зависимость доли сорбированного золота, неопределяемого пробирным анализом, по стандартной методике и по методике с предварительной высокотемпературной отмывкой золота*

as specified by the analysis data. However, the head gold content was higher in the initial solid phase without washing (0.60-5.82 g/t) by balance calculations than by direct analysis and closer to actual values determined by the amount of gold added in the experiments.

From 2.7% to 14.2% of gold (9.3% on average) was not determined in the "contaminated" samples by high-temperature washing followed by the analysis of solutions and washed cake as well as by balance calculations. It means that the proposed procedure reduced the fraction of such gold from 38.2% to 9.3% on average, or by 4.1 times.

Fig. 2 shows the fraction of adsorbed gold undetermined by direct fire assay with AAS finish and by the proposed procedure as a function of the amount of adsorbed gold in the solid phase.

CONCLUSIONS

Gold losses due to preg-robbing were estimated using the isotherms of gold adsorption by carbonaceous matter. The value of losses varied from 0.1 g/t to 1.1 g/t for the slurry liquid phase with a gold concentration of 0.01 mg/dm^3 to 0.05 mg/dm^3 which is typical for cyanidation tailings from existing mines. The data and the procedure can be used in the scientific and engineering studies of new gold-bearing materials.

The paper describes a new procedure for the analysis of gold in the liquid phase of carbonaceous cyanidation tailings. The advantages of the procedure include less measurement error compared to standard fire assay; the error was reduced from 38.2% to 9.2% during the experiments. The procedure can be used in existing mines for a more accurate estimation of actual gold losses with carbonaceous cyanidation tailings.

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INFORMATION ABOUT THE AUTHORS

Alexander V. Byvaltsev,
Cand. Sci. (Eng.),
Leading Researcher of the Hydrometallurgy Laboratory,
Irkutsk Research Institute of Precious and Rare Metals and Diamonds,
38, Gagarin Blvd., Irkutsk 664025, Russia
torrot_2008@mail.ru

Grigoriy I. Voiloshnikov,
Dr. Sci. (Eng.), Professor,
Deputy Director for Science, Methodology and Innovations,
Irkutsk Research Institute of Precious and Rare Metals and Diamonds,
38, Gagarin Blvd., Irkutsk 664025, Russia
✉ greg@irgiredmet.ru

Olga D. Khmelnitskaya,
Cand. Sci. (Eng.),
Leading Researcher of the Hydrometallurgy Laboratory,
Irkutsk Research Institute of Precious and Rare Metals and Diamonds,
38, Gagarin Blvd., Irkutsk 664025, Russia
hod@irgiredmet.ru

ИНФОРМАЦИЯ ОБ АВТОРАХ

Бывальцев Александр Владимирович,
к.т.н.,
ведущий научный сотрудник
лаборатории гидрометаллургии,
Иркутский научно-исследовательский институт
благородных и редких металлов и алмазов,
664025, г. Иркутск, Бульвар Гагарина, 38, Россия
torrot_2008@mail.ru

Войлошников Григорий Иванович,
д.т.н., профессор,
заместитель генерального директора по
научно-методической и инновационной деятельности,
Иркутский научно-исследовательский институт
благородных и редких металлов и алмазов,
664025, г. Иркутск, Бульвар Гагарина, 38, Россия
✉ greg@irgiredmet.ru

Хмельницкая Ольга Давыдовна,
к.т.н.,
ведущий научный сотрудник
лаборатории гидрометаллургии,
Иркутский научно-исследовательский институт
благородных и редких металлов и алмазов,
664025, г. Иркутск, Бульвар Гагарина, 38, Россия
hod@irgiredmet.ru

Authors' contribution

The authors contributed equally to this article.

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