

# Technological studies of the use of modifiers in the flotation of calcium-containing minerals

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**Abstract.** Using samples of skarn scheelite-sulfide ore from the Vostok-2 deposit, studies were carried out to model combinations of increased dose of inorganic modifiers (sodium carbonate, water glass, sodium sulfide) based on publications by foreign researchers on the achieved positive result in increasing the contrast of the mineral surfaces of scheelite and calcite and their floatability. Testing was carried out according to the standard flowsheet on tap and recycled water. Oxyhydril collector was sodium oleate of technical acid B-115, dose 250 + 50 g/t. The order of feeding modifiers (sequential supply and combination of a mixture of sodium carbonate (bicarbonate) + water glass), the effect of the temperature of the pulp upon contact, the dose of modifiers and the ratio of components in the mixture were studied. The dose of water glass varied from 350 to 1450 g/t, sodium carbonate (bicarbonate) varied from 0 to 11.5 kg/t, sodium sulfide up to 4.9 kg / t. With increased dose of inorganic modifiers, it was not possible to achieve selective depression of calcite and apatite while maintaining effective scheelite flotation. The use of sodium bicarbonate in comparison with sodium carbonate, at the same flow rates, creates a less alkaline pH in the flotation pulp, which leads to an increase in the concentration of hardness ions [ $\text{Ca}^{2+}$ ,  $\text{Mg}^{2+}$ ] and overdose of the collector. Using combinations of reagents: sodium carbonate (bicarbonate) + water glass, sodium sulfide + sodium hydroxide + water glass, even with a slight increase in the dose of water glass in the mixture, after heat treatment, concentrates of lower quality were obtained, with increased losses of scheelite compared to the factory mode according to the Petrov method.

## 1 Introduction

In the scheelite-sulphide ore of the Vostok-2 skarn deposit, the target mineral is scheelite ( $\text{CaWO}_4$ ) and gangue calcium minerals (calcite –  $\text{CaCO}_3$ , apatite –  $\text{Ca}_5[\text{PO}_4]_3(\text{F}, \text{Cl})$ , single grains of wollastonite –  $\text{CaSiO}_3$ ), which have similar flotation properties due to the same active calcium cation on the surface on which the collector is adsorbed. The separation of minerals is caused by the different bond strengths of the surface cation with the anion of the crystal lattice. According to the standard technology with a dose of sodium carbonate up to

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pH 9.8, hardness less than 1 mg-eq/l, water glass 350 g/t and sodium oleate 250 + 50 g/t, the following enrichment indicators in the main cycle are achieved in laboratory conditions:  $\alpha_{\text{WO}_3} > 0.6\%$ ,  $\alpha_{\text{CaCO}_3}$  4-25%,  $\alpha_{\text{Ca}_5(\text{PO}_4)_3(\text{F}, \text{Cl})}$  0.10-0.20%; recovery of calcium minerals, respectively, is as follows: scheelite 85-94%, apatite 70-75%, calcite 30-70%. With a decrease in the mass fraction of scheelite in the range  $\alpha_{\text{WO}_3}$  0.10- 0.6%, the recovery of scheelite is 40-86%, calcite is 65-80%.

Achieving contrast in the hydrophobicity of surfaces of the separated calcium minerals is a rather difficult problem to solve. It is possible to enhance the contrast of flotation of the separated minerals due to the reagent mode. Modifiers are paid much attention in the latest publications. In published studies by different researchers, the same range of reagents was used as modifiers: sodium carbonate (the main regulator of the environment), water glass (the main suppressor), heavy metal salts, and natural polymolecular compounds.

In Foucaud et al. [1], according to the presented recovery curves, higher results were obtained when using a mixture of water glass and salts of polyvalent ions (Fe (II), Zn) in the reagent mode; mixtures of water glass and sodium carbonate.

Methods for adding sodium carbonate with ethoxylated phenols (OP 10), combinations of sodium sulphide, sodium carbonate and water glass in the steaming of rough rare-earth concentrates have been presented for a long time in S.I. Polkin [2].

Chinese researchers Kang J., Liu Yu., Khoso S. Ah. Et al. [3], when upgrading a rough concentrate containing scheelite, wolframite, calcite, fluorite, presented an industrially developed alternative solution to the Petrov method. In the reagent mode of the rough cycle, the dose of the fatty acid collector was 1.5 kg/t; NaOH 2 kg/t, in the selection  $\text{Na}_2\text{SiO}_3$  95 – 90 kg/t,  $\text{Na}_2\text{S}$  5 – 8 kg/t. With the introduction of sodium sulfide into the steaming, after diluting the steamed product with water to 35% of the solid and carrying out three cleaning operations, the quality of the  $\text{WO}_3$  concentrate increased from 57.75 to 66.51% with an increase in the  $\text{WO}_3$  recovery from 69.38 to 83.25%.

Previously, the authors published the results of testing the properties of water glass modified with salts of heavy metals, a combination of water glass and  $\text{CaCl}_2$ , the use of CMC, tannin, starch for the flotation activity of calcium minerals, which showed no advantages over water glass in the cycle of obtaining a collective concentrate and selection by the Petrov method [4-6].

The results of industrial tests at Primorskaia Concentrating Factory (PCF) when replacing water glass with a mixture of water glass and ferrous sulfate showed a lack of stability in obtaining the quality of rough and final scheelite concentrates with an increase in scheelite losses.

In their studies, the authors have demonstrated a successful use of higher aliphatic isoalcohols to reduce the flotation activity of calcite while increasing the recovery of scheelite into a froth concentrate [7-8].

However, based on scientific publications by foreign researchers on the achieved results of the contrast in the separation of scheelite and calcite, due to the use of increased dose of sodium carbonate, water glass and sodium sulphide and on a greater availability of inorganic modifiers than oxyhydril nonionic surfactants, studies on the combination of these modifiers for ores of the Vostok-2 deposit were carried out.

## 2 Materials and Methods

The effect of a combination of environment modifiers and a water glass depressor on the floatability of calcium-containing minerals from the Vostok-2 deposit was tested according to the standard flotation scheme. The scheme of the rough cycle included the rough scheelite flotation and scavenge flotations (flotation for 10 minutes, cell volume 1.0 l, pulp temperature [T] during agitation with reagents and flotation 28-40°C) and cleaning of

combined concentrates (flotation for 5 min, cell volume 0.5-0.3 l, T-22°C). A pulp of the skarn ore was conditioned with reagents for 2 min.

Methodology for upgrading the rough concentrate: thickening (15 minutes in a 2-liter glass, water column height -190 mm), steaming in a water bath (time (t) – 60 min, T-85-90°C), two or three cleaning of the steamed concentrate (volume of cell 0.5-0.3-0.1 l, flotation for 5 min, T-20-25°C) and the scavenge flotation for 10 min. Combinations of modifiers during the heat treatment of rough concentrates were introduced into the hot steaming pulp (T-85°C).

The studies were carried out using recycled water, a mixture of recycled and tap water ratio is 1:5. When setting up laboratory experiments with ore samples, the initial weight of the portion was 1 kilogram. After grinding (to a content of 70-75% of the class -80+0 microns) and sulphide flotation, the sulphide tailings were divided into 4 weighed portions and in the experiments were feeding the rough scheelite cycle. When sampling flotation products during flotation at the concentration factory, the weight of the test portions was 250 – 350 g (sulphide flotation tailings), 120 – 140 g (rough concentrate). Weighed individual portions of sample were taken from the total sample either by separating with dividers, or by scooping out pulp equal in volume to the weighed portions, with active stirring of the original sample.

Working solutions of flotation reagents used in studies of ore and flotation products were used in the following concentrations, based on the calculation of 100% of their activity: 10% Na<sub>2</sub>CO<sub>3</sub> and NaHCO<sub>3</sub>; 10-50% Na<sub>2</sub>S; 5-30% Na<sub>2</sub>SiO<sub>3</sub>; sodium oleate saponified with alkali in laboratory conditions on a water bath with technical oleic acid B-115 (activity 98%); the concentration of the collector's working solution being 5%. When preparing a combination of reagents Na<sub>2</sub>CO<sub>3</sub> and Na<sub>2</sub>SiO<sub>3</sub>, the calculated amount of sodium carbonate solution was introduced into the water glass solution, the components of the mixture were mixed for 3 minutes, and then fed into the pulp, or the mixture was in contact for a day. In a mixture of Na<sub>2</sub>SiO<sub>3</sub> and NaHCO<sub>3</sub>, on the contrary, water glass was introduced into a sodium bicarbonate solution, otherwise a precipitate formed. Solutions of different combinations of modifiers, which during testing were introduced into the pulp of the flotation machine, retained their transparency, the agitation with the pulp for 2 min.

### 3 Results and Discussion

L.O. Filippov et al. [9-10] demonstrated that at optimal dose of modifiers of 1.150 kg/t of sodium carbonate and 1.225 kg/t of water glass (ratio 1:1.1) with dose of fatty acid collector 200 g/t, the contrast of the mineral surface of scheelite and calcite increases.

In a number of works, it was noted for a long time that the depressing effect of water glass on many minerals is reduced (or even eliminated) in the presence of sodium carbonate. According to N.A. Yanis [11], desorption of silicic acid products from the surface of minerals by carbonic acid anions occurs, active centers on minerals are restored, providing adsorption of the collector. M.M. Sorokin believed that it would be more correct to speak about the hydrolysis of acid anions rather than about desorption [6].

Table 1 shows the results of ore flotation of the Vostok-2 deposit in the cycle of rough scheelite flotation with the factory pulp taken from the tailings of sulfide flotation (*S tailings*). The dose of sodium carbonate (sodium bicarbonate) varied from 0 to 3.3 kg/t, water glass into rough flotation varied from 350 to 1,450 g/t, into cleaner flotation it was 100 g/t, the dose of sodium oleate was constant 250+50 g/t (rough and scavenge flotation).

**Table 1.** Influence of environment modifiers and their mixture with water glass on the floatability of calcium-containing minerals in the rough cycle

Tests conditions Dose, kg/t Na <sub>2</sub> CO <sub>3</sub> / NaHCO <sub>3</sub> <sup>(B)</sup> ; [pH; C <sub>hard</sub> , mg-eq/l]	Rough scheelite concentrate					Tailings	
	Yield %	Assay % WO <sub>3</sub>	Recovery, %			Assay % WO <sub>3</sub>	Recovery, % WO <sub>3</sub>
			WO <sub>3</sub>	CaCO <sub>3</sub>	P		
<i>Stailings of PCF with mass fraction WO<sub>3</sub>-0.69 %, CaCO<sub>3</sub>-4.60 %, P-0.21 %</i>							
<b>Recycled water (pH-8.7) + tap water (pH-7.0); water glass dose 350 g/t</b>							
<b>Separate sequential feed of reagents</b>							
0; [pH= 8.49; C <sub>hard</sub> =3.0]	3.28	12.83	61.24	26.01	19.04	0.15	19.64
0.21; [9.0; 2.6]	6.01	9.52	83.33	50.15	48.69	0.073	9.48
0.40; [9.5; 1.13]	6.95	8.42	84.46	57.87	49.92	0.058	7.40
0.85; [9.85; 1.0]	7.03	8.40	85.60	58.95	54.50	0.056	7.13
<i>Mixture (sodium carbonate + water glass; contact of mixture components for 3 min)</i>							
0.23; [9.5; 2.3]	7.56	7.40	81.34	61.06	53.50	0.082	10.35
0.91; [10.0-1.04]	9.67	5.83	82.09	63.48	61.10	0.063	7.56
<i>Mixture (sodium carbonate + water glass; contact of mixture components for 1 day<sup>1)</sup></i>							
0.25; [9.5; 2.0]	6.54	7.98	75.35	44.14	45.98	0.11	13.85
0.94; [9.8-1.1]	6.80	8.25	80.86	58.06	49.80	0.10	12.68
<i>Dose of water glass 450 g / t (ratio of sodium carbonate : water glass = 1:1)</i>							
0.44; [9.7; 1.39]	4.37	12.2	77.32	38.37	35.52	0.086	11.44
0.46 <sup>1)</sup> ; [9.78; 1.3]	4.92	10.2	72.84	33.38	39.63	0.095	12.49
0.44 <sup>2)</sup> ; [9.76; 1.8]	4.53	9.80	63.95	31.31	34.81	0.14	18.21
<i>Dose of water glass 1.450 g / t (ratio of sodium carbonate : water glass = 4.4:1)</i>							
3.3 <sup>1)</sup> ; [10.5; 0.72]	1.24	22.8	40.95	8.04	8.39	0.17	23.63
3.3 <sup>2)</sup> ; [10.4; 1.0]	0.42	50.5	30.44	1.27	1.08	0.25	35.40
<b>Recycled water pH-8.8; ratio of sodium carbonate : water glass (350 g/t)= 1(5):1</b>							
0.4; [9.5; 1.9]	5.29	9.75	74.85	-	-	0.136	18.01
0.95; [10; 1.0]	8.08	6.67	78.05	-	-	0.112	14.20
1.65; [10.9; 0.65]	8.53	5.76	71.11	-	-	0.176	22.08
0.98 <sup>B</sup> ; [8.2; 2.4]	8.20	6.54	77.85	-	-	0.11	13.92
<b>Recycled water; ratio sodium bicarbonate : water glass (450 g/t)= 8:1</b>							
3.4 <sup>B</sup> ; [8.96; 2.04]	8.92	6.29	80.93	-	-	0.109	13.77
3.55 <sup>1B)</sup> ; [9.57; 1.4]	15.84	3.58	82.24	-	-	0.10	11.08
3.4 <sup>2B)</sup> ; [9.60; 1.68]	15.45	3.57	84.01	-	-	0.077	8.64

Notes: <sup>1)</sup> contact of mixture components for 3 min; <sup>2)</sup> contact of mixture components for 1 day; B – bicarbonate; C<sub>hard</sub>- hardness ion concentration.

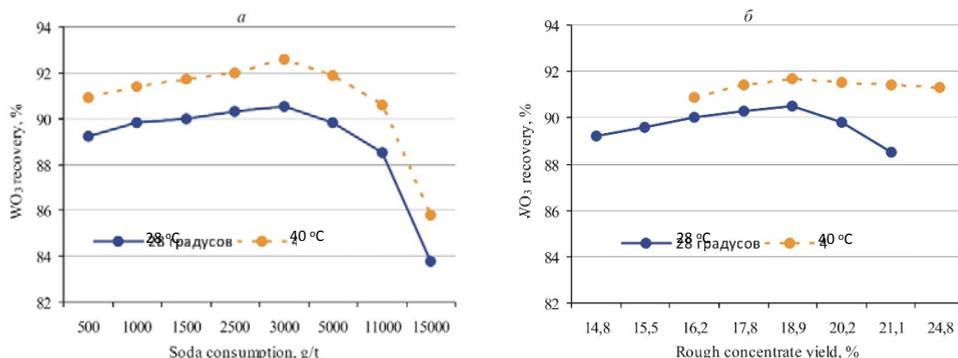
In the absence of sodium carbonate in the flotation pulp, a rough concentrate with an assay of tungsten trioxide 13%, scheelite recovery 60%, apatite and calcite recovery 20-26% was separated. An increase in the dose of sodium carbonate to 850 g / t led to the achievement of the optimum for scheelite pulp pH 9.8, a hardness of 1 mg-eq/l and an increase in the yield of the rough concentrate. The recovery of scheelite increased up to 85%, calcium impurities up to 55-59%.

In the combinations of a mixture of sodium carbonate (bicarbonate) and water glass, with different contact times of the components in the mixture, the tendency for the behavior of calcium minerals remained, as with the successive supply of reagents. Scheelite recovery ranges from 75-82%.

The reagent mode of the technology: the dose of sodium carbonate in a combination of 3.3 kg/t and water glass of 1.450 g/t (daily contact of the mixture components) made it

possible to separate a conditioned scheelite concentrate. This mode is an alternative to the Petrov method, but at the same time, scheelite recovery being 30%. According to the standard scheme, for a given quality of the final concentrate, scheelite recovery is 88% (see Table 2).

The figure shows the results of the influence of sodium carbonate dose up to 11.4 kg/t at constant dose of water glass 350+100 g/t and sodium oleate 250+50 g/t in two temperature modes of pulp contacts with reagents 28 and 40°C.



**Fig. 1.** Influence on the parameters of  $WO_3$  recovery into rough concentrate of sodium carbonate dose and pulp temperature upon contact with rough flotation reagents (a); the relationship between the extraction of  $WO_3$  into the concentrate and the yield of the rough concentrate at different dose of sodium carbonate and temperature of contact with reagents (b)

As can be seen from the figure, the dependence of recovery of  $WO_3$  in the rough concentrate on sodium carbonate dose first increases, passes through a maximum, and then gradually decreases. With an increase in the temperature of the pulp contact with reagents up to 40°C, the maximum is achieved with a lower dose of sodium carbonate (1.5 kg/t vs 2.9 kg/t). With an increase in sodium carbonate dose (from 0.4 to 11.4 kg/t), the degree of  $WO_3$  enrichment decreases from 6 to 4.2. The relationship between the recovery of  $WO_3$  and the yield of concentrate from the dose of sodium carbonate (b) shows that when the collective concentrate yield is more than 16 – 18%, the recovery of  $WO_3$  in the rough concentrate practically does not increase at 40°C, and decreases at 28°C. An increase in the dose of sodium carbonate leads to an increase in pH to 11, a decrease in pulp hardness to 0.3 mg-eq/l.

The data on upgrading rough concentrate are presented in Table 2. An increase in the contact time of sodium carbonate (bicarbonate) with concentrated water glass (C-30%) up to a day led to the formation of a gel-like heterogeneous mass and was not used in testing.

From the data in Table 2, it follows that with an increase in the proportion of sodium carbonate in the mixture, a more significant drop in the quality of the concentrate is observed due to an increase in the calcite content. Scheelite recovery for concentrates with the same quality is higher when finishing rough concentrates using the Petrov method than with a combination of modifiers.

In Kang et al. [3], the high technological results achieved in the cycle of selection of the rough concentrate with sodium sulfide are presented.

**Table 2.** Influence of modifiers on the flotation of Ca-minerals in the upgrading cycle.

Test conditions		Final scheelite concentrate						Upgrading tailings, %	
C, %	P, kg/t Sodium carbonate (B) + water glass	Aassy ( $\beta$ ), %			Recovery ( $\epsilon$ ), %			$\beta$	$\epsilon$
		WO <sub>3</sub>	CaCO <sub>3</sub>	P	WO <sub>3</sub>	CaCO <sub>3</sub>	P	WO <sub>3</sub>	
Rough concentrate of PCF – mass fraction WO <sub>3</sub> -7 %, CaCO <sub>3</sub> -32.6 %, P-1.83 %									
2.21	0+31.0	39.86	12.7	2.25	91.01	9.41	32.31	0.69	5.23
2.40	0+33.6	55.87	7.26	1.20	88.02	3.63	11.19	0.72	5.81
3.00	0+38.4	66.11	6.41	1.30	83.80	1.7	6.05	0.86	10.67
3.00	0.8+43.2	62.41	9.73	1.62	82.24	2.70	7.88	0.94	11.86
3.00	0.7 <sup>B</sup> +39.6	58.00	11.95	1.53	84.57	3.78	8.44	0.86	10.40
3.45	2.4+43.2	57.89	9.89	1.27	86.35	3.15	7.26	0.78	9.44
3.35	2.4 <sup>B</sup> +44.0	61.07	8.78	0.73	85.10	2.62	3.88	0.77	9.40
4.03	7.7+41.6	55.84	11.06	0.91	85.18	3.59	5.24	0.69	7.76
3.50	8.4+45.6	42.28	13.38	1.21	85.73	5.86	9.29	0.72	7.61

Note <sup>B</sup> bicarbonate was used in a mixture with water glass; contact of mixture components for 3 min;  
Abbreviations: C – residual concentration of water glass in steaming, P – dose of reagents

Sodium sulfide, sodium hydrosulfide, ammonium sulfide are widely used in the process of flotation of sulfide ores, less often in reagent modes of flotation of rare metal ores [2]. In sulfide flotation, soluble sulfide compounds perform a variety of functions from activator to suppressor. In aqueous solutions, according to the data on the solubility of sulfides and the degree of hydrolysis [6], ions and molecules are present [HS<sup>-</sup>], [OH<sup>-</sup>], [Na<sub>2</sub>S], [S<sup>2-</sup>]. As a result of the oxidation of the sulfide ion in the liquid phase of the pulp, thiosulfate ions [S<sub>2</sub>O<sub>3</sub><sup>2-</sup>] and sulfate ions [SO<sub>4</sub><sup>2-</sup>] can accumulate. The introduction of an environment regulator (Na<sub>2</sub>CO<sub>3</sub>, NaOH up to pH  $\geq$  11) increases the resistance of sodium sulfide to oxidation [11], and an increase in the pulp temperature during steaming T – 90°C slows down the decomposition of sodium sulfide due to a decrease in the solubility of oxygen in the pulp.

Soluble salts of heavy metals (for example, iron) have a significant effect on the flotation of calcite with a cationic collector in environment of sodium sulfide. It is assumed that the depression in this case in the presence of sodium sulphide is associated with the appearance on the surface of sludge coatings of sulfides formed in the pulp volume, which is facilitated by a low charge of the calcite surface.

It is believed that the oxyhydril collector on the calcite surface is kept at a temperature of agitation with sodium sulfide up to T – 90°C for 30 minutes [12].

Table 3 shows the results of upgrading rough concentrates at PCF with sodium sulfide. Steaming temperature 90°C, time 60 minutes. The yield of steaming feed was 7.1% of the ore, the mass fraction in the original rough concentrate: WO<sub>3</sub> – 4,7 %, P – 1,4 %, CaCO<sub>3</sub> – 41,6 %, extraction into concentrate from ore: WO<sub>3</sub> – 95 %, P – 71 %, CaCO<sub>3</sub> – 64 %.

As follows from the data in Table 3, heat treatment of the rough concentrate without water glass, in the environment of sodium hydroxide and sodium sulfide (experiments 1 – 3) does not allow obtaining the standard quality of the concentrate, the degree of WO<sub>3</sub> concentration is 1.1-1.5. Three recleanings of the steamed concentrate were carried out. The introduction of 4.9 kg / t into the steaming pulp of water glass (experiments 4) ensured the production of a conditioned WO<sub>3</sub> concentrate, recovery being 81%.

**Table 3.** The results of concentration in selection in different modes with Na<sub>2</sub>S and NaOH.

Test No.	Steaming conditions of rough concentrate at PCF	Assay (β), %			Recovery (ε), % WO <sub>3</sub>	WO <sub>3</sub> in final tailings, %	
		WO <sub>3</sub>	P	CaCO <sub>3</sub>		β	ε
					WO <sub>3</sub>		
1	Na <sub>2</sub> SiO <sub>3</sub> -0 kg/t; pH =9.63	5.59	1.49	43.95	94.25	0.50	0.39
2	NaOH-6.4 kg/t; pH-12.84	5.34	1.46	43.07	93.35	1.90	0.64
3	Na <sub>2</sub> S+NaOH – (4.9+3.5 kg/t); pH 13.0*	6.87	1.61	55.23	86.48	1.12	2.01
4	Na <sub>2</sub> SiO <sub>3</sub> – 4.9 kg/t; C-4.3%; pH-11.4	50.53	0.75	30.93	80.65	0.59	9.40
5	NaOH + Na <sub>2</sub> SiO <sub>3</sub> – (3.7+1.3 kg/t)	30.55	0.63	58.11	72.92	0.57	6.40
6	Na <sub>2</sub> S+NaOH + Na <sub>2</sub> SiO <sub>3</sub> – (4.9+3.5+3.3 kg/t); pH-13	54.00	0.64	23.16	68.37	0.73	7.70
7	Na <sub>2</sub> S+NaOH (pH-13) steaming; +Na <sub>2</sub> SiO <sub>3</sub> steaming (4.7+3.3+3.2 kg/t)	41.83	1.09	38.92	73.33	0.79	8.27

Experiment 7 reproduces the scheme is given in [3], but the results obtained on the rough concentrate at PCF  $\beta_{\text{WO}_3}$ -41.83% and  $\epsilon_{\text{WO}_3}$ -73.33% are lower than the standard mode by the Petrov method (experiment 4).

## 4 Conclusion

As a result of the studies performed to assess the change in the surface contrast of calcium-containing minerals based on the use of a combination of environment modifiers (sodium carbonate (bicarbonate), sodium hydroxide, sodium sulfide and water glass), it can be stated that it was not possible to achieve selective depression of calcite and apatite while maintaining effective scheelite flotation. The best results for rough scheelite concentrates of PCF were obtained using the standard technology with upgrading according to the Petrov method.

1. The use of sodium bicarbonate in comparison with sodium carbonate, at the same flow rates, creates a less alkaline pH of the pulp and greater rigidity in the flotation pulp. In open cycle experiments, similar indicators of scheelite enrichment were obtained, but in a closed cycle an increase in the concentration of hardness ions [Ca<sup>2+</sup>, Mg<sup>2+</sup>] in the liquid phase middling product pulp will lead to a significant increase in scheelite losses, or an increase in collector dose will be required.

2. Heat treatment of the rough concentrate at T = 85 – 90°C without water glass does not allow obtaining high-quality scheelite concentrates, a low degree of scheelite concentration of 1.2 has been achieved with a high degree of recovery of all calcium-containing minerals.

3. On a mixture of sodium carbonate (bicarbonate) and water glass, even with a slight increase in the dose of water glass in the mixture, after heat treatment, concentrates of lower quality were obtained, in comparison with the factory procedure by the Petrov method. With an increase in the proportion of sodium carbonate in the mixture, a more significant decrease in the mass fraction of scheelite in the concentrate is observed.

4. The conditional quality of the concentrate was not obtained from rough scheelite concentrate, in the environment of sodium sulphide and alkali (pH 13, dose 62 – 43 kg / t of steaming feed) without sodium silicate. With water glass and sodium sulphide, the results are significantly lower than using the PCF technology.

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