OBTAINMENT OF SCHEELITE-SULPHIDE PRECONCENTRATE FROM POLYMETALLIC ORE FROM "RUDNIK" MINE

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Abstract

The possibility of preconcentration of scheelit and sulphide minerals from mine "Rudnik" polymetallic ore, using the gravity concentration process was investigated in this paper. Because of wolfram minerals occurrence in some parts of mine, the aim of this investigation was the processing of the ore at the shaking table. With this process, the preconcentrat of heavy minerals (sulphide and scheelite) was obtained. That preconcentrate will be the starting material for flotation, and about 60% mass of waste material will be remove.

***Key words:** scheelit, preconcentration, sulfide minerals.*

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1. Introduction

Polymetallic ore (Pb-Zn-Cu), from mine "Rudnik", is utilized by mining pit. During the exploitation, the occurrence of wolfram-scheelite minerals ($CaWO_4$) was observed (1). As it is known, that the wolfram is of great economically importance, so the aim of this work was the attempt of exploitation the scheelite mineral from this bed. For its great importance, the wolfram is wide range used for production steel alloys, fibers for light bulbs, as well as powder for carbide production which is used as additive for cementation during sintering process. Wolfram has very high temperature of melting point (3410^0C), as well as the very high temperature of boiling point (5700^0C) (2), what leading to the unique property of wolfram, besides carbon, to heat at high temperatures without softening. Among known materials, the wolfram has highest value for stretch module ($E = 41.4 \times 1010$ N/m^2 (42190 Kp/mm^2)), what makes it most elastic material. At the room temperature the wolfram is resistant for acid influence, even including aqua regia. The mixture of fluorine acid and nitric acid can dissolve the wolfram (3).

2. Determination of physical/chemical and mineralogical properties of scheelite sample from mine "Rudnik"

The mass of scheelite sample, shaped from prospect hole IH-679-92 "New shaft" Rudnik - "Rudnik, was 175 kg, which made this sample repre-sentative. The Figure 1 represented the place and the way of sampling.
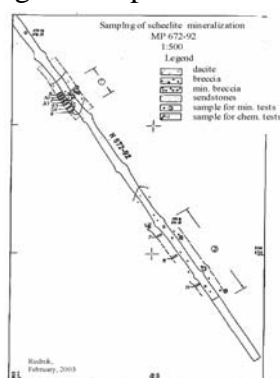

Fig. 1. The way of sampling of the scheelite sample at mine "Rudnik"

2.1. Chemical analysis of scheelite ore sample

Table 1. Chemical analysis of sample from prospect hole IH-672-92

Sample mark	Content,									
	%								g/t	
	WO$_3$	W	Mo	Pb	Zn	Fe	S	Cu	Ag	Au
0	1.06	0.84	0.016	0.27	0.06	1.86	0.42	0.01	9	trace
1	0.787	0.625	/	0.02	trace	1.68	0.62	trace	5	trace
1/1	0.094	0.075	/	0.02	0.02	1.77	0.63	trace	2	trace
2	0.032	0.025	/	0.06	0.02	4.94	2.06	0.03	7	trace
3	0.063	0.05	0.005	0.87	1.54	3.25	1.90	0.03	13	trace
3/1	1.92	1.52	0.010	0.61	0.63	2.60	1.03	0.05	15	trace
4	8.82	7.00	/	0.38	0.03	2.51	1.03	0.01	77	trace
4/1	0.126	0.10	/	0.16	0.06	3.26	0.65	0.01	19	trace
5	1.512	1.20	/	0.44	0.05	2.61	/	0.02	88	trace
5/1	0.063	0.05	/	0.10	0.07	1.77	/	0.01	22	trace
6	0.035	0.028	0.013	0.39	0.09	0.37	0.59	0.03	5	trace
7	0.032	0.025	/	0.08	0.03	4.38	/	0.02	13	trace
8	0.032	0.025	/	0.68	0.16	5.12	/	0.02	47	trace
11	0.032	0.025	/	0.32	0.07	3.54	/	0.02	5	trace

The main sample was consist of fourteen samples (streak method was used) and the results of chemical analysis of those samples is given in Table 1. It could be observed (Table 1.) that amount of WO$_3$ varied through series during analysis from 0.03% to almost 9%. With the respect to valuable elements, in one of tests was observed significant amount of Zn (about 1.5%) and for all tests the different amount of Ag was observed. According to these results, from those series of samples, two composites were made for the following processing procedure. The composites were marked as K1 and K2 and the both of them have mass of 34 kg.

Table 2. Chemical analysis of composite K2

	Content, %														g/t	g/t
	WO$_3$	Mo	Pb	Zn	Cu	Fe	Bi	S	SiO$_2$	Al$_2$O$_3$	MgO	CaO	K$_2$O	Na$_2$O	Sn	Ag	Au
Com K-2	0.82	0.01	0.41	0.08	0.006	2.81	0.08	1.38	59.7	14.2	1.6	3.57	3.49	1.27	>0.02	36	0.25

2.2. Mineralogical analysis

With the aim to identify the macroscopic mineral composition of samples, mineralogical analysis was performed using stereomicroscope with reflected light. Qualitative microscopic mineralogical analysis was performed under reflected as well as under transmitted light using polarization microscope (4).

2.2.1. Qualitative microscopic analysis of composite K2 (6 mine and 6 thin section samples)

Macroscopic structure: Sample is consist of slim to powdery aggregates (100% -2.0mm) made from light grey magmatic rock and altered sandstone. It could be clearly observed the deposit and isolated crystalls of scheelite (observed under scheelite tube - short waves). Also, among scheelite crystalls it could be seen minor sulphide mineralization with visible pyrhotite crystalls.

Mineralogical composition: pyrrhotite, pyrite, marcasite, chalcopyrrhotite, chalcopyrite, cubanite, native bismuth, platinum group of minerals, Pb-Pi sulphosalts, axinite, Pb-Ag-Bi sulphosalts, Ag-telluride, galena, sphalerite, arsenopyrite, linnaeite, molybdenite, scheelite, magnetite, hematite, limonite, cerusite, rutile, leucoxene, sphene, anatase, k-feldspar (sanidine, adular), plagioclase, garnets, amphiboles, biotite, chlorites, clinozoisite, epidote, zircon, apatite, kaolinite, calcite.

Microscopic structure: Scheelite occurance in idiomorphic developed grains, uncommonly formating thin fibre with quartz crystals. The most occurance of scheelite is in shape of isolated grains, which are placed in sulphide-quartz matrix, or rearly in sulphide-quartz-carbon matrix. Scheelite minerals contains small pins, drops and shapeless oval grains of native bismuth and Pb-Bi(Ag) sulphosalts. The presence of scheelite in silicate matrix is not deremined.

Pyrrhotite is the most common sulphide mineral that appears in its characteristic tabular forms. Based on the visual optical characteristics it responds to the hexagonal and monoclinic varieties. It is always replaced with Pb-Bi-sulphosalts, chalcopyrite, arsenopyrite and partially transformed into

pyrite during oxidative processes.

Native bismuth appears either within pyrrhotite aggregates or as inclusions in scheelite, occurring in the form of drops. From Pb-Bi(Ag)-sulphosalts for the time being are determined heyrovskite ($Pb_6Bi_2S_9$) and axinite ($CuPbBiS_3$). The apperence of Ag-Pb-Bi sulphosalts is possible. The exact determination of them is possible by applying electronic microsonde. The other minerals appiers in small degree. The primary rock, according to its mineral composition and structure, belongs to quartzlatite and sandstones.

2.2.2. *Quantitative microscopic analysisis of composite K2 (6 mine and 6 thin section samples)*

Quantitative microscopy analysis were performed at sample K2, which was sieved in six classes: 1) -2+0.9 mm; 2) -0.9+0,5 mm; 3) -0.5+0.25 mm; 4) -0.25+0.1 mm; 5) -0.1+0.063 mm; 6)-0.063+0.0 mm. All classes, exept the one marked as 6 class, have been used for gravity concentration. No good results were observed with classes marked as class 1 and class 2. Class marked as 6 (-0.063+0.0 mm) is provide for direct flotation.

Quantitative microscopic analysis was done at the main mineras, as follows:

1) Sulphide minerals: pyrrhotite, pyrite, marcasite, arsenopyrite, halkopyrite, sphalerite, galena, Pb-Bi(Ag) sulphosalts, native bismuth

2) Oxide minerals: scheelite, hematite, limonite, cerusite

3) Waste minerals: biotite, clinozoisite, garnets, zircon, apatite, leucoxene, quartz, calcite, K-feldspar, plagioclase.

The composite K2 is consist of sulphide minerals (about 6%), oxide minerals (about 1%) and waste minerals (about 93%). Scheelite appears like free standing grains (about 60%) as well as like complex and simple intergrowth (about 40%), impregnation and incluzion. The mostly part of intergrowth scheelite is like impregnation to quartz. Pyrrhotite, among all sulphide minerals, is present with 66%.

3. Experimental procedure

The aim of this work was to get scheelite concentrate with 60% WO3.

Experimental work relating to this paper consisted of gravity concentration with the aim to get preconcentrate of scheelite and sulphide minerals from ore.

The preconcentrate, observed like mention before, will be used as starting sample for flotation process, just in case that using gravity concentration method will not be able to separate sulphide minerals from scheelite. The flotation process will be consist of two stages: the first stage including obtainment of collective concentrate of sulphide minerals and the second stage is based on receiving the scheelite concentrate. With the aim to recieve satisfide quality of obtained sceelite concentrate the magnetic and electrostatic concentration can be performed. Also, using the #L fraction from gravity concentration, the feldspar concentrate will be observed using flotation method in acid medium. The characteristisc that scheelite concentrate have to require is given in Table 3.

Table 3. Market requirement for scheelite concentrate quality

Element or compound	Concentrate of natural scheelite, %	Concentrate of artificial scheelite, %
WO_3, min.	60.00	60.00
Sn, max.	0.10	0.05
Cu, max	0.05	0.05
As, max	0.10	0.05
Sb, max	0.10	0.05
Bi, max	0.25	0.25
Mo, max	0.40	0.25
P, max	0.05	0.05
S, max	0.50	0.40
Mn, max	1.00	0.25
Pb, max	0.10	0.10
Zn, max	0.10	0.10

One more market requirement for scheelite concentrate is that the grain size in concentrate should be higher than 0.5 mm for 90% of concentrate, so the sintering of flotation concentrate is required to be providing (5). In the case that there is no possibility to obtaining the flotation concentrate with satisfied quality, the contamination is necessary to be chemically removed. Solvated tungsten is precipitated in the form of artificial scheelite (6). Totally weight of all samples which were used for preparation of composites K1 and K2 is 175 kg. Top size of grains was about 60 mm, so after

the two-stage grinding and mineralogical analysis determined the top size of grains for gravity preconcentrate process. The scheme of scheelite sample treatment is shown in Figure 2.
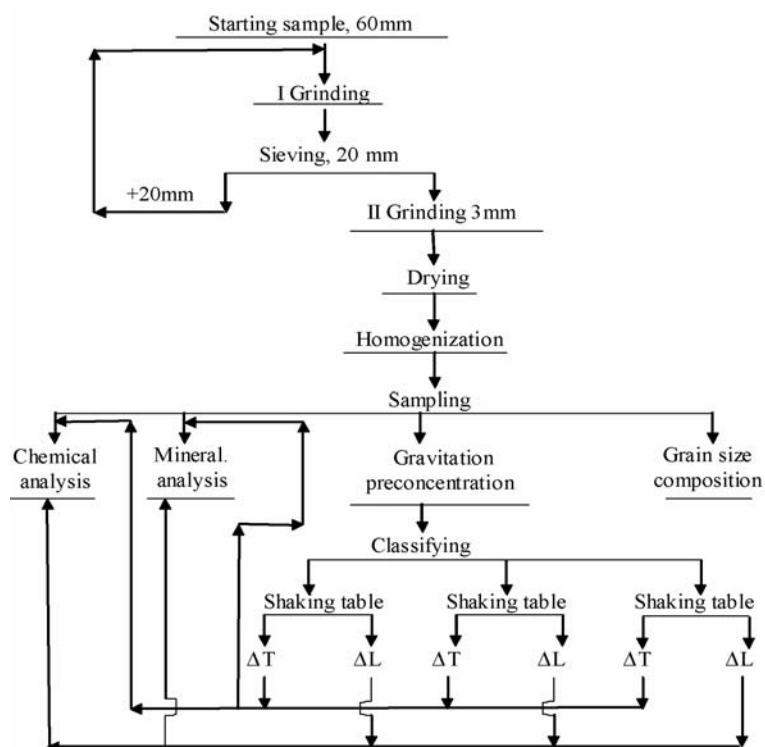

Fig. 2. Written scheme of treatment of scheelite sample K2

3.1. *Determination of composition according to grain size of scheelite sample K2*

At starting sample of composite K2, after grinding by shortcone grinder "Symons", with the outcome of 3 mm is determined composition according to grain size by sieving through a series of sieves. Each of sieved classes was weighing and their mass is represented in Table 4.

Table 4. Composition of scheelite sample K2 according to grain size

Size class [mm]	M, %	↓ΣM, %	↑Σ M, %
-4.76 + 3.36	0.29	0.29	*100.00*
-3.36 + 2.38	1.31	1.60	*99.71*
-2.38 + 1.68	2.86	4.46	*98.40*
-1.68 + 1.19	6.25	10.71	*95.54*
-1.19 + 0.833	14.01	24.72	*89.29*
-0.833 + 0.589	13.50	38.22	*75.28*
-0.589 + 0.417	16.89	55.11	*61.78*
-0.417 + 0.295	8.96	64.07	*44.89*
-0.295 + 0.208	7.34	71.41	*35.93*
-0.208 + 0.149	6.54	77.95	*28.59*
-0.149 + 0.104	4.03	81.98	*22.05*
-0.104 + 0.074	4.80	86.78	*18.02*
-0.074 + 0.053	1.23	88.01	*13.22*
-0.053 + 0.037	2.10	90.11	*11.99*
-0.037 + 0.0	9.89	100.00	*9.89*
Input	**100.00**	/	/

Based on mineralogical analysis and values for specific mass of each useful mineral and minerals waste in sample K2, it was decided to perform gravity concentration on sample K2. Applying of the gravity concentration method at shaking table is required sieving of sample, according to concentration criteria, to classes on which can be applied concentration procedure (7, 8). According to results of mineralogical analysis and intergrowth of minerals, as well as according to the fact that scheelite mineral is smoother than quartz mineral (Moss's hardness is 4.5 to 5), it was decided the top size of grain for shaking table to be 2 mm. Sample of 2+0.0 class is sieved to four classes and the mineralogical and chemical analysis were performed with the aim to determine the distribution of scheelite sample according to grain size. These results are represented as metallurgical balance in Table 5.

Table 5. Metallurgical balance of metals of composite K2 ore according to grain size

Size class, mm	M, %	W, %	WO$_3$, %	R/WO$_3$, %
-2+0.9	36.22	0.45	0.57	26.69
-0.9+0.5	19.53	0.57	0.72	18.17
-0.5+0.25	11.56	0.63	0.80	11.95
-0.25+0.1	10.60	0.81	1.02	13.97
-0.1+0.062	5.66	0.91	1.15	8.41
-0.062+0.0	16.43	0.78	0.88	20.81
Input	100.00	0.61	0.76	100.00

On the basis of values for specific mass of quartz, $\gamma SiO_2 = 2.7 \text{ g/cm}^3$, as main component in waste mineral, for specific mass of scheelite, $\gamma CaWO_3 = 6.0 \text{ g/cm3}$, for specific mass of other useful minerals, as well as on criteria of concentration (about $\xi = 3$) it was decided to sieve the sample for shaking table for the first series of tests to classes described below:

-2+0.9 mm
-0.9+0.5 mm $\qquad \varepsilon = \gamma CaWO_3 - 1/\gamma SiO_2 -1 = 6-1/2.7-1 = 3$
-0.5+0.25mm
-0.25+0.1mm
-0.1+0.062 mm
-0.062+0.0mm directly to flotation

According to results of mineralogical analysis, free standing scheelite grains appears at values lower than 0.9 mm for all classes for shaking table, so the concentration criteria taken to be lower than 3.0. Lower concentration criteria and narrow classes are chosen for intergrowth minerals (9, 10, 11). However, such a way of classifying for the first class gives no results at shaking table (visually). For the next class, after the processing at shaking table, the products are given to mineralogical microscopic analysis using scheelite tube. According to the results, the performed analysis didn't give expected results. So, it was decided to break the class -2.0 + 0.5 mm to class -0.5 mm and to sieve on two classes (-0.5 + 0.15 mm and -0.15 + 0.045 mm) with concentration criteria of $\xi = 3$. The idea of such a sieving was to determine the possibility of getting satisfied concentration with wider classes using lower grain sizes with higher liberation. So, during the second series of test at shaking table were treated follows classes:

-0.5 + 0.15 mm
-0.15 + 0.045 mm
-0.125 + 0.037 mm
-0.045+0.0 mm directly to flotation

Those classes are treated at shaking table, in such a way that after treatment of each class the two products were observed ΔT and ΔL. Each of those products is decontaminated with the aim to better differentiation of minerals. After that, all products were sent to mineralogical analysis. The result of mineralogical analysis shows that the differentiation is well done. When this was confirmed, all products were sent to chemical analysis.

The results of chemical analysis in metallurgical balance are shown in Table 6.

Table 6. Metallurgical balance of the first series of preconcentration tests of scheelite ore K2

Products, mm	M*, %	Mtot. %	Pb, %	W, %	WO₃, %	Na₂O, %	K₂O, %	Ag, g/t	Bi, %	IWO₃, %	IPb, %	IAg, %	IK₂O, %
-0.5+0.25 ΔT	18.89	5.50	2.00	3.12	3.93	0.63	1.68	175.0	0.300	23.32	19.75	19.03	2.68
-0.5+0.25ΔL	81.11	23.64	0.09	0.05	0.063	1.48	4.22	7.36	0.029	1.61	3.79	3.44	28.98
cl. -0.5+0.25	100.00	29.14	0.45	0.631	0.793	1.32	3.74	39.0	0.080	24.93	23.54	22.47	31.66
-0.25+0.1 ΔT	14.58	3.90	3.44	5.31	6.69	0.53	1.22	275.0	0.500	28.14	24.09	21.20	1.39
-0.25+0.1 ΔL	85.42	22.82	0.07	0.037	0.047	1.49	4.10	12.72	0.02	1.12	2.78	5.74	27.18
cl.- -0.25+0.1	100.00	26.72	0.56	0.805	1.015	1.35	3.68	51.0	0.090	29.26	26.87	26.94	28.57
-0.1+0.062ΔT	13.22	1.89	4.56	6.75	8.51	0.42	1.16	400.0	0.650	17.35	15.47	14.95	0.63
-0.1+0.062ΔL	86.78	12.38	0.09	0.025	0.032	1.48	4.10	8.09	0.016	0.43	1.95	1.98	14.75
cl. -0.1+0.062	100.00	14.27	0.68	0.914	1.153	1.34	3.71	60.0	0.100	17.78	17.42	16.93	15.38
-0.5+0.062ΔT	16.10	11.29	2.93	2.08	6.03	0.56	1.43	247.21	0.428	68.82	59.31	55.18	4.70
-0.5+0.062ΔL	83.90	58.84	0.08	0.04	0.05	1.48	4.15	9.59	0.023	3.16	8.52	11.16	70.91
cl.-0.062+0.00	100.00	29.87	0.60	0.670	0.87	1.06	2.81	57.0	0.10	28.03	32.17	33.66	24.39
Input	**100.00**	**100.00**	**0.56**	**0.735**	**0.927**	**1.25**	**3.44**	**50.58**	**0.092**	100.0	100.00	100.00	100.00

M*- in this column are given the mass % calculated with the respect on each class

3.2.Comment on results of preconcentration of the first series of tests

According to the results shown in Table 6, preconcentration process is well done on each of three classes under the grain size interval of -0.5 to 0.062 mm, what is confirmed by obtaining the preconcentrate of ΔT fraction in what were concentrated scheelite and sulphide minerals, and of ΔL fraction in what were concentrated the silicate minerals (feldspar above all). An insufficiency was recognized in relatively high content of class -0.062 mm (almost about 30%) with the respect to input. Also, the content of sulphide (before of all galena) and scheelite minerals is high, the advantage of those minerals (including Ag) in this class is high. An anomaly was observed in the first two classes: in class -2+ 0.9 mm and in class -0.9+ 0.5 mm. The separation of

"soft" and "hard" minerals from those two classes didn't give satisfied results, as it was well done in three other classes, mention before. According to these results it could be concluded that liberation of minerals in this two classes is not satisfied (10). With the aim to improve the liberation of minerals, those two classes were mixed and grinned to class -0.5 mm, and then to classify at two classes, -0.5+0.15mm and -0.15+0.045 mm with concentration criteria $\xi = 3$. The class with lesser grain size, staying after classifying, -0.045+0.00 mm, will directly go to flotation. According to this, the mass content of class with lesser grain size which is directly goes to flotation is smaller with the respect to the first series of probes. All products of second series of probes are chemically analyzed and the results are represented in Table 7.

Table 7. Metallurgical balance of the second series of preconcentration tests of scheelite ore K2

Products, mm	M*,%	Mtot.,%	Pb, %	W, %	WO₃ %	Na₂O, %	K₂O, %	Ag, g/t	Bi, %	IWO₃, %	IPb, %	IAg, %	IK₂O, %
-0.5+0.15 ΔT	18.02	11.58	1.06	2.31	2.91	0.87	2.82	76.0	0.260	51.76	44.02	33.64	9.25
-0.5+0.15ΔL	81.98	52.64	0.07	0.074	0.094	1.48	3.98	9.02	0.016	7.6	13.22	18.14	59.34
cl. -0.5+0.15	100.00	64.22	0.25	0.475	0.602	1.35	3.77	21.1	0.060	59.36	57.24	51.78	68.59
-0.15+0.045ΔT	34.24	5.97	1.23	2.371	2.99	0.74	2.61	93.0	0.280	27.41	26.18	21.22	4.41
-0.15+0.045ΔL	65.76	11.46	0.095	0.037	0.047	1.62	4.04	9.80	0.014	0.83	3.89	4.29	13.12
cl. -0.15+0.045	100.00	17.43	0.484	0.833	1.05	1.32	3.55	38.3	0.105	28.24	30.07	25.51	17.53
-0.5+0.045ΔT	21.49	17.55	1.12	2.332	2.94	0.82	2.75	81.78	0.267	79.17	70.20	54.86	13.66
-0.5+0.045ΔL	78.51	64.10	0.07	0.068	0.086	1.51	3.99	9.16	0.156	8.43	17.11	22.43	72.46
cl.-0.045+0.00	100.00	18.35	0.194	0.348	0.44	1.07	2.67	32.4	0.050	12.40	12.69	22.71	13.88
Input	100.00	100.00	0.28	0.52	0.65	1.29	3.53	26.2	0.066	100.00	100.00	100.00	100.00

3.3. Comment on results of preconcentration process of additionally classified ore

On the basis of the scheelite ore preconcentration results (Table 7) it could be concluded that the preconcentration process is well done because the ΔT fraction consist of sulphide and scheelite minerals and ΔL fraction is consist of silicate minerals (feldspar is of great interest) even the classes treated at shaking table were wider, in other words the concentration criteria was $\xi = 3$.

By comparison of the first and the second series of tests given in table 6 and table 7, respectively, it could be observed that in the first series of tests,

where the classes are narrowly, the better quality of product is observed with high insufficiency, so the summary ΔT fraction represented just 13 mass % with the respect to input on table (calculated without class -0.062 mm); at the same time the high insufficiency of WO_3 (about 97.6%) was observed. In the second series of tests, where the wider classes are presented, the quality of products is not satisfied, meaning that the summary of ΔT fraction is above 34 mass % what is much higher with the respect to input; the insufficiency of WO_3 is about 96%. Similarly, all of this can be applied on all other mineral with the respect to whish fraction have been concentrate, what leading to conclusion that the preconcentration with additionally classified ore is insufficiency. In the second series of tests, the mass percent of series (-0.045+0.0 mm) which is not treated on shaking table is lower (about 18%) than in the first series of tests where the mass percent of class (-0.062+0.00 mm) which is not treated on shaking table is higher (about 30%).

4. Conclusions

On the basis of experimental results observed during experimental work it could be concluded:

- applying the gravity concentration method on composite ore who is consist of sulphide minerals, is not possible to get concentrate of scheelite mineral higher than 60% because concentrating of scheelite mineral influence the concentrating of sulphide minerals from those paragenesys;

- if the aim is to observed the concentrate of scheelite mineral by applying the gravity concentration method, than it is necessary to process ore from the part where the scheelite and silicate minerals are present without sulphide content;

- if the aim is to get high quality product using preconcentration process it is necessary to make more narrow classifying of samples with the respect to classifying for applying concentration process (Table 6 and 7) because of very complex mineral paragenesys; if the aim is to get smaller number of classes, the quality of observed preconcentrate will be lower for the same insufficiency;

- from the results of metallurgical balance, presented in Table 6 and Table 7, it could be observed that applying the preconcentration method

allowing the elimination of 60% of waste minerals, represented like ΔL fraction; also it could be observed that in ΔL fraction are concentrated the feldspar minerals and that those fraction is a good start point for concentrate the minerals mention above; the ratio K_2O to Na_2O is about 3:1 what makes the insufficiency of those mineral in this fraction of about 65%;

    - from the results of metallurgical balance, presented in Table 6, it could be observed that applying the preconcentration method is good because the mass percent of summary preconcentrate is only 11.29% with the respect to starting sample; degree of concentration for scheelite is about 7 times with the insufficiency of about 69%; degree of concentration for Pb and Ag is about 5 times with the insufficiency of about 66%;

    - also, from the results of metallurgical balance, represented in Table 6 and Table 7, it could be observed that there is great difference in mass of this classes, which have not been treated at shaking table (classes -0.062+0.0 mm and 0.045+0.0 mm; difference in mass 29.87% and 18.35%, respectively), with respect to input; the conclusion of results represented below could be that it is necessary to down size of grains which prepared for treatment at shaking table, with the aim to the lower mass of starting samples go directly to flotation process, without primarily applying the preconcentration process.

5. Aknowledgements

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