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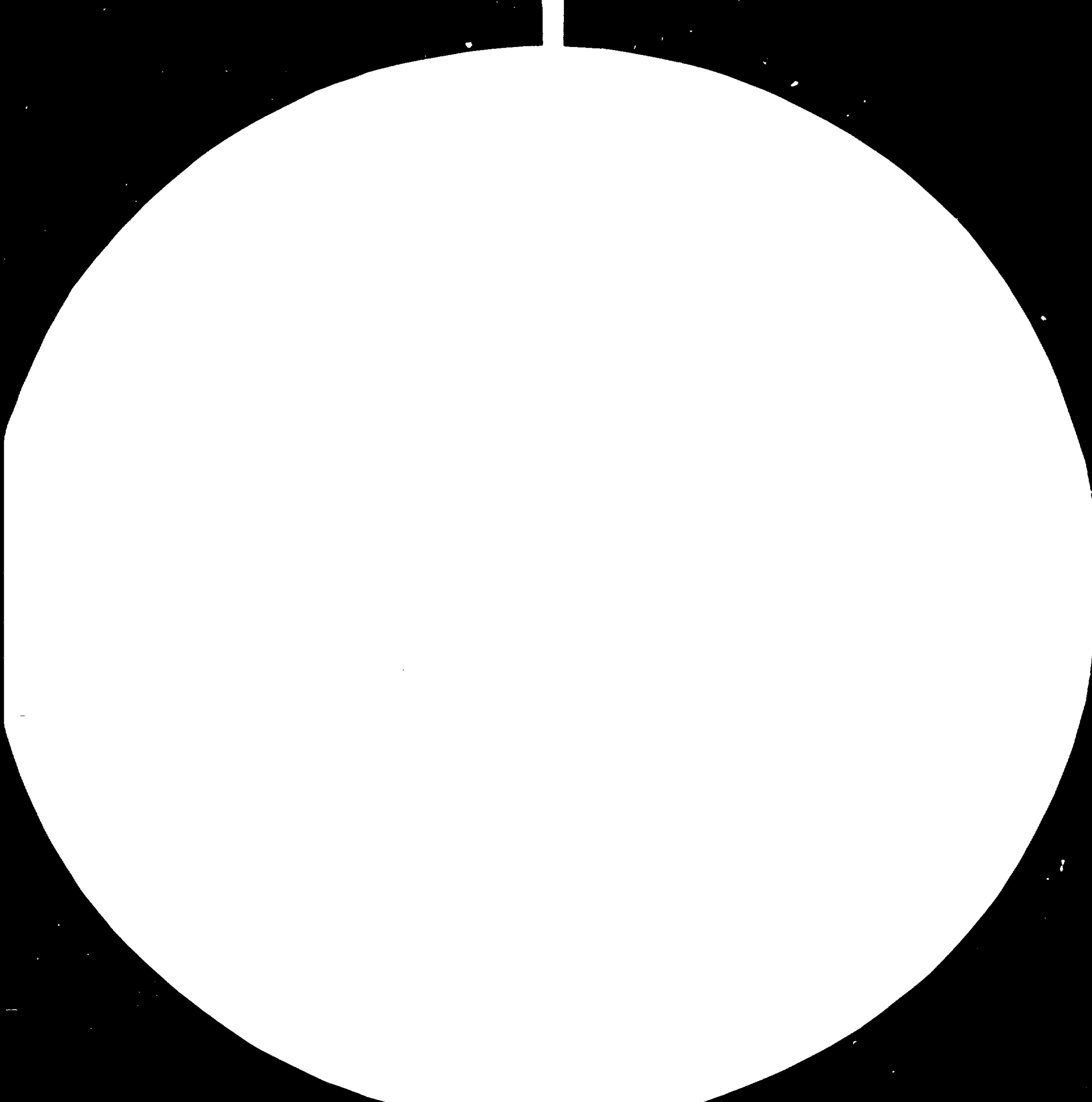

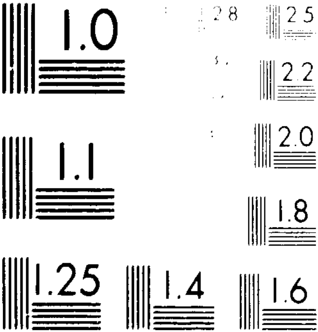
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TSVETMETPROMEXPORT

10129

LABORATORY TESTS FOR DETERMINING THE AMENABILITY OF THREE SAMPLES OF MASSIVE SULPHIDE POLYMETALLIC ORES TO BENEFICIATION

/PORTUGAL/

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LABORATORY TESTS FOR DETERMINING THE AMENABILITY OF THREE SAMPLES OF MASSIVE SULPHIDE POLYMETALLIC ORES TO BENEFICIATION

(PORTUGAL)

FINAL REPORT

Developed by
Scientific-Research and Design Institute
of Mechanical Treatment of Minerals
"MEKHANOBR"
1980

ABSTRACT

The amenability of three samples of pyrite polymetallic ore of complex composition, that had been sent from Portugal, was investigated. The ore is characterized by fine non-uniform dissemination of sulphides in pyrite.

Bulk-selective flotation was found to be advantageous for these ores. Lead, copper and zinc concentrates were produced (samples 1 and 2) which proved suitable to be handled by conventional methods of metallurgical treatment, the same concerns the zinc product of the sample 3 (zinc content 37--40%).

Recoveries of lead, copper and zinc in corresponding concentrates were 30-42.6%, 50.4 - 67% and 55-69.8% respectively.

In principle, the KIVZET - process can be used for treating the semi-products obtained according to the above flowsheet.

Bulk lead-copper-zinc flotation concentrates can be treated by using an oxidation-autoclave method with subsequent metals recovery from leach products. The results obtained qualify the combined flowsheet to be technologically competitive with the bulk-selective flotation flowsheet.

Basic combined flowsheets for ore treatment have been proposed.

CONTENTS

1. INTRODUCTION

The so-called cupriferous pyrite is one of the rare, but most important minerals in Portugal.

The Portuguese pyrite zone is located in the province Alentejo. This is one of the largest deposits of massive pyrites in the world, its reserves exceeding 200 mln. tons. The deposit can be specified as a massive complex sulphide mineralization, since its ores contain galena, sphalerite and copper minerals.

Pyrites in Portugal have been mined for many years as raw materials for industries using sulphur and for extraction of non-ferrous metals from calcines. Amounts of ore mined and treated are very small. Preliminary investigations on pyrites, according to the information available, conducted in 1972-73 (we don't know the country where the testwork was done), made it possible to conclude, that these pyrites can be regarded not only as sulphur-bearing raw material, containing sulphur, but as a polymetallic ore as well, which is especially important taking into account a constantly increasing demand for non-ferrous metals inside the country. This factor may be a major impetus for an increase in production of pyrites mined.

However, the characteristics of the Portugese pyrites and their complex mineral and chemical composition made it necessary to carry out special extensive investigations. The present investigation is a part of such investigations, accomplished, within the frame of contract N 78/87 between the United Nations Industrial Development Organization (UNIDO) and V/O TSVETMETPROMEXPORT, on the subject "Laboratory tests for determining the amenability of three samples of massive sulphide polymetallic ores to beneficiation (Portugal)".

The main objective of this investigation was to determine probable process data and to develop an expedient flowsheet for beneficiation of three pyrite-bearing ore samples.

As specified in the contract, in the process of conducting these investigations, the Customer has been given two intermediate reports: the first one entitled "Mineral and Chemical Composition of Samples" was delivered in February, 1979; the second one under the title "Investigations of Ore Treatment by Flotation" was presented in September, 1979. The latter gives the results of the investigations based on sample treatment by bulk-selective flotation and by bulk flotation with subsequent selection of bulk lead-copper-zinc concentrate. The report comprises no results on selective flotation, since in the process of this research investigation no expediency to continue the investigations in this direction was realized.

The present report is a final report where in separate sections are presented the results of the research investigations concerned with a hydrometallurgical treatment of a bulk-lead-copper-zinc concentrate (ore samples 2 and 3) and a zinc concentrate recovered from ore sample 3. In addition a conclusion is drawn on a feasibility of the "KIVZET" - process for some beneficiation products.

Since the results of the investigations on mineral and chemical composition and those of the flotation tests on ore samples are completely presented in the two above-mentioned intermediate reports, in the present report these parts are set forth to such an extent, so as to make a general evaluation of the samples amenability to beneficiation.

2. Mineral and chemical composition of ore samples.

The composition of the ore samples under investigation was specified on the basis of the study of their mineral, chemical and phase composition (see Tables 2.1 - 2.4), and a following conclusions was drawn: the ore of the samples contains 81.4-88.2% pyrite; it means, that, as a matter of fact, it is a pyrite polymetallic ore with dissemination of lead, copper, and zinc minerals. Its characteristic feature is a fine and rather non-uniform dissemination of all valuable minerals in the pyrite, this

Table 2.1.

Results of spectral analysis

Elements	Sensitivity	Content in samples, %		
		1	2	3
Silicon	0.001	tenths	beginning integers	integers
Aluminium	0.001	tenths	tenths	tenths
Magnesium	0.0003	tenths	tenths	tenths
Calcium	0.005	hundredths	hundredths	hundredths
Iron	0.001	integers	integers	integers
Manganese	0.001	tenths	tenths	hundredths
Nickel	0.001	thousandths	thousandths	thousandths
Cobalt	0.001	hundredths	hundredths	hundredths
Titanium	0.001	thousandths	thousandths	thousandths
Molibdenum	0.0003	thousandths	thousandths	thousandths
Zirconium	0.003	thousandths	thousandths	thousandths
Copper	0.001	integers	tenths	integers
Lead	0.01	integers	integers	integers
Antimony	0.01	hundredths	tenths	hudnredths
Silver	0.001	thousandths	thousandths	thousandths
Bismuth	0.001	hundredths	thousandths	hundredths
Arsenic	0.1	tenths	tenths	tenths
Zinc	0.1	integers	integers	integers
Cadmium	0.01	thousandths	thousandths	thousandths
Tin	0.003	hundredths	hundredths	hundredths
Barium	0.03	hundredths	tenths	tenths
Yttrium	0.001	traces	traces	traces

The following elements were not detected in samples: chromium, vanadium, tungsten, hafnium, niobium, tantalum, baryllium, thallium, germanium, gallium, indium, lanthanum, strontium, lithium, phosphorus, uranium, thorium, scandium, cerium, rhenium.

Table 2.1.

Results of chemical analysis of ore samples.

Components	Content, %		
	Sample 1	Sample 2	Sample 3
Lead	1.22	0.95	0.80
Copper	0.85	0.30	1.17
Zinc	2.86	2.68	2.08
Sulphur	48.46	47.47	44.50
Iron	40.73	40.37	38.75
Arsenic	0.86	0.78	0.78
Aluminium oxide	0.14	0.22	0.18
Magnesium oxide	< 0.05	~ 0.2	< 0.05
Calcium oxide	0.14	0.16	0.16
Silicon oxide	0.65	2.42	7.80
Manganese	0.12	0.10	0.08
Cobalt	0.0366	0.012	0.043
Titanium oxide	< 0.03	< 0.03	< 0.03
Cadmium	0.0066	0.009	0.0068
Bismuth	0.022	0.0066	0.019
Sodium oxide	0.15	< 0.010	< 0.10
Potassium oxide	0.3	0.1	0.3
Tin	< 0.01	0.019	0.013
Molibdenum	< 0.005	< 0.005	< 0.005
Antimony	0.05	0.107	0.065
Mercury	0.011	0.010	0.010
Ore contains, g/t			
Gold	0.85	0.82	0.45
Silver	20.9	42.7	20.94

Table 2.3.

Results of phase chemical analysis
for detecting compounds of lead, copper and zinc.

Types of compounds	Content, %					
	absolute			relative		
	Samples					
	1	2	3	1	2	3
Lead compounds:						
GALENA	1.02	0.79	0.68	83.6	83.2	85.1
OXIDES	0.09	0.06	0.03	7.4	6.3	3.7
LEAD COMBINED WITH PYRITE	0.11	0.10	0.09	9.0	10.5	11.2
TOTAL LEAD	1.22	0.95	0.80	100.0	100.0	100.0
Copper compounds:						
CHALCOPYRITE	0.42	0.05	0.77	48.8	15.0	63.1
GRAY ORE	0.44	0.26	0.44	51.2	85.0	36.1
SECONDARY SULPHIDES	-	-	0.01	-	-	0.8
TOTAL COPPER	0.86	0.31	1.22	100.0	100.0	100.0
Zinc compounds:						
SPHALERITE	2.57	2.39	1.91	89.9	89.2	93.2
SMITHSONITE	0.29	0.29	0.14	10.1	10.8	6.8
TOTAL ZINC	2.86	2.68	2.05	100.0	100.0	100.0

Mineral composition and size of mineral inclusions in

Item No.	Mineral	Sample 1				Sample 2		
		Content, %	Size of inclusions, mm			Content, %	Size of inclu	
			from	up to	dominating		from	up
	<u>Ore minerals</u>	99.0				96.0		
1	Pyrite	88.2	0.004	9.0	0.04-0.1	87.4	0.004	10.
2	Sphalerite	5.0	0.001	3.0	0.02-0.03	4.3	0.002	0.
3	Arsenopyrite	1.7	0.004	0.1	0.01-0.02	1.6	0.008	0.
4	Chalcopyrite	1.3	emulsion--type inclusions less than 0.001	0.2	0.01-0.02	0.2	emulsion--type inclusions	0.9-
5	Galena	1.3	0.001	0.1	0.006-0.02	1.1	0.002	0.
6	Smithsonite	0.3	0.01	0.2	0.03-0.05	0.5	0.01	0.
7	Gray ore x/	1.0	0.004	0.1	0.01-0.03	0.5	0.008	0.
8	Bournonite	0.1	0.004	0.05	0.01-0.02	0.2	0.03	-0.0
9	Boulangerite	0.1	0.008	0.05	0.01-0.03			
10	Anglesite	solitary grains	0.02	-0.03		solitary grains	0.03	-0.0
11	Pyrrhotite	solitary grains	0.008	-0.02				
12	Stannite	solitary grains						
13	Magnetite	-	--	-	-	0.2	0.1	0.
14	Bornite	-	-	-	-	-	-	-
	<u>Non-metallic minerals</u>	1.0				4.0		
15	Quartz and chalcedony	0.6	0.01	0.6	0.05-0.1	2.1	0.01	0.
16	Sericite	0.2	0.01	0.2	0.04-0.06	1.2	0.008	0.
17	Chlorite	0.2	0.02	0.2	0.03-0.05	0.3	0.01	0.
18	Feldspars	solitary grains	0.05	-0.1		0.4	0.05	-0.
19	Calcite							

NOTE: x/ In samples 1 and 3 - tennantite, in

SECTION 1

Table 2.4

and size of mineral inclusions in samples

	Sample 2				Sample 3			
	Content, %	Size of inclusions, mm			Content, %	Size of inclusions, mm		
inating		from	up to	dominating		from	up to	dominating
	96.0				91.3			
.04-0.1	87.4	0.004	10.0	0.06-0.1	81.4	0.002	6.0-7.0	0.04-0.1
.02-0.03	4.3	0.002	0.5	0.02-0.04	3.4	0.002	1.0-2.0	0.01-0.03
.01-0.02	1.5	0.008	0.2	0.02-0.04	1.5	0.004	0.1	0.01-0.03
.01-0.02	0.2	emulsion-type inclusions	0.9-1.0	0.008-0.04	2.3	emulsion-type inclusions	0.4-0.5	0.01-0.03
.06-0.02	1.1	0.002	0.2	0.01-0.03	0.8	0.002	0.2	0.02-0.03
.03-0.05	0.5	0.01	0.2	0.03-0.05	0.6	0.02	0.2	0.03-0.05
.01-0.03	0.5	0.008	0.4	0.03-0.04	1.0	0.004	0.2	0.02-0.03
.01-0.02	0.2	0.03	-0.04		0.3	0.002	0.1	0.02-0.03
.01-0.03								
	solitary grains	0.03	-0.04		solitary grains	0.02	-0.03	-
-	0.2	0.1	0.15	0.03-0.04	-	-	-	
-	-	-	-	-	solitary grains	0.004	-0.01	
	4.0				8.7			
.05-0.1	2.1	0.01	0.25	0.05-0.1	7.5	0.02	1.0-2.0	0.06-0.1
.04-0.06	1.2	0.008	0.25	0.02-0.03	0.6	0.01	0.4	0.03-0.05
.03-0.05	0.3	0.01	0.1	0.03-0.04	0.3	0.02	0.2	0.03-0.05
	0.4	0.05	-0.08		0.3	0.03	-0.05	

samples 1 and 3 - tennantite, in the sample 2 - tetrahedrite.

SECTION 2

dissemination varying from 0.001-0.008 mm up to the predominant size of the following inclusions: for galena 0.006-0.02 mm (sample 1) and 0.01-0.03 mm (samples 2 and 3); for chalcopyrite 0.01-0.03 mm (samples 1 and 3) and 0.008-0.04 mm (sample2); for gray ore 0.01-0.04 mm; for sphalerite 0.02-0.03 mm, the latter containing 4-7% of iron as an isomorphous admixture. It should be noted that quite often mineral grains contain microinclusions of pyrite, and it was just the presence of finest inclusions of pyrite and sphalerite that made it unfeasible to obtain a zinc concentrate from sample 3.

The presence of finest inclusions of galena in the pyrite is confirmed by the fact, that in the ores and beneficiation products there is galena "combined" with pyrite; this galena was detected by phase chemical analysis at the size of the ground sample under investigation 92-95%- 0.44 mm.

The presence of minerals showing a complex composition, such as bournonite ($CuPbSb_2$), boulangerite ($Pb_5Sb_4S_{11}$) and gray ore (tennantite in samples 1 and 3 and tetrahedrite in sample 2) complicated, as it will be evident further on, the process of minerals selection, and, as a result, a concentration of antimony within the lead and copper concentrates occurred. Ores contain arsenic and mercury, the latter being concentrated within zinc concentrates (660-1200 g/t of ore).

Thus, in spite of the commercial contents of lead, zinc and copper in the samples, due to the afore-mentioned features of the ore mineral and chemical composition, the samples under consideration are difficult for beneficiation.

3. Investigations of ore samples amenability to beneficiation.

3.1. Conditions of testwork.

The size of the ore samples delivered was 10-100 mm. After the general sample preparation and provision of average samples, to study the mineral and chemical composition of the ore, the ore intended for flotation testwork was subjected to

grinding, in small portions, up to the size of -3 mm, in view to prevent excessive oxidation of sulphides during storage. As practice showed, an oxidation of sulphides still occurred, which manifested itself in a decreased flotation acitivity of copper and especially lead minerals.
The natural pH of the pulp decreased from 5,1 - 5,8 at the start of the testwork to 4,4 - 4,8 at its completion, which seemed to be the result of increased amounts of ferrous iron and sulphates in the pulp.

The grinding of the ore weighted portions and the regrinding of the beneficiation products were effected in laboratory ball mills designed by the Mekhanobr Institute. Ball charge and balls of a corresponding size were chosen for different stages of grinding; thus, for the regrinding of the beneficiation products, 3-5 mm balls were used.

The ore was ground with a solid/liquid ratio 1 : 0,5, while for the regrinding of the beneficiation products, this ratio was 1 : 1.

Rougher and scavenger operations were performed in a 3 l flotation cell. The rest of the operations were handled by using 1 to 0.05 l cells. All the flotation cells were of mechanical type simulating industrial machines designed by the Mekhanobr Institute. The air feed rates, controlled by flowmeters and rotameters, amounted in all operations to 1 l per 1 l of cell per minute.

In the laboratory investigations a sedimentation technique was used for classification of materials. When classifying, 0.01 - 0.02 mm fractions were removed.

Standard reagents used in the investigation were those largely applied at domestic concentrators. All reagents, except lime and activated charbon, were added as aqueous solutions. Methylisobutylcarbinol (MIBC) was used as a frother for all the beneficiation circuits.

The concentration of hydrogen ions was measured with a glass electrode.

The tests simulating a continuous process with the use of four, five or ten batches (the latter was the case with the lead-copper flotation) have been run for four to five days. One batch was treated for a day with all final products recovered. The middlings of the corresponding circuits remained to be treated the next day. The composition of water used in the flotation tests is given in Table 3.1.

Table 3.1.

Composition of water used in flotation tests

Components	Unit of measurement	Content
pH	-	6.7
Alkalinity	mg.-eq.	0.32
Oxidability	mgO_2/l	5.9
Dry residue	mg/l	79.5
Total iron	mg/l	0.16
Calcium	mg/l	7.5
Sulphates	mg/l	21.5
Chlorides	mg/l	8.5
Bicarbonates	mg/l	15.0
Total hardness	mg-eq.	0.91

3.2. Investigations with the use of bulk-selective flotation circuit

3.2.1. Lead-copper rougher flotation.

It was considered expedient to grind the ore before the flotation , so as to produce a minimum yield of + 0.044 fraction,which was done on the basis of the character of valuable minerals dissemination in the pyrite and taking into account the data obtained as a result of the testwork on a probable desintegration of mineral interlocks during the grinding process.

These analyses showed that in the + 0.044 mm fraction 70% to 90 - 100% of sphalerite, chalcopyrite, gray ore and practically total galena are presented by interlocks, while a noticeable desintegration of interlocked minerals could be observed only beginning from a -0.03 + 0.019 mm fraction. Yet it was advisable to minimize sulphides sliming, since the experience had proved that increased amounts of fine slimes of valuable minerals at the presence of pyrite not only result in a decreased recovery of valuable components, but sharply deteriorate the process selectivity. The tested regimes of grinding and classification did not allow to avoid sulphides sliming. Thus, when the -0.044 mm fraction amounted to 94.7%, 91.9% and 92.5% in respective samples 1,2 and 3, a yield of a -0.010 mm fraction was 34-39% (see diagrams in Fig. 3.1). A slight increase in the -0.044 mm fraction content was accompanied by a considerable increase in the -0.010 mm fraction yield, which, as a result,would have an adverse effect on technological results.

At a fine non-uniform dissemination of valuable minerals, as a rule, multi-stage grinding and flowsheets are used. However, the tests conducted on the tested samples, ground in two--five stages, showed that at the optimum rate of reagent addition for each ore sample under laboratory conditions the ore can be ground in two stages, since subsequent grinding stages did not result in a somewhat appreciable increase in metals recovery,due mainly to the above-mentioned character of dissemination. The frother flotation products assaying a low metal content are represented by pyrite comprising inclusions of lead, zinc, and copper minerals of the size not exceeding 0.002-0.008 mm.

In the rougher lead-copper flotation circuit, after a number of reagent addition patterns had been tested for depressing sphalerite and pyrite, there has been applied such a pattern,that ensured the most efficient effect, as compared to other patterns, though even in this case metal recoveries obtained were not high.

This pattern is based on using sulphoxide compounds in alkaline medium with subsequent oxidation of the pulp, before flo-

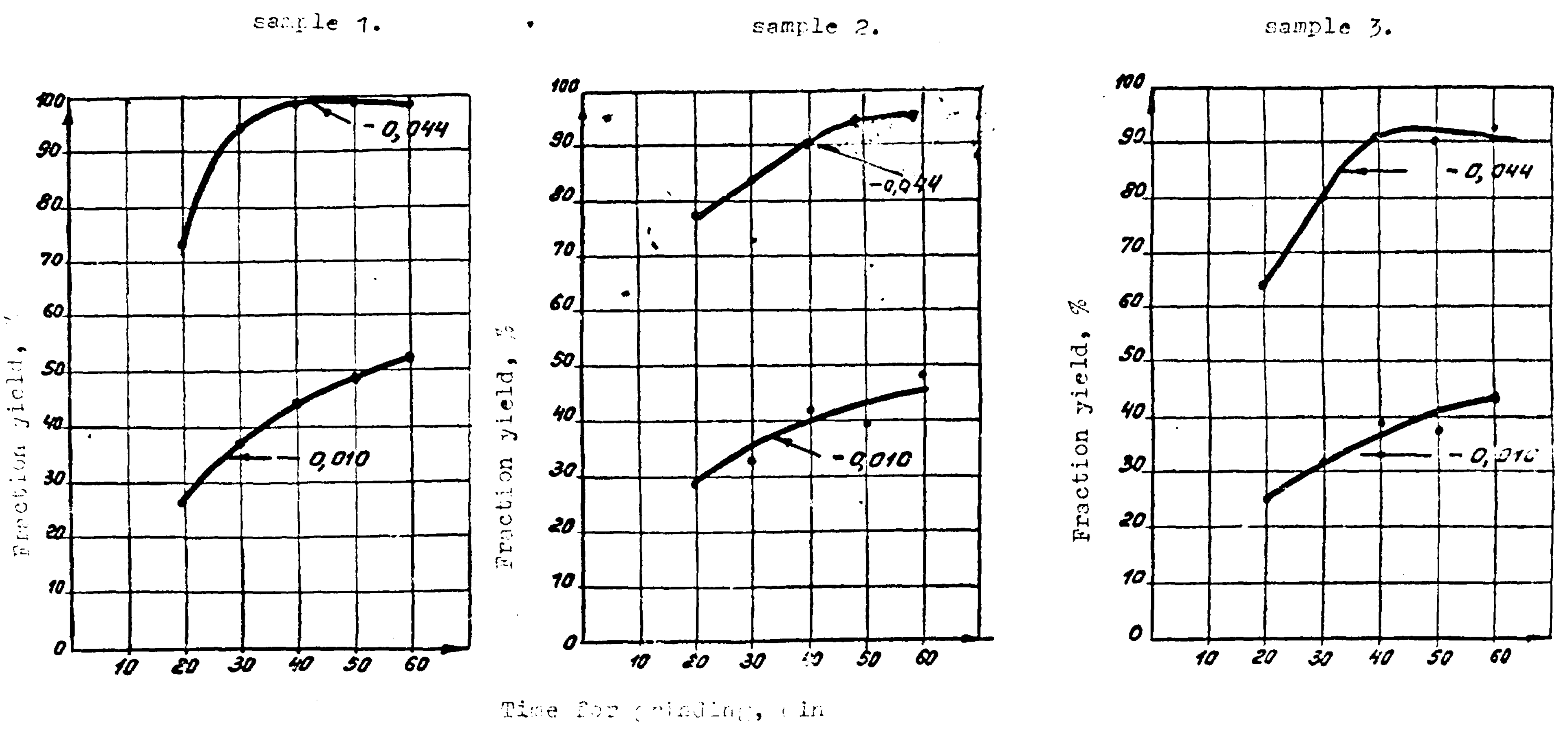


Fig. 3.1. 0.044 mm and 0.01 mm fraction content as a function of time for ore sample grinding

tation, and addition of zinc sulphate and cyanide into this oxidation operation. This method of pyrite depression is under the law protection in the USSR.

Sodium dithionite ($Na_2S_2O_4$) was used as a sulphoxide compound and soda - as an alkali modifier. Sodium dithionite is a technical product used in textile industry.

The required degree of oxidation, before flotation, was controlled according to the pulp activity to oxygen, by measuring the level of oxygen demand the solid phase of the pulp, in ml/g.hr.

The application of other reductants, as well as the changing of soda for lime had a negative effect upon the technological results. The pyrite and sphalerite depression in the cleaning operations was achieved by adding reagents similar to those fed into the rougher flotation circuit.

The test results of the compared reagent addition patterns (as estimated on the data, obtained in the rougher lead--copper flotation of sample 1) are given in Table 3.2. The results testify the efficiency of the pattern selected as compared with the other ones tested. Comparatively low lead and copper recoveries into a rougher lead-copper concentrate seems to be a consequence of the character of dissemination of the above sulphides in the pyrite and of their considerable sliming in the grinding operations. Thus, losses of lead and copper in the 0.005mm fraction in the tailings of the lead-copper flotation are as high as 30-40 % each; but at the same time a predominating amount of lead and copper minerals, contained in +0.005 mm fractions, is presented by inclusions in the pyrite; the size of these inclusions is such that they could not be liberated by the grinding means available.

The flowsheet and the reagent addition patterns for the closed-circuit flotation tests were determined on the basis of the open-circuit flotation tests. The results of the tests carried out with the use of the optimum flowsheets and reagent addition patterns for each individual ore sample are listed in Table 3.3, the flowsheets are given in Figs. 3.2 and 3.3. Due to the unsatisfactory distribution of the middlings produced under closed-circuit conditions in the I-st recleaning circuit for the lead-copper concentrate of ore sample 2, these middlings were removed from the process as a final product.

Table 3.2.

Results of comparative lead-copper flotation tests carried out on ore sample I.

Product	Yield, %	Content, %			Recovery,%			Total lead and copper recovery, %	Selectivity index, %	Remarks
		lead	zinc	copper	lead	zinc	copper			
Lead-copper concentrate	17.2	4.15	3.90	77.5	15.2	80.5	158.0	142.8		Flotation conditions in Fig. 3.2.
Lead-copper concentrate	16.7	3.50	1.48	3.68	54.7	9.1	68.5	123.2	114.1	Test has been carried out under conditions other than those in Fig.3.2.

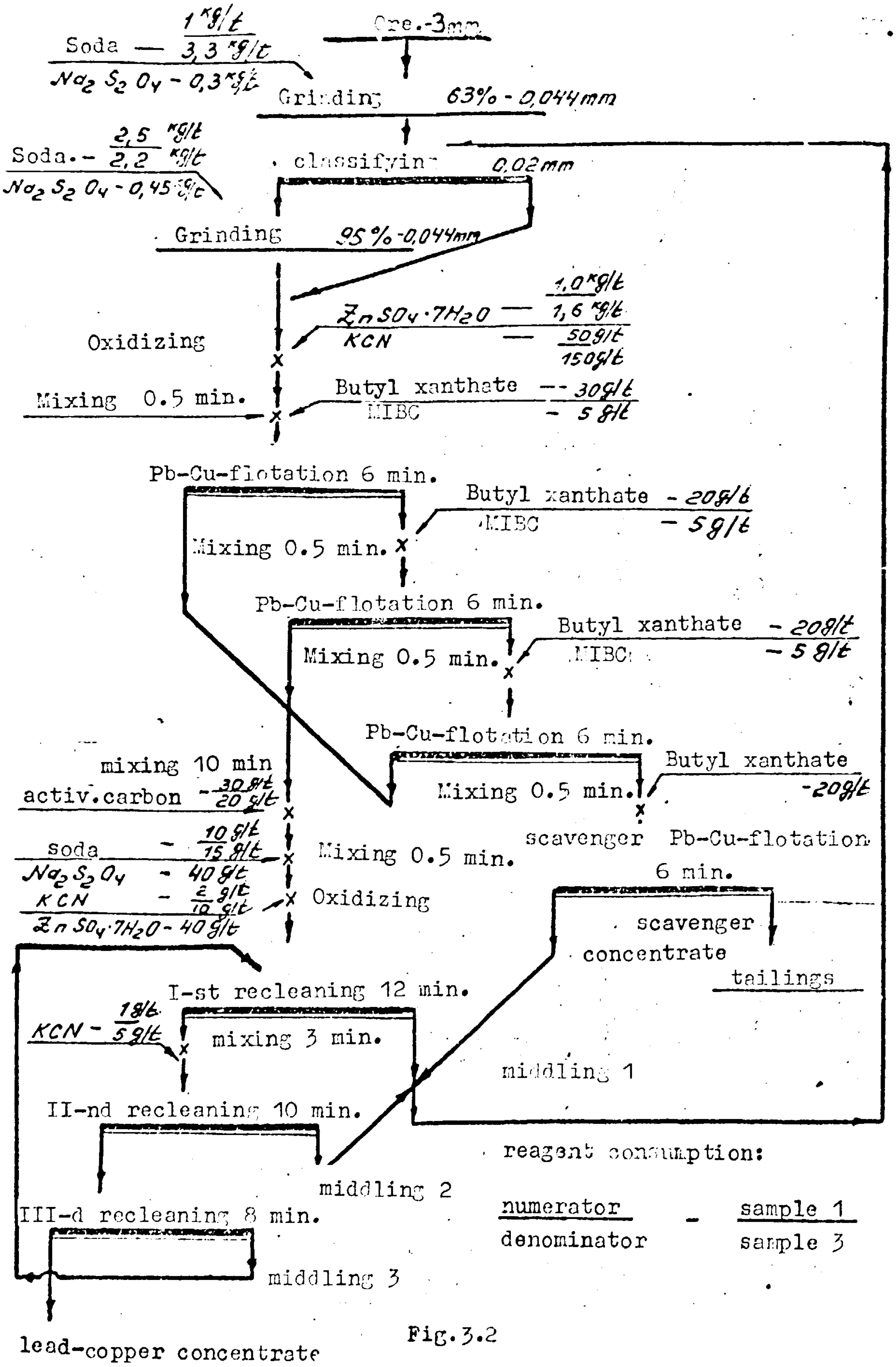


Fig.3.2

Flowsheet, applied in tests, coducted on the principle of continuos operation (tests 88 and 1049)

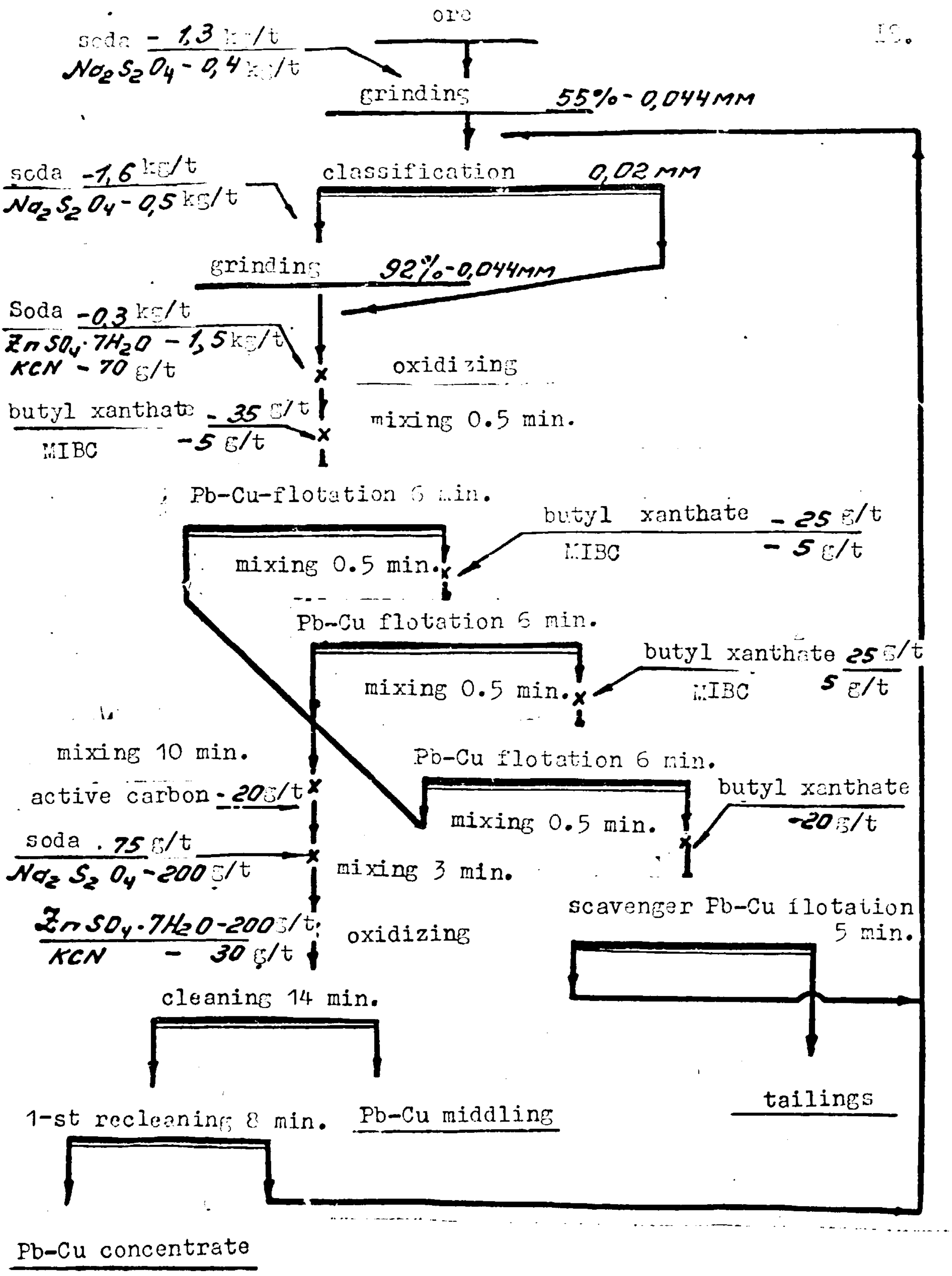


Fig.3.3. Flowsheet, applied in test 550, conducted on the principle of continuous operation

Table 3.3.

Results of lead-copper flotation tests carried out under continuous process conditions

Sample No.	Test No.	Product	Yield, %	Content, %			Recovery, %			Remarks
				lead	zinc	copper	lead	zinc	copper	
1	88	Lead-copper concentrate	7.5	10.44	2.88	8.53	65.3	8.0	77.6	Fig. 3.2
		Tailings	92.5	0.45	2.68	0.20	34.7	92.0	22.4	
		Ore	100.0	1.20	2.70	0.33	100.0	100.0	100.0	
2	550	Lead-copper concentrate	4.4	11.27	5.90	4.68	58.3	9.6	66.3	
		Lead-copper middlings	7.7	0.95	3.64	0.20	8.6	10.4	5.0	Fig.3.3
		Tailings	87.9	0.32	2.45	0.10	33.1	80.0	28.7	
		Ore	100.0	0.85	2.69	0.30	100.0	100.0	100.0	
3	1049	Lead-copper concentrate	6.8	7.47	4.06	13.91	62.9	14.7	80.8	
		Tailings	93.2	0.32	1.86	0.24	37.1	85.3	19.2	Fig.3.2
		Ore	100.0	0.81	2.01	1.17	100.0	100.0	100.0	

The expediency of the application of this technique when beneficiating the above sample was governed by the mineral and chemical composition of the product, namely: at a product size 100% - 0.2 mm, free lead and copper minerals are practically absent in it; galena, chalcopyrite and tetrahedrite are represented by 0.002-0.08 mm inclusions in pyrite, seldom in sphalerite. Small amounts of bournonite and boulangerite contained in the product are 0.002-0.004 mm inclusions in pyrite as well. When conducting the tests on ore samples 1 and 3 the distribution of the middlings produced in the lead-copper flotation circuit under conditions of a continuous process was satisfactory.

3.2.2. Zinc flotation.

The presence of a large amount of pyrite in the ores,their intimate association with all sulphides including sphalerite, as well as the presence of an isomorphous iron admixture in sphalerite made it difficult to obtain high grade zinc concentrates.

The zinc recovery from all the ore samples did not cause extreme difficulties without preliminary thickening of the lead--copper flotation tailings. The sphalerite activation was carried out by means of copper sulphate; the pyrite depression was conducted through pulp aeration with lime being fed into this operation up to pH = 11.8 - 12.0 in the flotation process,which corresponded to a 280 - 300g/m3 content of free calcium oxide in the pulp, and mixing, before flotation, with small amounts of cyanide.

A number of collectors tested in this flotation circuit has not shown any advantage over butyl xanthate, with the use of which all subsequent investigations were conducted. The investigations have shown an advisability to beneficiate the middlings of the zinc flotation circuit of all ore samples in a separate circuit and to remove from the process a part of the pyrite as a cell product (zinc flotation tailings). Highly various and, on the whole, low metallurgical results were obtained in the zinc flotation circuit (Table 3.4). When treating ore sample 2, with a zinc recovery into a zinc concentrate being 69.3% (as considered from the ore), there has been produced a zinc concentrate with 51.38%

Table 3.4.

Results of zinc flotation tests carried out under continuous process conditions

Sample No.	Test No.	Product	Yield,%		Content, %			Recovery, %						Remarks
			from ore	per operation	lead	zinc	copper	lead		zinc		copper		
								from ore	per operation	from ore	per operation	from ore	per operation	
1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
		Zinc concentrate	3.0	3.2	0.50	50.31	0.28	1.3	3.5	55.5	61.6	0.9	4.3	
		Zinc concentrate of middlings flotation	1.0	1.1	0.75	35.94	2.54	0.7	1.8	13.2	15.1	2.9	13.5	Fig.3.4
1	98	Total zinc concentrate	4.0	4.3	0.56	46.63	0.86	2.0	5.3	68.7	76.7	3.8	17.8	
		Zinc flotation tailings	13.4	14.4	0.45	2.25	0.39	5.2	14.2	11.0	12.4	6.0	27.2	
		Tailings	75.5	81.3	0.45	0.35	0.14	28.8	80.5	9.3	10.9	12.3	550	
		Lead-copper flotation tailings	92.9	100.0	0.45	2.61	0.20	36.0	100.0	89.5	100.0	22.1	100.0	
		Zinc concentrate	3.6	4.1	0.52	51.88	0.26	2.2	6.6	69.3	86.6	3.0	10.4	
		Zinc flotation tailings	5.5	6.3	0.48	1.36	0.16	3.1	9.4	2.8	3.5	2.8	9.8	Fig.3.5

1	2	3	4	5	6	7
2	550	Tailings	78.8	89.6	0.30	0.27
		Lead-copper flotation tailings	87.9	100.0	0.32	2.45
3	1049	Zinc concentrate	2.5	2.7	0.38	40.31
		Zinc concentrate of middlings flotation	2.8	3.0	0.72	14.2
		Total zinc concentrate	5.3	5.7	0.56	2.65
		Zinc flotation tailings	7.6	8.2	0.50	0.73
		Tailings	79.7	86.1	0.32	0.21
		Lead-copper flotation tailings	92.6	100.0	0.35	1.76

8	9	10	11	12	13	14	15
0.09	27.8	84.0	7.9	9.9	22.9	79.8	
0.10	33.1	100.0	80.0	100.0	28.7	100.0	
0.64	1.1	2.9	49.6	62.0	1.4	5.9	
1.74	2.4	6.2	19.5	24.3	4.1	17.8	Fig.3.4
0.70	3.5	9.1	69.1	86.3	5.5	23.7	
0.42	4.6	11.8	2.7	3.4	2.7	11.7	
0.22	30.9	79.1	8.3	10.3	14.9	64.6	
0.29	39.0	100.0	80.1	100.0	23.1	100.0	

x)Reagent consumption is represented in g/t of ore

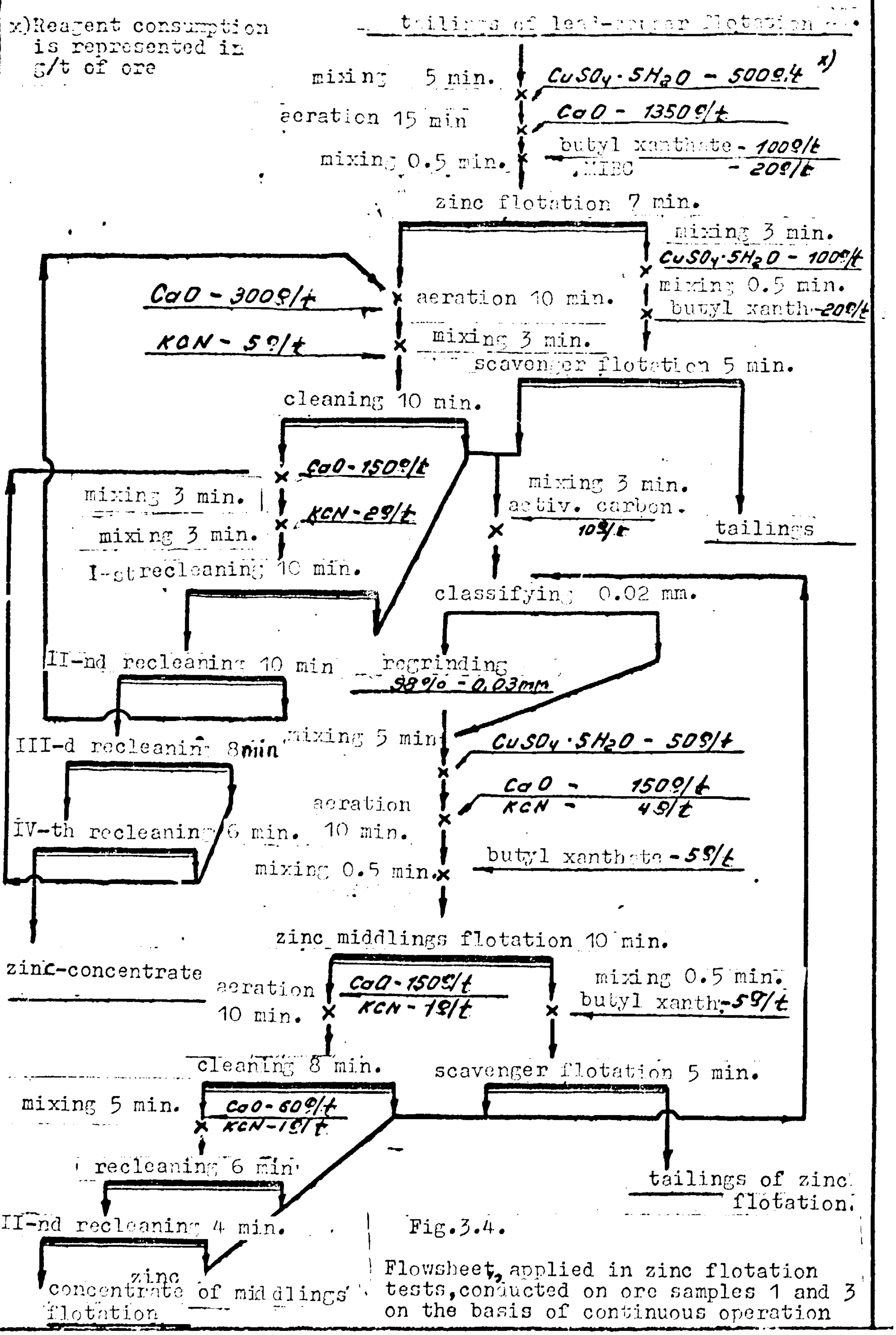
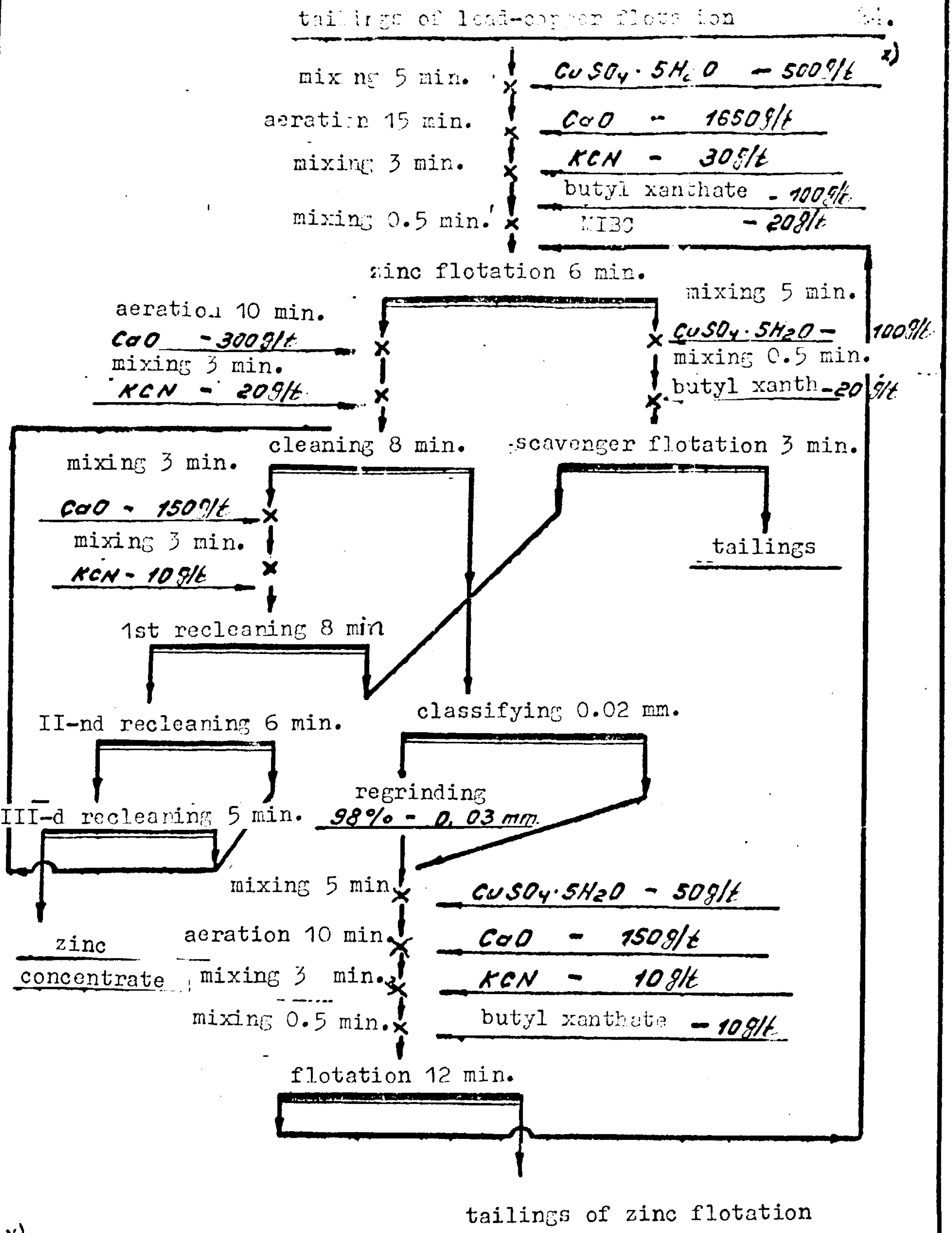


Fig.3.4.

Flowsheet, applied in zinc flotation tests, conducted on ore samples 1 and 3 on the basis of continuous operation



x) reagent consumption in g/t

Fig.3.5. Flowsheet, applied in zinc-flotation tests, conducted on the basis of continuous operation on ore sample 2.

zinc (tests 550, Fig. 3.5). When carrying out the tests on ore sample 1 according to the flowsheet in fig. 3.4 (test 98), a zinc concentrate containing 50.31% zinc was produced with zinc recovery into it being 55.5%; a semiproduct was produced from the middlings; into this semiproduct 13% of zinc was recovered with its zinc content being 35.94%. When treating ore sample 3, no zinc concentrate was produced by using all the flowsheets tested. In the best test (test 1059, Table 3.4) carried out according to the flowsheet in Fig. 3.4, two zinc products were obtained - one containing 40.31% zinc, the other - 14.2% zinc, with zinc recoveries into them being 49.6% and 19.5% - respectively.

Known flotation methods, used for pyrite removal and zinc content increase in zinc concentrates and products, have not yielded satisfactory results in this case, that can be explained by the presence in these products of large amounts of free 0.008-0.02 mm sphalerite grains, as if "filled" with macroinclusions of pyrite, and larger grains of pyrite with sphalerite inclusions, the size of which does not exceed 0.006-0.008 mm.

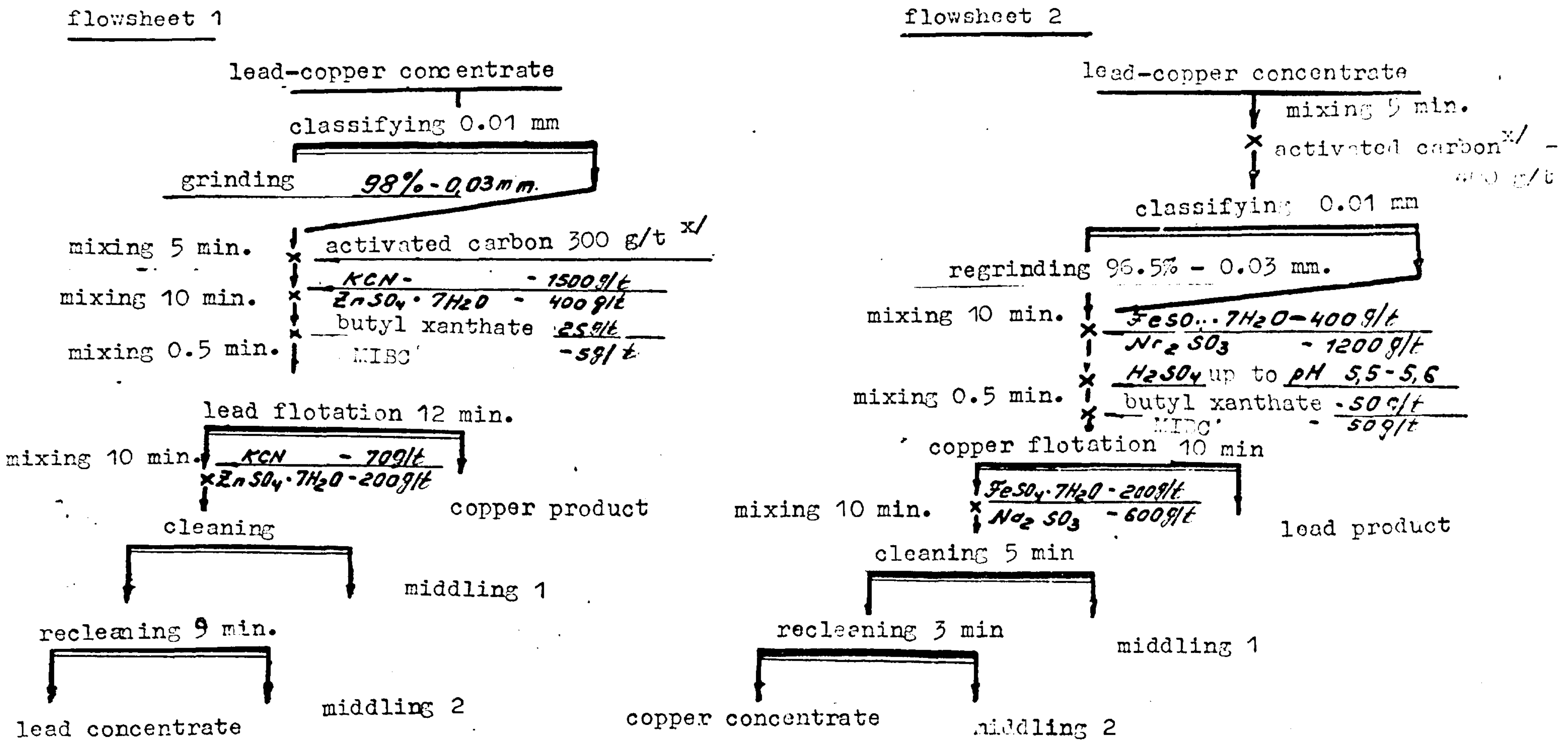
3.2.3. Lead-copper concentrate selection.

In the practice of selective flotation one of the difficult tasks is lead-copper concentrate separation. In our case the task was aggravated in that the product to be separated contained a large amount of pyrite and was rather a fine material (100% - - 0.030 mm fraction), and in spite of the regrinding operation prior to separation, the pyrite occurred not only in a free state, but also with inclusions of minerals to be separated.
As it was mentioned above, the lead minerals were represented not only by galena, but also by bournonite and boulangerite, the copper ones - by chalcopyrite, gray ore (tennantite and tetrahedrite), whose behaviour during the separation process was not clear. According to the chemical phase analysis, the lead-copper concentrate contains copper in the form of gray copper within the range of 44-48% (samples 1 and 3) to 90% (sample 2).

Table 3.5.

Results of Pb-Cu concentrate separation tests in the open cycle

Sample Nos.	Test Nos.	Product	Yield, %	Content, %			Recovery, %			Selectivity index, %	Remarks
				Pb	Zn	Cu	Pb	Zn	Cu		
1	90	Pb-concentrate	13.3	37.5	2.36	4.82	51.9	10.3	7.8	44.1	Fig.3.6 Flowsheet 1
		Middlings 2	6.7	7.75	2.95	10.90	5.4	6.5	8.9		
		Middlings 1	22.2	6.15	3.00	11.00	14.3	21.8	29.6		
		Cu-product	57.8	4.71	2.06	7.66	28.4	61.4	53.7		
		Pb-Cu-concentrate	100.0	8.24	3.05	8.24	100.0	100.0	100.0		
2	352	Cu-concentrate	15.6	4.10	3.35	18.25	5.5	9.4	69.0	63.5	Fig.3.6 Flowsheet 2
		Middlings 2	3.1	10.20	4.30	4.08	2.7	2.4	3.1		
		Middlings 1	21.9	11.40	4.70	1.65	21.7	18.5	8.8		
		Pb-product	59.4	13.60	6.52	1.33	70.1	69.7	19.1		
		Pb-Cu-concentrate	100.0	11.53	5.56	4.12	100.0	100.0	100.0		
3	1065	Pb-concentrate	12.5	36.16	3.85	4.75	63.1	9.7	4.5	58.6	Fig.3.6 Flow sheet 1
		Middlings + Cu-product	87.5	3.01	5.13	14.93	36.9	90.3	95.5		
		Pb-Cu-concentrate	100.0	7.16	4.97	13.66	100.0	100.0	100.0		



x) reagent consumption represented in grams per ton of bulk copper-lead concentrate

Fig. 3.6. Flowsheet, applied in tests on lead-copper concentrate separation

In the present investigations cyanide and cyanide - free flowsheets were used because of their ability to provide the highest selection results at the stage of research. In both cases, before the separation process, a desorption of reagents with activated coal was carried out, the consumption of the latter being 75 - 80 g/t of ore; this corresponded to a 350 - - 400 g/t bulk concentrate. The cyanide technique provides for a concentrate treatment before separation by cyanide and zinc sulphate in a soda medium, at pH = 9 - 9.5 for copper minerals depression, the second technique serves for concentrate treatment - by ferrous sulphate and sodium sulphate at pH = 5.5 - 5.6, created by sulphuric acid, to depress lead minerals. The cyanide - free flowsheet was required for separation of the lead-copper concentrate obtained from ore sample 2, since during the tests it was found out that in the presence of large amounts of tetrahedrite the separation of lead minerals from copper ones by cyanide was unsatisfactory. The copper minerals floated actively into a froth product together with the lead minerals, and their floatability increased with greater cyanide addition. It should be noted, that in our case due to the large amounts of pyrite in the lead-copper concentrate, with the use of both reagent patterns, out of the cell products, there have not been obtained corresponding concentrates - additional beneficiation was required for the flotation circuit tailings. The results of several lead--copper concentrate separation tests carried out in an open circuit under optimum conditions are shown in Table 3.5, the flowsheets and test conditions are given in Fig. 3.6. The method of lead-copper concentrate separation in the absence of cyanide is under the law protection in the USSR.

3.2.4. Testwork concerned with production of pyrite concentrate.

The tailings of the ore flotation circuit for ore samples 1 and 2 contain 50-58% of sulphur and are, as a matter of fact, a high quality pyrite concentrate: a pyrite flotation circuit is envisaged only for beneficiation of ore sample 3. With this purpose in view, after the zinc flotation ~~circuit, the~~ tailings of this circuit were thickened up to 50-60 % solids, repulped,

Table 3.6.

Results of flotation tests with pyrite recovered from flotation ore cycle tailings of sample 3 with ferrous sulphate

Test Nos.	Product	Yield, %		Content, %				Recovery, %							
		Ore	Operation	Pb	Zn	Cu	S	Pb		Zn		Cu		S	
								ore	operation	ore	operation	ore	operation	ore	operation
1062	Pyrite concentrate	61.6	77.3	0.18	0.15	0.18	50.31	13.4	43.3	4.7	55.9	9.5	63.8	69.6	85.4
	Tailings	18.1	22.7	0.80	0.42	0.35	29.2	17.5	56.7	3.7	44.1	5.4	36.2	11.8	14.6
	Initial product	79.7	100.0	0.32	0.21	0.22	45.52	30.9	100.0	8.4	100.0	14.9	100.0	81.4	100.0

and mixed with ferrous sulphate (7 kg/t of ore) or sulphuric acid (4 kg/t) to reduce the pulp alkalinity. The flotation was run at $pH \sim 7$ with butyl xanthate and a frother. There was produced a pyrite concentrate containing 50.31% sulphur, the recovery of the latter from the ore being 69.6% (as considered from the flotation - it was 85.40%). The data given in Tables 3.6 and 3.7 testify that the pyrite flotation tailings (as compared with the pyrite concentrate) are beneficiated with lead, zinc and copper, and are presented by a finer material, the predominant metals concentration occurring within a 0.01 mm fraction. Losses (from the product) in this fraction were: for lead - 85.3%, for zinc 83.5%, for copper 75.1%. The mineralogical analysis of the pyrite flotation tailings showed, that alongside with pyrite, in the tailings in noticeable amounts are present non-sulphide minerals. The sulphides are presented either as free fine grains (0.006 to 0.002mm and finer), or as emulsion-type dissemination in pyrite (lead, copper, and zinc minerals) and non-sulphide minerals (all sulphides including pyrite).

The difficulty to estimate fine materials under the microscope did not make it possible to evaluate the character of the minerals being lost with a sufficient degree of accuracy (free fine grains, mineral inclusions), but the order of the values given above are illustrative of the fact that their recovery are a problem. Besides, into the pyrite concentrate there have been mainly floated coarse free grains of pyrite and grains containing microinclusions of valuable minerals, the presence of which has been traced in all the size fractions.

We suppose that if it is necessary to reduce non-ferrous metal contents in pyrite concentrates of samples 1 and 2, a refloating of ore flotation tailings will be required.

3.2.5. Final experiments, conducted on the basis of continuous operation.

The experiments with a view to determine probable metallurgical data of the ore samples were conducted according to the complete flowsheet, under conditions of continuous operation, on the basis of the data obtained in the tests with an open circuit. The results obtained are given in Table 3.8; the flowsheets - in Figs. 3.7 - 3.9.

The table shows that lead, copper and zinc concentrates have been produced as a result of the ore samples beneficiation (zinc concentrate was obtained from samples 1 and 2). Lead, copper and zinc recoveries into respective concentrates totalled 30-4[illegible].6% lead (lead content in the concentrate being 40.3--50.8%), 50.4 - 67% copper (copper content in the concentrate being 16-24%), and 55.5 - 69.8 % zinc(Zn content of the concentrate being 50.31-51.88%). The zinc concentrate could not be produced from sample 3, the reason for it being specified earlier (Section 3.2.2). Two zinc products have been produced: a rich one containing 40.3% zinc, with a 49.5% zinc recovery, and a poor one, produced when processing the middlings of the zinc flotation in a separate circuit, this last one containing 14.2% zinc with a 19.5% zinc recovery.

It is necessary to take into consideration, that certain difficulties have been encountered at the final stage of the experiments, because of the ore oxidation which had a negative effect on the final metallurgical results, especially in the case of sample 3, namely: the copper content in the copper concentrate was lower, and the zinc content in the lead concentrate - higher than the values, obtained in the course of conducting the investigations.

The following products (except the zinc products of sample 3) are of interest owing to high metal recoveries into them: the zinc product containing 35. 94% zinc, produced from the middlings of the zinc flotation circuit for ore sample 1 with a zinc recovery 13.2%; the copper flotation tailings; the final products of the lead-copper flotation circuit for ore samples 1 and 3; these products contain 3.9 - 6.5% lead, 5.43 - 6.14% zinc and 2.86 -- 4.31% copper, a cumulative metal recovery into them being 23.9 - 45.3%; the lead-copper middlings and the tailings of the lead flotation circuit of sample 2, a cumulative metal recovery into them being 40.8%.

The final beneficiation products were analysed for silica, for accompanying and precious metals and for detrimental impurities (Tables 3.9 and 3.10).

Content and distribution of metals acco
of pyrite cycle sample 3 fl

Size, mm	Yield, %		Pyrite concentrate								
	ore	product	Content,%				Pb		Zn		Distributi
			Pb	Zn	Cu	S	ore	product	ore	product	ore
+0.074											
-0.074+0.044	4.56	7.4	0.22	0.27	0.34	46.23	1.25	9.3	0.63	13.4	1.32
-0.044+0.030	3.82	6.2	0.17	0.18	0.35	51.12	0.80	6.0	0.35	7.4	1.16
-0.030+0.019	10.10	16.4	0.14	0.175	0.16	49.70	1.71	12.8	0.75	16.0	1.41
-0.019+0.015	12.57	20.4	0.14	0.13	0.13	49.82	2.13	15.9	0.68	14.5	1.36
-0.015+0.010	13.06	21.2	0.17	0.13	0.16	50.07	2.72	20.3	0.86	18.4	1.81
-0.010	17.49	28.4	0.23	0.16	0.16	52.08	4.79	35.7	1.43	30.3	2.44
Initial product	61.60	100.0	0.18	0.15	0.18	50.31	13.40	100.0	4.7	100.0	9.5

SECTION 1

Table 3.7

ibution of metals according to product sizes
pyrite cycle sample 3 flotation

ntrate						Pyrite flotation tailings						
Distribution, %						Yield,%		Content, %				Pb
Zn		Cu		S								
ore	product	ore	product	ore	product	ore	product	Pb	Zn	Cu	S	ore
						0.36	2.0	0.64	0.49	0.59	11.68	0.3
0.63	13.4	1.32	13.9	4.7	6.8	1.17	6.5	0.38	0.22	0.54		0.5
0.35	7.4	1.16	12.2	4.3	6.3	0.62						
0.75	16.0	1.41	14.8	11.2	16.2		3.4	0.24	0.19	0.21	14.60	0.2
0.68	14.5	1.36	14.3	13.9	20.2	1.32	7.3	0.38	0.20	0.18	13.60	0.6
0.86	18.4	1.81	19.1	14.5	21.1	1.95	10,8	0.41	0.22	0.19	14.06	1.0
1.43	30.3	2.44	25.7	20.2	29.4	12.68	70.0	0.97	0.50	0.38	36.00	14.9
4.7	100.0	9.5	100.0	68.8	100.0	18.10	100.0	0.80	0.42	0.35	29.2	17.5

SECTION 2

Table 3.7

ite flotation tailings

Content, %				Distribution, %							
Pb	Zn	Cu	S	Pb		Zn		Cu		S	
				ore	product	ore	product	ore	product	ore	product
0.64	0.49	0.59	11.68	0.3	1.6	0.08	2.3	0.18	3.4	0.4	3.4
0.38	0.22	0.54		0.5	3.1	0 13	3.4	0.54	10.0		
0.24	0.19	0.21	14.60	0.2	1.0	0.06	1.6	0.11	2.0	0.2	1.7
0.38	0.20	0.18	13.60	0.6	3.5	0.13	3.5	0.20	3.7	0.4	3.4
0.41	0.22	0.19	14.06	1.0	5.5	0.21	5.7	0.31	5.8	0.6	5.2
0.97	0.50	0.38	36.00	14.9	85.3	3.09	83.5	4.06	75.1	10.2	86.3
0.80	0.42	0.35	29.2	17.5	100.0	3.70	100.0	5.4	100.0	11.8	100.0

SECTION 3

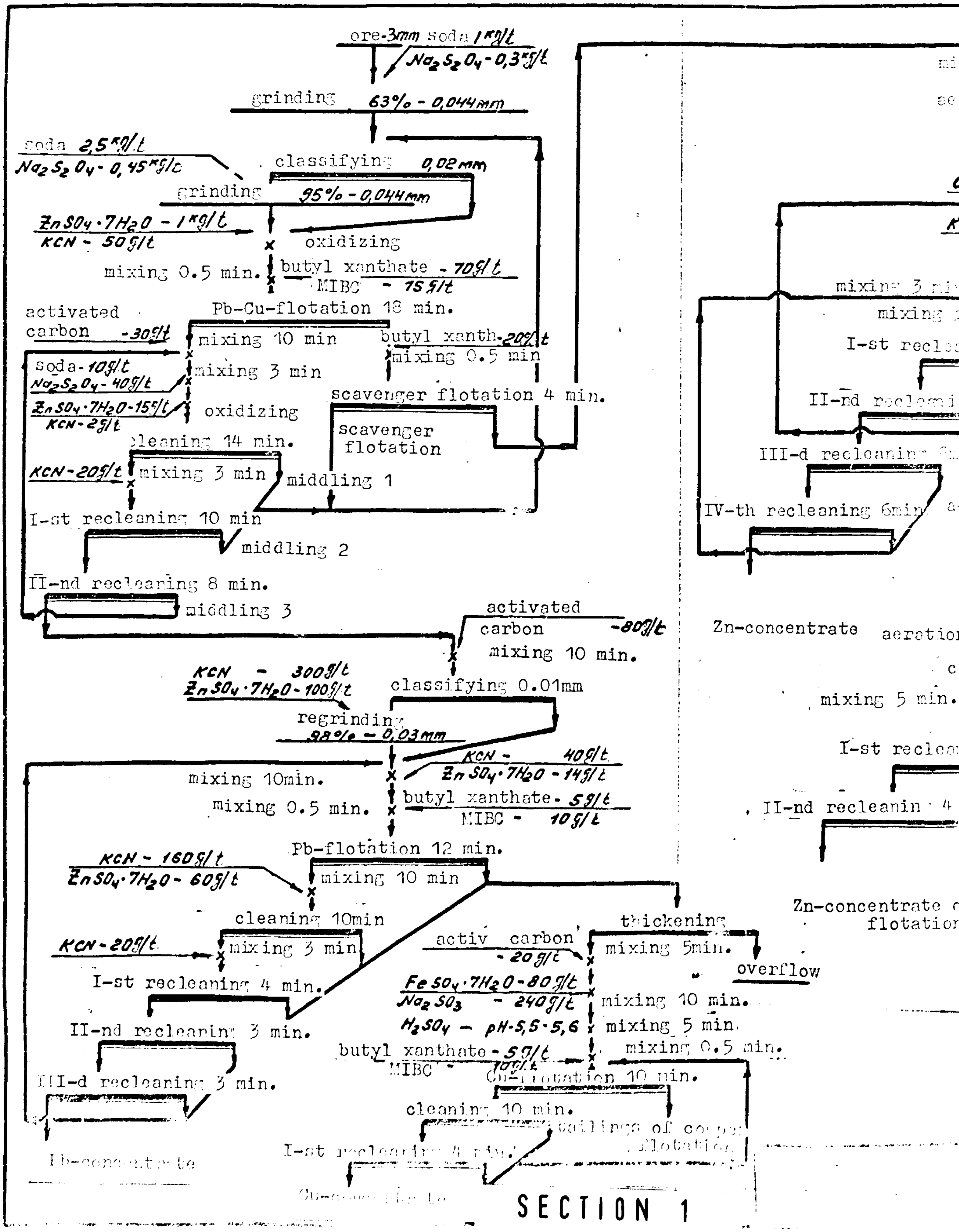
Table 3.8

Results of final tests on ore samples, conducted on ore samples on the basis of continuous operation.

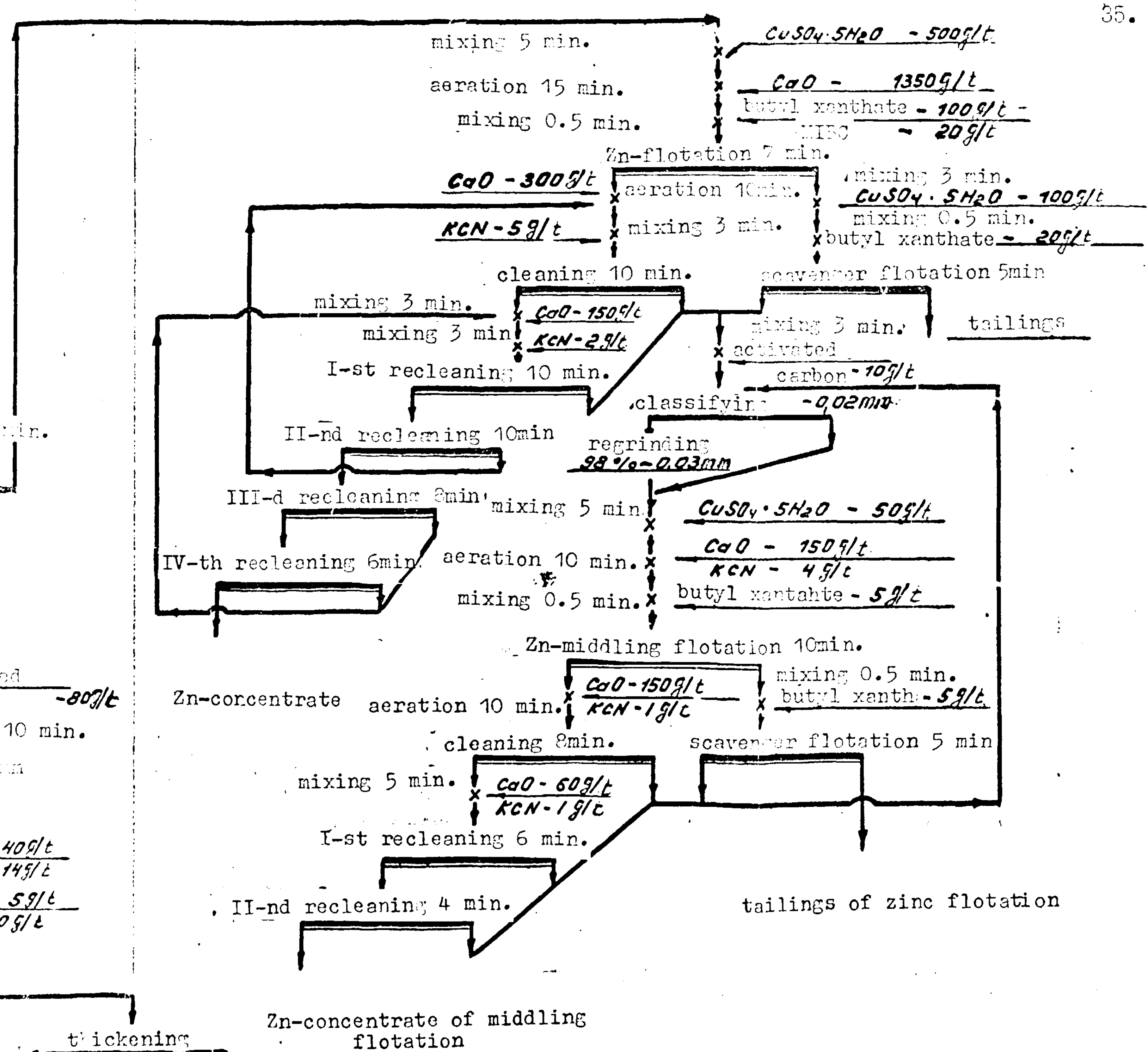
Sample Nos.	Test Nos.	Products	Yield, %		Content, %			Recovery, %						Remarks
			ore	operation	Pb	Zn	Cu	Pb		Zn		Cu		
								ore	operation	ore	operation	ore	operation	
1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
1	98	Pb-concentrate	0.7	9.8	50.8	2.10	3.56	30.0	46.9	0.5	4.8	2.9	3.7	
		Cu-concentrate	2.2	31.0	5.86	2.05	24.00	10.9	17.0	1.7	16.2	61.1	78.4	Fig.3.
		Cu-flotation tailings	4.2	59.2	6.50	5.43	2.86	23.1	36.1	8.3	79.0	13.9	17.9	
		Pb-Cu concentrate	7.1	100.0	10.67	4.05	9.47	64.0	100.0	10.5	100.0	77.9	100.0	
		Zn-concentrate	3.0		0.51	50.31	0.28	1.3		55.5		0.9		
		Middlings flotation Zn-concentrate	1.0		0.75	35.94	2.54	0.7		13.2		2.9		
		Zn-flotation tailings	13.4		0.45	2.25	0.39	5.2		11.0		6.0		
		Tailings	75.5		0.45	0.35	0.14	28.8		9.8		12.3		
		Ore	100.0		1.18	2.72	0.86	100.0		100.0		100.0		

1	2	3	4	5	6
2	559	Pb-concentrate	1.0	26.3	40.30
		Cu-concentrate	0.9	23.7	5.00
		Pb-flotation tailings	1.9	50.0	2.39
		Pb-Cu-concentrate	3.8	100.0	12.98
		Pb-Cu middlings	7.9		1.30
		Zn-concentrate	3.6		0.75
		Zn-flotation tailings	5.5		0.69
		Tailings	79.2		0.36
		Ore	100.0		0.95
3	1064	Pb-concentrate	0.6	8.2	49.31
		Cu-concentrate	4.7	63.5	2.76
		Cu-flotation tailings	2.1	28.3	3.95
		Pb-Cu-concentrate	7.4	100.0	6.87
		Zn-product	2.5		0.38
		Middlings flotation Zn-concentrate	2.8		0.72
		Zn-flotation tailings	7.6		0.50
		Py-concentrate	61.6		0.18
		Tailings	18.1		0.80
		Ore	100.0		0.83

7	8	9	10	11	12	13	14	15
4.00	2.65	42.6	81.6	1.5	21.1	9.3	14.5	
4.30	16.0	4.8	9.2	1.4	19.7	50.4	78.4	Fig. 3.8
6.00	0.70	4.8	9.2	4.2	59.2	4.6	7.1	
5.07	4.84	52.2	100.0	7.1	100.0	64.3	100.0	
3.64	0.20	10.9		10,8		5.5		
51.88	0.26	2.8		69.8		3.3		
1.36	0.16	4.0		2.8		3.1		
0.32	0.086	30.1		9.5		23.8		
2.68	0.29	100.0		100.0		100.0		
3.02	4.28	35.5	58.2	0.9	4.5	2.2	2.9	
5.50	16.71	15.6	25.6	12.7	63.8	67.0	87.1	
6.14	4.31	9.9	16.2	6.3	31.7	7.7	10.0	
5.48	12.18	61.0	100.0	19.9	100.0	76.9	100.0	
40.31	0.64	1.1		49.5		1.4		
14.20	1.74	2.4		19.5		4.1		Fig. 3.9
0.73	0.42	4.6		2.7		2.7		
0.15	0.18	13.4		4.7		9.5		
0.42	0.35	17.5		3.7		5.4		
2.03	1.17	100.0		100.0		100.0		


ore-3mm soda 1kg/t
$Na_2S_2O_4$-0,3kg/t
grinding 63% - 0,044mm
soda 2,5kg/t
$Na_2S_2O_4$ - 0,45kg/t
classifying 0,02mm
grinding 95% - 0,044mm
$ZnSO_4 \cdot 7H_2O$ - 1kg/t
KCN - 50g/t
oxidizing
mixing 0.5 min.
butyl xanthate - 70g/t
MIBC - 15g/t
activated carbon -30g/t
Pb-Cu-flotation 18 min.
mixing 10 min
butyl xanth.-20g/t
mixing 0.5 min
soda-10g/t
$Na_2S_2O_4$-40g/t
mixing 3 min
$ZnSO_4 \cdot 7H_2O$-15g/t
KCN-2g/t
oxidizing
scavenger flotation 4 min.
scavenger flotation
cleaning 14 min.
KCN-20g/t
mixing 3 min
middling 1
I-st recleaning 10 min
middling 2
II-nd recleaning 8 min.
middling 3
activated carbon -80g/t
mixing 10 min.
KCN - 300g/t
$ZnSO_4 \cdot 7H_2O$-100g/t
classifying 0.01mm
regrinding 98% - 0,03mm
KCN - 40g/t
$ZnSO_4 \cdot 7H_2O$ - 14g/t
mixing 10min.
mixing 0.5 min.
butyl xanthate- 5g/t
MIBC - 10g/t
Pb-flotation 12 min.
KCN - 160g/t
$ZnSO_4 \cdot 7H_2O$ - 60g/t
mixing 10 min
cleaning 10min
KCN-20g/t
mixing 3 min
I-st recleaning 4 min.
II-nd recleaning 3 min.
III-d recleaning 3 min.
Pb-concentrate
thickening
activ carbon - 20g/t
mixing 5min.
overflow
$FeSO_4 \cdot 7H_2O$-80g/t
Na_2SO_3 - 240g/t
mixing 10 min.
H_2SO_4 — pH-5,5-5,6
mixing 5 min.
mixing 0.5 min.
butyl xanthate - 5g/t
MIBC - 10g/t
Cu-flotation 10 min.
cleaning 10 min.
tailings of copper flotation
I-st recleaning 4 min.
Cu-concentrate
mixing 3 mi
mixing
I-st reclea
II-nd reclean
III-d recleaning 6m
IV-th recleaning 6min
Zn-concentrate
aeration
mixing 5 min.
I-st reclea
II-nd recleaning 4
Zn-concentrate
flotation

SECTION 1



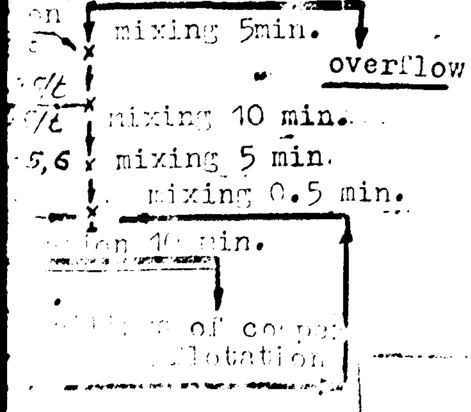
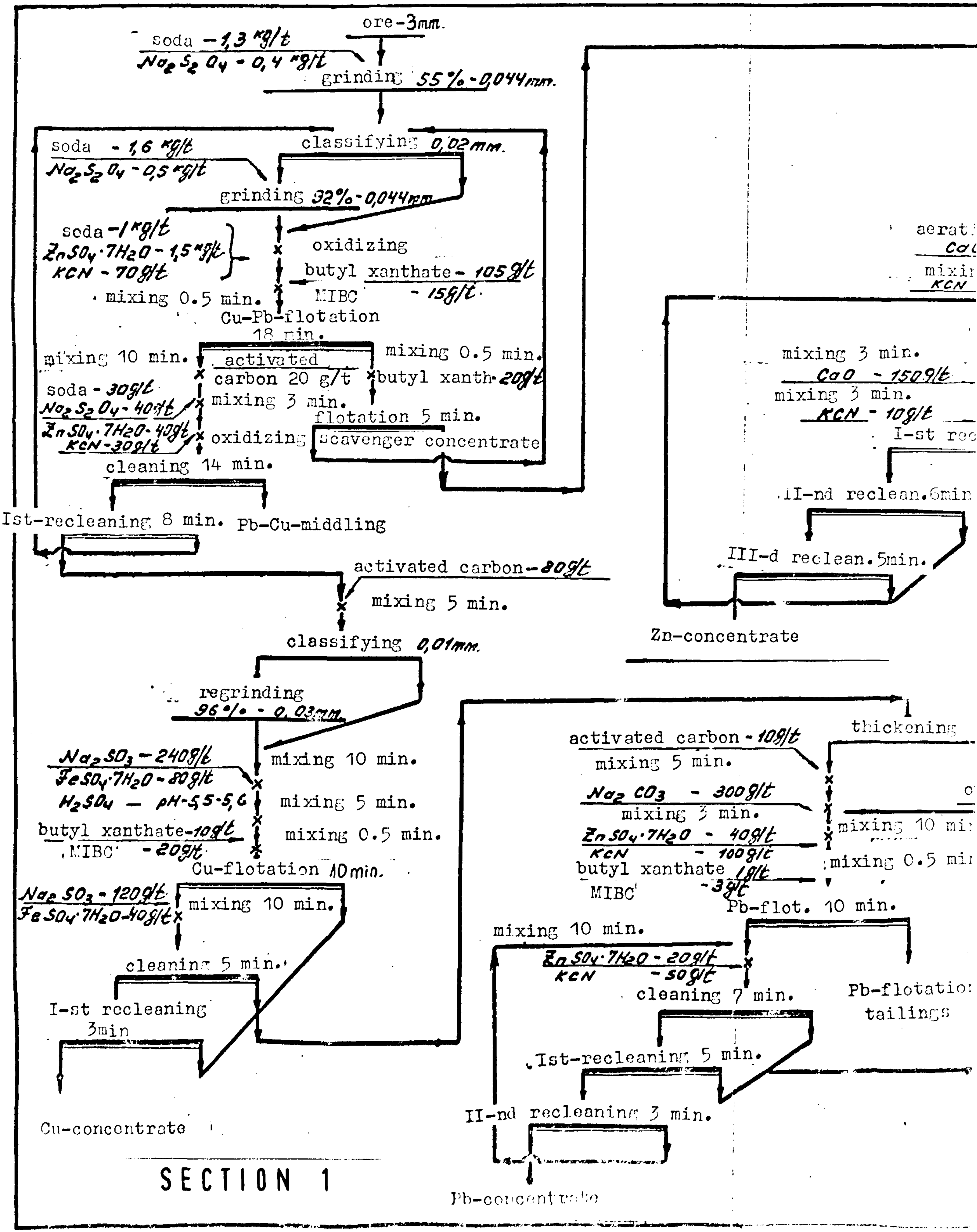


Fig. 3.7. Flowsheet of the final test, conducted on ore sample 1 on the basis of continuous operation (test 98)

SECTION 2


ore-3mm.
soda -1,3 kg/t
$Na_2S_2O_4$ - 0,4 kg/t
grinding 55% -0,044mm.
classifying 0,02mm.
soda - 1,6 kg/t
$Na_2S_2O_4$ - 0,5 kg/t
grinding 92% -0,044mm
soda -1 kg/t
$ZnSO_4 \cdot 7H_2O$ - 1,5 kg/t
KCN - 70g/t
oxidizing
butyl xanthate - 105g/t
MIBC - 15g/t
mixing 0.5 min.
Cu-Pb-flotation 18 min.
mixing 10 min.
activated carbon 20 g/t
mixing 0.5 min.
butyl xanth. 20g/t
soda - 30g/t
$Na_2S_2O_4$ - 40g/t
mixing 3 min.
flotation 5 min.
$ZnSO_4 \cdot 7H_2O$ - 40g/t
KCN - 30g/t
oxidizing
scavenger concentrate
cleaning 14 min.
Ist-recleaning 8 min.
Pb-Cu-middling
activated carbon - 80g/t
mixing 5 min.
classifying 0,01mm.
regrinding 96% - 0,03mm.
Na_2SO_3 - 240g/t
$FeSO_4 \cdot 7H_2O$ - 80g/t
H_2SO_4 — pH-5,5-5,6
mixing 10 min.
mixing 5 min.
butyl xanthate - 10g/t
MIBC - 20g/t
mixing 0.5 min.
Cu-flotation 10min.
Na_2SO_3 - 120g/t
$FeSO_4 \cdot 7H_2O$ - 40g/t
mixing 10 min.
cleaning 5 min.
I-st recleaning 3min
Cu-concentrate
aerat
CaO
mixi
KCN
mixing 3 min.
CaO - 150g/t
mixing 3 min.
KCN - 10g/t
I-st rec
II-nd reclean. 6min
III-d reclean. 5min.
Zn-concentrate
thickening
activated carbon - 10g/t
mixing 5 min.
Na_2CO_3 - 300g/t
mixing 3 min.
$ZnSO_4 \cdot 7H_2O$ - 40g/t
KCN - 100g/t
butyl xanthate 1g/t
MIBC - 3g/t
mixing 10 mi
mixing 0.5 mi
Pb-flot. 10 min.
mixing 10 min.
$ZnSO_4 \cdot 7H_2O$ - 20g/t
KCN - 50g/t
cleaning 7 min.
Pb-flotation tailings
Ist-recleaning 5 min.
II-nd recleaning 3 min.
Pb-concentrate

SECTION 1

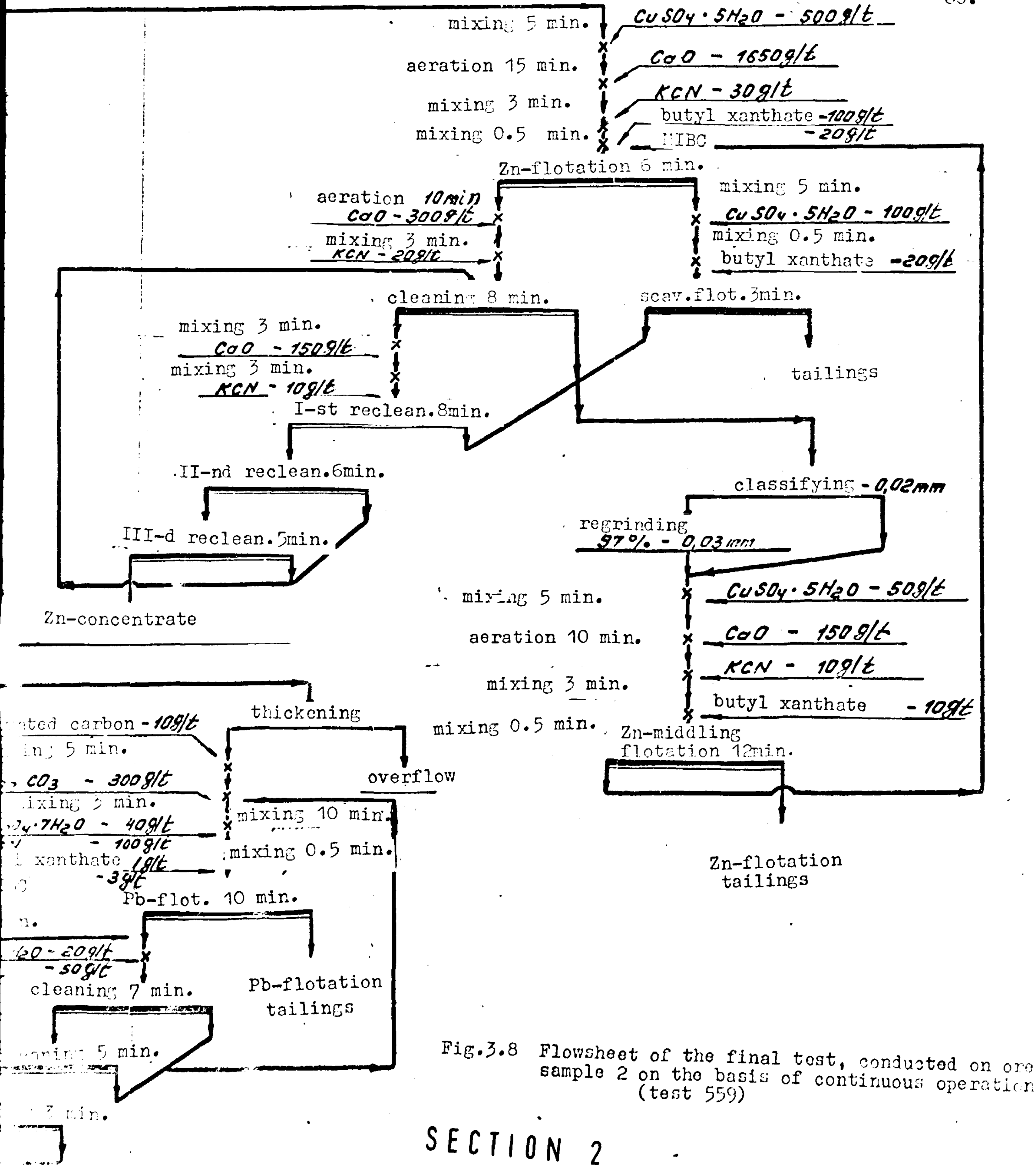
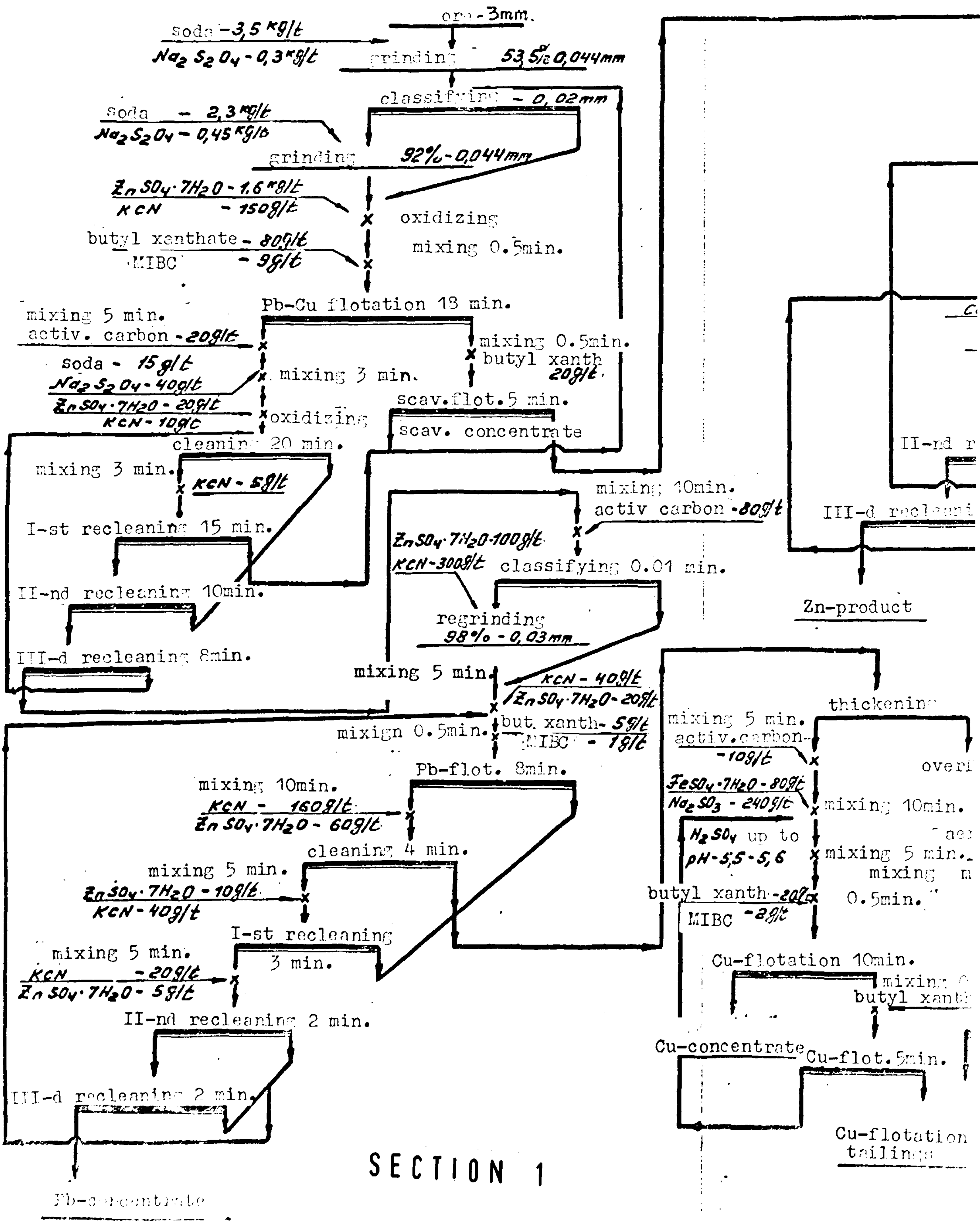


Fig.3.8 Flowsheet of the final test, conducted on ore sample 2 on the basis of continuous operation (test 559)

SECTION 2


ore -3mm.
soda -3,5 kg/t
$Na_2S_2O_4$ - 0,3 kg/t
grinding
53,5% 0,044mm
classifying - 0,02mm
soda - 2,3 kg/t
$Na_2S_2O_4$ - 0,45 kg/t
grinding
92% - 0,044mm
$ZnSO_4 \cdot 7H_2O$ - 1.6 kg/t
KCN - 150g/t
oxidizing
butyl xanthate - 80g/t
MIBC - 9g/t
mixing 0.5min.
Pb-Cu flotation 18 min.
mixing 5 min.
activ. carbon - 20g/t
soda - 15g/t
$Na_2S_2O_4$ - 40g/t
mixing 3 min.
$ZnSO_4 \cdot 7H_2O$ - 20g/t
KCN - 10g/t
oxidizing
mixing 0.5min.
butyl xanth 20g/t
scav.flot. 5 min.
scav. concentrate
cleaning 20 min.
mixing 3 min.
KCN - 5g/t
I-st recleaning 15 min.
II-nd recleaning 10min.
III-d recleaning 8min.
mixing 10min.
activ carbon - 80g/t
$ZnSO_4 \cdot 7H_2O$ - 100g/t
KCN - 300g/t
classifying 0.01 min.
regrinding
98% - 0,03mm
mixing 5 min.
KCN - 40g/t
$ZnSO_4 \cdot 7H_2O$ - 20g/t
but. xanth - 5g/t
MIBC - 1g/t
mixign 0.5min.
Pb-flot. 8min.
mixing 10min.
KCN - 160g/t
$ZnSO_4 \cdot 7H_2O$ - 60g/t
cleaning 4 min.
mixing 5 min.
$ZnSO_4 \cdot 7H_2O$ - 10g/t
KCN - 40g/t
I-st recleaning 3 min.
mixing 5 min.
KCN - 20g/t
$ZnSO_4 \cdot 7H_2O$ - 5g/t
II-nd recleaning 2 min.
III-d recleaning 2 min.
Pb-concentrate
thickening
mixing 5 min.
activ.carbon - 10g/t
overf
$FeSO_4 \cdot 7H_2O$ - 80g/t
Na_2SO_3 - 240g/t
mixing 10min.
H_2SO_4 up to pH - 5,5 - 5,6
mixing 5 min.
mixing
0.5min.
butyl xanth - 20g/t
MIBC - 2g/t
Cu-flotation 10min.
mixing
butyl xanth
Cu-concentrate
Cu-flot. 5min.
Cu-flotation tailings
II-nd r
III-d reclean
Zn-product

SECTION 1

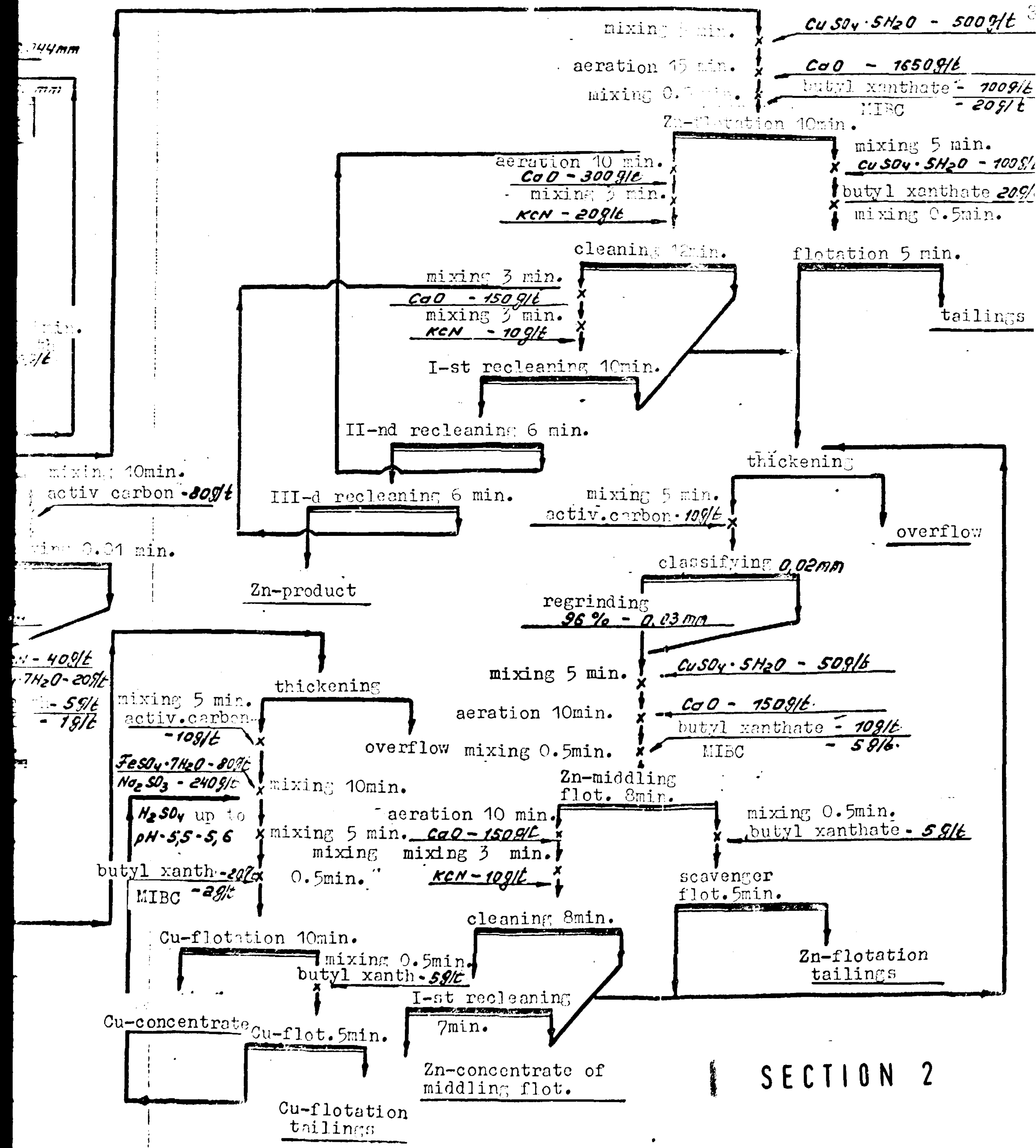


Fig.3.9. Flowsheet of the final test, conducted on o
sample 3 on the basis of continuous operati
(test 10[illegible])

As seen from Tables 3.9 and 3.10 and comparing their data with those given in Table 3.8, it follows, that the tailings of the zinc flotation circuit for all ore samples are similar to those of the ore flotation circuit, as to their sulphur and iron content, and may become a supplementary source of pyrite production. 75-82% of arsenic (as related to its content in the ore) remained in the pyrite-bearing tailings of the ore flotation circuit. The lead concentrates of all the samples contain 1.32-2.76 % antimony; 4% of antimony is contained in the copper concentrate produced from ore sample 2. The copper concentrate contains silver as much as 2370 g/t, its recovery being 49.2% (the copper recovery into the concentrate was 50.4%). Silver is contained in all lead concentrates, in amounts of 700 - 777 g/t. In no products there was gold concentration of practical value, since 75-85% of gold remained in the tailings of the ore flotation circuit. It is possible to assume that in this case gold is associated with pyrite. With mercury content in the ore being 0.010 - 0.011 %, its amounts in the zinc concentrates reached 0.066-0.120%.

To evaluate the results from the standpoint of the ore mineral and chemical composition we have studied the composition of some final beneficiation products, by making use of dispersion, mineralogical, x-ray diffraction, phase-chemical and chemical analyses.

The data obtained make it possible to conclude:

- Most metal losses in the tailings of the ore flotation circuit are concentrated in a 0.010 mm fraction and make up 12.6--20% lead, 6.3 - 10.9% zinc, and 6.0-11.5% copper (as related to their content in the ore). Though this fraction was not detected with a microscope, one can still assume, that it is the size of minerals in this fraction, that creates difficulties for the flotation process and desintegration of intergrowths, that may occur in this fraction. The fact, that the values of metal content in all the other fractions are close to each other testifies about the presence of fine dissemination of the sulphides; this assumption was corroborated by a mineralogical analysis which showed that lead, copper and zinc mineral inclusions (integrowths) are prevailing in the pyrite, the size of inclusions and free grains being 0.004 - 0.008 mm. A characteristic feature, especially for the tailings of ore sample [illegible], is the presence of sphalerite (impregnated with finest inclusions of pyrite) and inclusions of sulphides, mostly pyrite, in the non-sulphide minerals. The observed individual inclusions of anglesite and smithsonite in the pyrite having the size of 0.004-0.005 mm contain, in their turn, microinclusions of pyrite. The results of the phase chemical analysis confirm, in their turn, the data obtained by the mineralogical analysis relative to the forms of the metals lost in the tailings and show a close connection existing between the lead minerals and the pyrite, this being evidenced by the fact that the tailings of all the samples show the presence of 3.1 % to 7.7% lead associated with pyrite. 9.7% to 15% of copper (from the ore copper content) is lost in the form of gray ore in the tailings of the ore flotation circuit; the final products of the lead-copper concentrate separation are 80-85% pyrite occurring, normally, as free grains, while the lead and copper minerals in more or less equal amounts occur as 0.001--0.002 mm inclusions in the pyrite and as free grains whose size doesn't exceed 0.004 mm; the same is the size of free sphalerite grains; pyrite is a prevailing mineral in the tailings of the zinc flotation circuit (90-92% of the product), it occurs mostly as free grains, while lead minerals (galena and, rarely, anglesite), copper minerals (chalcopyrite and gray ore) and smithsonite are encountered exclusively in the form of inclusions and microinclusions in the pyrite; free grains of sphalerite in the product prevail over its intergrowths. It is at the expense of the sphalerite that the zinc is lost in this product; more than a half of copper and zinc minerals in the lead concentrate occur as intergrowths with pyrite, rarely, with galena and sphalerite. Free grains of minerals are often overground and have a size of 0.003-0.006 mm. A high content of antimony in the lead concentrate of all the samples is caused by the presence of 10% to 15% of bournonite containing about 25% of antimony; a characteristic feature of the copper concentrates is the presence of sphalerite occurring as free grains, often overground, the size of intergrowths of sphalerite with pyrite encountered is 0.006-0.008 mm, while the lead minerals in the product prevail as intergrowths with other minerals, the size of their inclusions not exceeding 0.004-0.006 mm.

Contents and recovery of metall impurities
into beneficiation products

le os.	Products	Yield, %	Sulphur	Iron	Arsenic	Cadmium	Bismuth	Antimony	Cobalt	Mercury	Si ci ox
			Contents, %								
	Lead concentrate	0.7	21.34	15.19	0.44	0.0075	0.33	1.32	0.009	0.0155xx/	
	Copper concentrate	2.2	32.26	30.49	0.40	0.004	0.047	0.068	0.011	0.0155	
	Copper flotation tailings	4.2	46.44	38.64	0.71	0.014	0.035	0.065	0.031		
	Lead-copper concentrate	7.1	39.56	33.80	0.59	0.010	0.067	0.188	0.0226		
	Zinc concentrate	3.0	33.00	13.29	0.40	0.009	0.0071	0.037	0.0083	0.066	
	Zinc concentrate of middlings flotation	1.0	34.85	27.36	0.48	0.072	0.018	0.035	0.016		
	Zinc flotation tailings	13.4	49.00	41.50	1.02	0.005	0.058	0.055	0.044		
	Tailings	75.5	50.00	42.52	0.80	0.002	0.018	0.046	0.038		
	Ore	100.0	48.46	40.73	0.80	0.0066	0.027	0.057	0.0366	0.11	
	Lead concentrate	1.0	26.89	17.15	0.52	0.011	0.044	1.48	0.005	0.027	0.1
	Copper concentrate	0.9	36.77	30.72	0.74	0.016	0.021	4.0	0.0055	0.059	0.1
	Lead flotation tailings	1.9	44.60	39.00	0.68	0.012	0.030	0.040	0.079		1.8
	Lead-copper concentrate	3.8	38.08	31.29	0.64	0.012	0.031	1.3568	0.042		0.9
	Lead-copper middlings	7.9	47.34	42.44	0.61	0.009	0.0074	0.085	0.011		1.0
	Zinc concentrate	3.6	32.60	13.40	0.40	0.125	0.0026	0.031	0.005	0.120	0.2
	Zinc flotation tailings	5.5	49.00	41.50	0.73	0.002	0.0065	0.030	0.012		3.0
	Tailings	79.2	47.86	42.00	0.75	0.004	0.0055	0.055	0.011		2.0
	Ore	100.0	47.46	40.57	0.72	0.009	0.0066	0.107	0.012	0.010	2.3
[illegible]	Lead concentrate	0.6	22.43	11.90	0.28	0.006	0.022	2.75	0.0055	0.0155	0.1
	Copper concentrate	4.7	35.19	30.72	0.34	0.011	0.014	0.12	0.021	0.008	0.3
	Copper flotation tailings	2.1	45.66	40.95	0.71	0.003	0.017	0.045	0.068		1.8
	Lead-copper concentrate	7.4	37.13	32.10	0.44	0.0097	0.085	0.313	0.033		0.
	Zinc product	2.5	35.09	28.84	0.52	0.071	0.016	0.018	0.020	0.076	0.
	Zinc concentrate of middlings flotation	2.8	40.85	33.28	0.92	0.028	0.011	0.026	0.041		2.0
	Zinc flotation tailings	7.6	45.41	40.96	0.83	0.004	0.013	0.059	0.058		0.5
	Pyrite concentrate	61.6	50.31	44.23	0.63	traces	0.01	0,033	0.040	0.043	3.9
	Tailings	18.1	29.20	25.19	1.16	0.0176	0.031	0,086	0.045		31.5
	Ore	100.0	44.5	38.97	0.73	0.0058	0.019	0.065	0.041	0.010	3.2

SECTION 1

Note: x/ The beneficiation products were not analysed for

overy of metall impurities
products

Table 3.9

						Recovery, %							
	Bismuth	Antimony	Cobalt	Mercury	Silicium oxide	Sulphur	Iron	Arsenic	Cadmium	Bismuth	Antimony	Cobalt	Silicium oxide
5	0.33	1.32	0.009	0.0155 xx/	x/	0.3	0.3	0.4	0.8	8.7	16.2	0.2	
	0.047	0.068	0.011	0.0155		1.5	1.6	1.1	1.3	3.8	2.6	0.7	
	0.035	0.065	0.031			4.0	4.0	3.7	8.9	5.5	4.8	3.5	
	0.067	0.188	0.0226			5.8	5.9	5.2	11.0	18.0	23.6	4.4	
	0.0271	0.037	0.0083	0.066		2.0	1.0	1.5	45.0	0.8	1.9	0.7	
2	.013	0.035	0.016			0.7	0.7	0.6	10.9	0.6	0.6	0.4	
5	.093	0.055	0.044			13.6	13.6	17.1	10.2	29.3	13.0	16.1	
2	.018	0.046	0.038			77.9	78.8	75.6	22.9	51.3	60.9	78.4	
36	.027	0.057	0.0366	0.11		100.0	100.0	100.0	100.0	100.0	100.0	100.0	
1	.044	1.48	0.005	0.027	0.15	0.6	0.4	0.7	1.2	6.7	13.8	0.4	0.06
	.021	4.0	0.0055	0.059	0.15	0.7	0.7	0.9	1.6	2.9	33.6	0.4	0.06
	.030	0.040	0.079		1.80	1.8	1.8	1.8	2.6	8.6	0.7	12.5	1.44
	.031	1.3568	0.042		0.97	3.1	2.9	3.4	5.4	18.2	48.1	13.3	1.56
	.0074	0.085	0.011		1.00	7.9	8.3	6.7	7.9	8.9	6.3	7.2	3.32
	.0026	0.031	0.005	0.120	0.20	2.4	1.2	2.0	50.2	1.4	1.0	1.5	0.30
	.0065	0.080	0.012		3.0	5.7	5.6	5.5	1.2	5.5	4.1	5.5	6.94
	.0055	0.055	0.011		2.64	80.9	82.0	82.4	35.3	66.0	40.5	72.5	87.88
	.0066	0.107	0.012	0.010	2.38	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.00
	.022	2.75	0.0055	0.0155	0.18	0.3	0.3	0.2	0.5	0.7	25.6	0.1	0.01
	.014	0.12	0.021	0.008	0.30	3.7	3.7	2.2	7.6	3.3	8.6	2.4	0.17
	.017	0.045	0.068		1.60	2.2	2.2	2.0	2.5	27.6	1.5	3.5	0.41
	.035	0.313	0.033		0.66	6.2	6.2	4.4	10.6	31.6	35.7	6.0	0.59
	.016	0.018	0.020	0.076	0.88	2.0	1.8	1.8	26.2	2.0	0.7	1.2	0.126
	.011	0.026	0.041		2.0	2.6	2.4	3.5	11.6	1.5	1.1	2.8	0.67
	.013	0.059	0.058		0.50	7.8	8.0	8.6	4.5	4.9	7.0	10.7	0.46
	.01	0,033	0.040	0.048	3.93	69.6	70.0	53.0	-	31.5	31,5	59.6	29.10
	.031	0,086	0.045		31.57	11.8	11.6	28.7	47.1	28.5	24,0	19.7	68.92
	.019	0.065	0.041	0.010	3.29	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0

products were not analysed for

SECTION 2

	4.2	48.44	38.84	0.71	0.014	0.035	0.065	0.031			4.0	4.	3.
	7.1	39.56	33.80	0.59	0.010	0.057	0.188	0.0226			5.8	5.	5.
	3.0	33.00	13.29	0.40	0.009	0.0071	0.037	0.0083	0.066		2.0	1.0	1.5
of tion	1.0	34.35	27.36	0.48	0.072	0.018	0.035	0.016			0.7	0.7	0.8
	13.4	49.00	41.50	1.02	0.005	0.058	0.055	0.044			13.6	13.6	13.1
	75.5	50.00	42.52	0.80	0.002	0.018	0.046	0.038			77.9	78.8	75.0
	100.0	48.46	40.73	0.80	0.0066	0.027	0.057	0.0366	0.11		100.0	100.0	100.0
	1.0	26.89	17.15	0.52	0.011	0.044	1.48	0.005	0.027	0.15	0.6	0.4	0.7
te	0.9	36.77	30.72	0.74	0.016	0.021	4.0	0.0055	0.059	0.15	0.7	0.7	0.9
	1.9	44.60	39.00	0.68	0.012	0.030	0.040	0.079		1.80	1.8	1.8	1.9
	3.8	38.08	31.29	0.64	0.012	0.031	1.3568	0.042		0.97	3.1	2.9	3.4
	7.9	47.34	42.44	0.61	0.009	0.0074	0.085	0.011		1.00	7.9	8.3	6.7
	3.6	32.60	13.40	0.40	0.125	0.0026	0.031	0.005	0.120	0.20	2.4	1.2	2.0
	5.5	49.00	41.50	0.73	0.002	0.0065	0.080	0.012		3.0	5.7	5.6	5.5
	79.2	47.86	42.00	0.75	0.004	0.0055	0.055	0.011		2.64	80.9	82.0	82.4
	100.0	47.46	40.57	0.72	0.009	0.0066	0.107	0.012	0.010	2.38	100.0	100.0	100.0
	0.6	22.43	11.90	0.28	0.006	0.022	2.75	0.0055	0.0155	0.18	0.3	0.3	0.2
te	4.7	35.19	30.72	0.34	0.011	0.014	0.12	0.021	0.008	0.30	3.7	3.7	2.2
	2.1	45.66	40.95	0.71	0.008	0.017	0.045	0.068		1.60	2.2	2.2	3.0
	7.4	37.13	32.10	0.44	0.0097	0.035	0.313	0.033		0.66	6.2	6.2	4.4
	2.5	35.09	28.84	0.52	0.071	0.016	0.018	0.020	0.076	0.88	2.0	1.8	1.3
of tion	2.8	40.85	33.28	0.92	0.028	0.011	0.026	0.041		2.0	2.6	2.4	3.5
	7.6	45.41	40.96	0.83	0.004	0.013	0.059	0.058		0.50	7.8	8.0	3.6
te	61.6	50.31	44.25	0.63	traces	0.01	0,033	0.040	0.048	3.93	69.6	70.0	55.0
	18.1	29.20	25.19	1.16	0.0176	0.031	0,086	0.045		31.57	11.8	11.6	28.7
	100.0	44.5	38.97	0.73	0.0068	0.019	0.065	0.041	0.010	8.29	100.0	100.	100.0

Note: x/ The beneficiation products were not analysed for silicium oxide content because of a low silicium oxide content in the ore sample 1 (o,65 %).

xx/ Only ore and concentrates were analysed for mercury content.

SECTION 3

Table 3.10

Gold and silver content and recovery in beneficiation products

Sample No.	Products	Yield, %	Content, g/t		Recovery, %	
			gold	silver	gold	silver
1	Lead concentrate	0.7	1.9	777.0	1.5	19.9
	Copper concentrate	2.2	0.43	183.0	1.1	14.7
	Copper flotation tailings	4.2	1.0	33.8	4.8	5.2
	Zinc concentrate	3.0	0.34	58.0	1.2	6.4
	Zinc concentrate of middlings flotation	1.0	1.0	52.0	1.2	1.9
	Zinc flotation tailings	13.4	0.95	18.8	14.6	9.2
	Tailings	75.5	0.87	15.4	75.6	42.7
	Ore	100.0	0.87	27.3	100.0	100.0
2	Lead concentrate	1.0	0.6	729.0	0.7	16.8
	Copper concentrate	0.9	0.6	2370.0	0.6	49.2
	Lead flotation tailings	3.8	0.77	13.0	1.8	0.7
	Lead-copper middlings	7.9	0.85	15.0	8.1	2.7
	Zinc concentrate	3.6	0.30	41.0	1.3	3.4
	Zinc flotation tailings	5.5	0.85	14.3	5.7	1.8
	Tailings	79.2	0.85	13.8	81.8	25.4
	Ore	100.0	0.82	43.24	100.0	100.0
	Lead concentrate	0.6	0.5	702.0	0.7	20.1
	Copper concentrate	4.7	0.33	131.0	3.5	29.4
	Copper flotation tailings	2.1	0.52	10.1	2.4	1.0
	Zinc product	2.5	0.29	50.0	1.6	6.0
	Zinc concentrate pf middlings flotation	2.6	0.41	38.0	3.3	5.1
	Zinc flotation tailings	7.6	0.25	12.5	4.2	4.5
	Tailings	79.7	0.48	8.9	84.3	33.9
	Ore	100.0	0.45	20.94	100.0	100.0

Tetrahedrite is a major copper mineral in the copper concentrate of sample 2, the content of antimony in this mineral reaching 29%; high concentration of silver in this product can be explained by the presence of this mineral in the amount of 2370 g/t; the lead and copper concentrates contain pyrite mostly as free grains, except when it contains inclusions of other sulphides. In the zinc concentrates of samples 1 and 2 only a half of the pyrite is represented by grains, free from intergrowths, the rest of the pyrite - by intergrowths with sphalerite of 0.012 mm size, sometimes it forms microinclusions in sphalerite. The zinc products of sample 3 have been considered above.

Summarizing the data obtained in investigating the mineral and chemical composition of the beneficiation products, it follows that the process of regrinding the beneficiation products up to 90-95% - 0.03 mm provides, in the first instance, for pyrite liberation; high metal losses in the tailings of the ore flotation circuit and in the tailings of the other corresponding flotation circuits are caused mainly by unfeasibility of liberation of such fine mineral inclusions in the pyrite by grinding means available, yet preventing the sulphides from sliming.

The flotation flowsheets applied did not allow to recover the slimed sulphides. All this accounts for high mutual metal losses in different concentrates.

3.3. Bulk flotation flowsheet investigations

In accordance with the program of the testwork, bulk flotation flowsheet investigations on ore samples were carried out.

When beneficiating ores with such a high pyrite content and a fine dissemination of valuable minerals as is our case according to a flowsheet providing for lead, copper and zinc minerals flotation to obtain a bulk concentrate, it is unlikely to hope for high **results** as to a recovery of metals and a grade of concentrates. Only the question of partial depression of pyrite can be considered.

On the basis of the mineral and chemical composition data and the results of the tests, ore samples were ground, before the bulk flotation, up to the size, chosen for the lead-copper flotation (Section 3.2.1).

Pyrite depression in the rougher flotation cycle was carried out by adding lime during the aeration procedure. To prevent a depressing action of calcium ions on the surface of galena, xanthate and a frother were added in the grinding cycle, but even in this case, it was possible to carry out bulk flotation with pH not higher, than 8.0 - 8.5, as with rising pH galena depression was observed. As at the accepted pH value zinc was not sufficiently recovered into a bulk concentrate (only 35-58%), to increase the floatability of sphalerite, copper sulphate was added to the bulk flotation tailings, and the sphalerite was subjected to reflotation. Amyl xanthate was used as a collector in the ore flotation circuit.

The results of the bulk flotation investigations, carried out in two directions, are given below.

The first one provides for obtaining and subsequent flotation separation of the bulk lead-copper-zinc concentrate into corresponding selective concentrates; the other one provides for obtaining only a bulk concentrate with the highest possible content of lead, zinc and copper for conducting research investigations in order to find a hydrometallurgical method of their separation into monometallic products. The flowsheet and conditions for obtaining the rough lead-copper-zinc concentrate are the same for both directions.

The conditions for the lead-copper-zinc concentrate separation, as well as those for the lead-copper concentrate separation, were defined on the basis of the ivestigations conducted according to the flowsheet with the use of the bulk-selective flotation. In figures 3.10-3.12 are given the flowsheets and conditions of the tests, based on a continuous process principle, following the first direction; the beneficiation results for every ore sample are listed in Table 3.11. According to the data, of all the ore samples there have been produced: lead concentrates with a low lead content (33.3 - 41.8 %) and a 29.2-37.2% recovery into the concentrate; copper concentrates with 15.5 - 22.05% copper

Table 3.11

Results of tests carried out under continuous process conditions on ore samples according to a flowsheet of collective flotation followed by a bulk concentrate selection

Sample Nos.	Product	Yield, %	Content, %			Recovery, %			Remarks
			Lead	Zinc	Copper	Lead	Zinc	Copper	
1	2	3	4	5	6	7	8	9	10
1	Lead concentrate	1.0	41.80	3.20	2.00	36.2	1.2	2.4	
	Copper concentrate	3.8	3.81	4.05	15.50	12.5	5.8	68.6	
	Copper flotation tailings	9.5	1.13	4.20	0.71	9.3	15.0	7.8	Fig.3.10
	Lead-copper concentrate	14.3	5.43	4.09	5.20	58.0	22.0	78.8	
	Zinc concentrate	3.4	0.74	45.38	0.24	2.2	58.0	0.9	
	Tailings of zinc flotation 1	18.6	0.78	1.05	0.26	12.5	7.3	5.6	
	Tailings of zinc flotation 2	6.4	1.05	2.31	0.32	5.8	5.5	2.4	
	Tailings of zinc flotation 3	1.2	2.08	4.50	0.50	2.2	2.0	0.7	
	Total zinc flotation tailings	26.2	0.90	1.51	0.28	20.4	14.8	8.7	
	Tailings	56.1	0.40	0.25	0.18	19.4	5.2	11.6	
	Ore	100.0	1.16	2.66	0.86	100.0	100.0	100.0	

1	2	3	4	5
2	Lead concentrate	0.9	36.84	4.04
	Copper concentrate	0.6	8.75	7.10
	Zinc concentrate	3.4	2.00	49.00
	Tailings of zinc flotation 1	16.2	0.81	1.64
	Tailings of zinc flotation 2	5.3	1.25	2.25
	Tailings of zinc flotation 3	14.0	0.35	1.35
	Total zinc flotation tailings	35.5	0.60	1.63
	Tailings	59.6	0.39	0.30
	Ore	100.0	0.89	2.49
3	Lead concentrate	0.7	33.30	4.15
	Copper concentrate	3.2	3.20	2.29
	Copper flotation tailings	8.9	1.41	5.05
	Lead-copper concentrate	12.8	3.60	4.31
	Zinc concentrate	2.3	0.56	40.31
	Tailings of zinc flotation 1	20.1	0.56	0.49
	Tailings of zinc flotation 2	7.2	0.53	1.32
	Total zinc flotation tailings	27.3	0.56	0.71
	Tailings	57.6	0.30	0.29
	Ore	100.0	0.80	1.84

6	7	8	9	10
3.44	37.2	1.5	11.5	
17.17	5.9	1.7	38.0	
0.28	7.6	66.9	3.6	Fig.3.11
0.17	11.1	10.7	10.3	
0.28	7.4	4.8	5.5	
0.16	5.5	7.6	8.2	
0.18	24.0	23.1	24.0	
0.11	25.3	6.8	22.9	
0.27	100.0	100.0	100.0	
5.85	29.2	1.6	3.4	
22.05	12.8	4.0	58.7	Fig.3.12
2.45	15.8	24.4	18.1	
7.54	57.8	30.0	80.2	
1.46	1.6	50.4	2.8	
0.41	14.1	5.3	6.9	
0.42	4.8	5.2	2.5	
0.41	18.9	10.5	9.4	
0.16	21.7	9.1	7.6	
1.20	100.0	100.0	100.0	

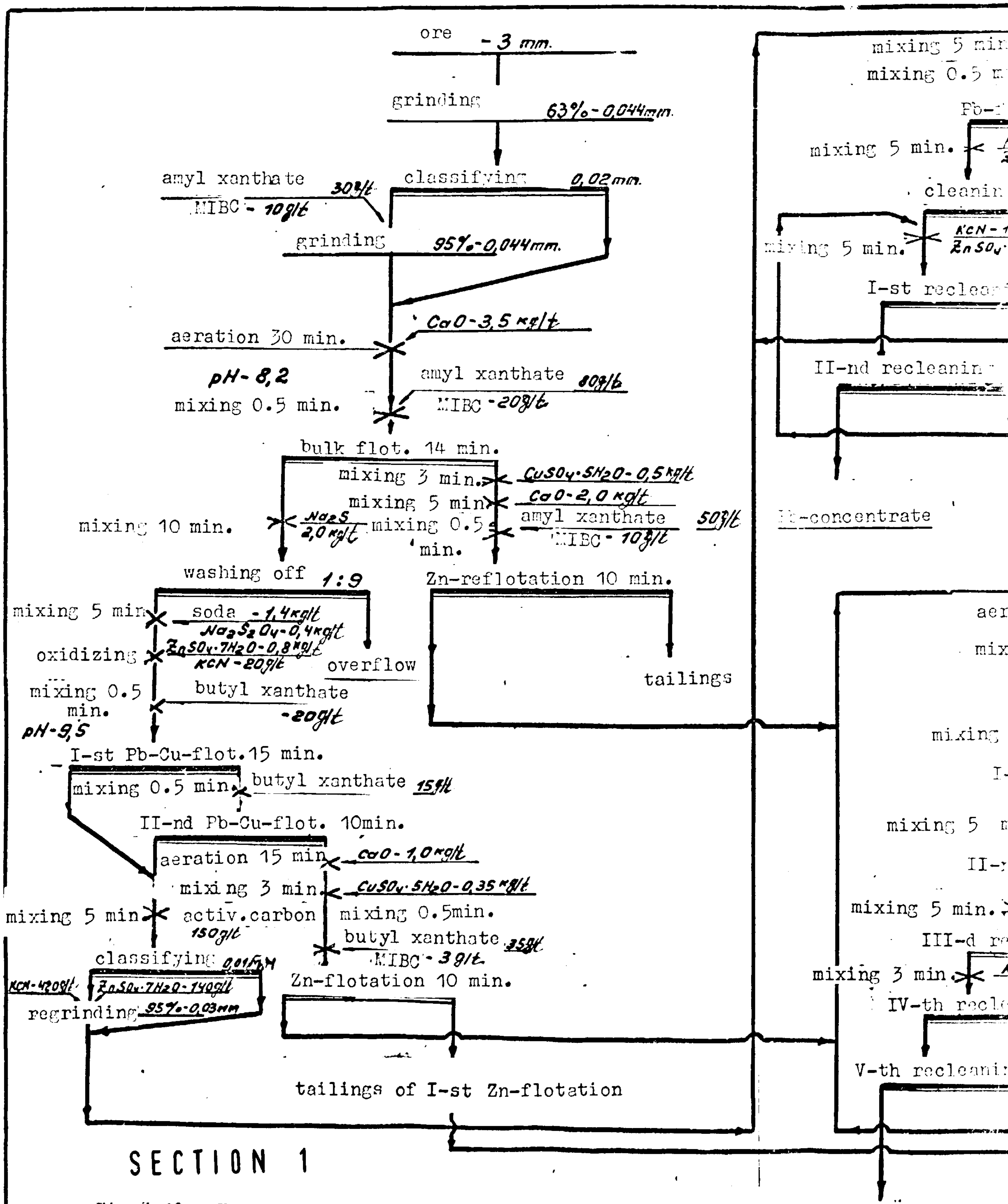
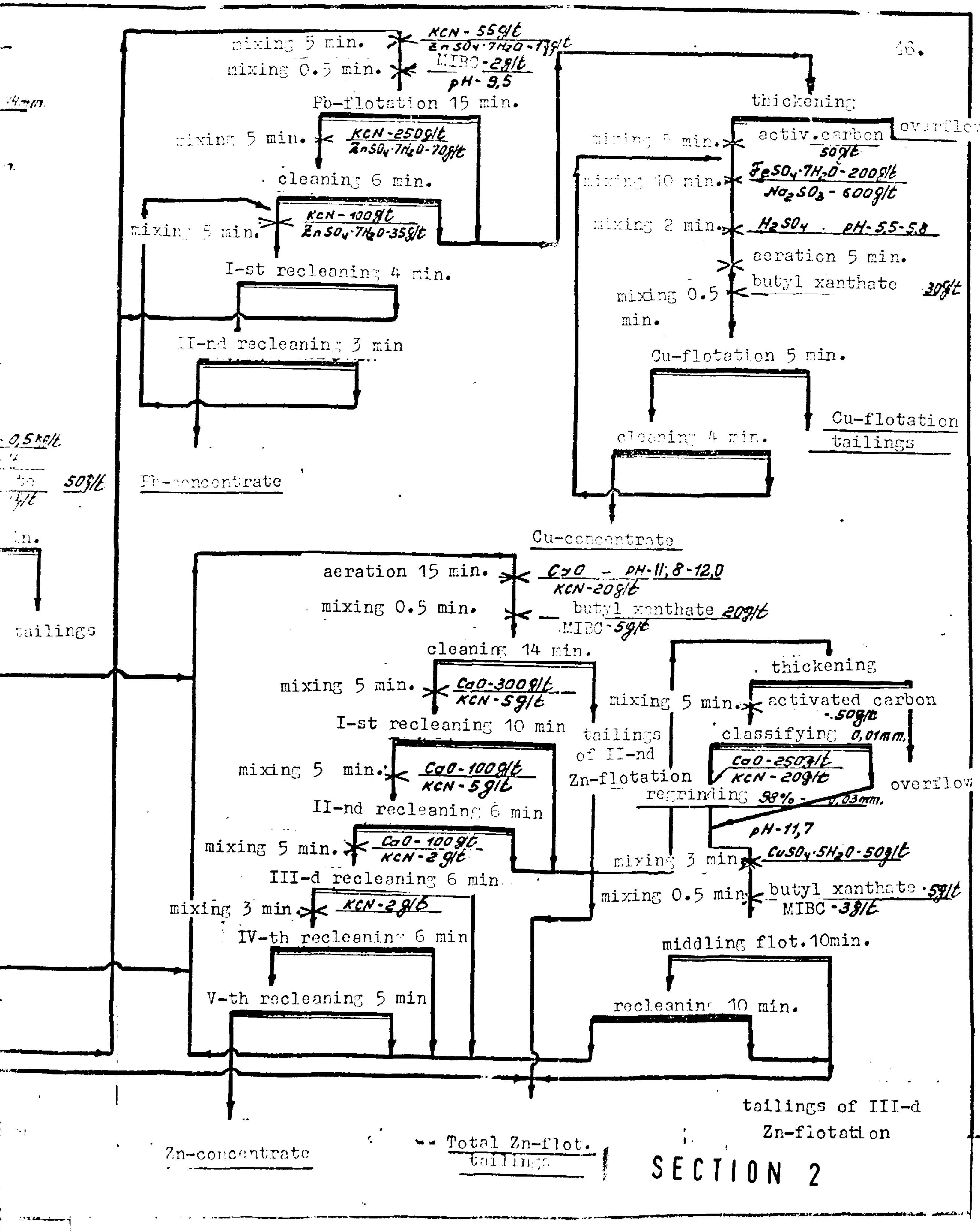
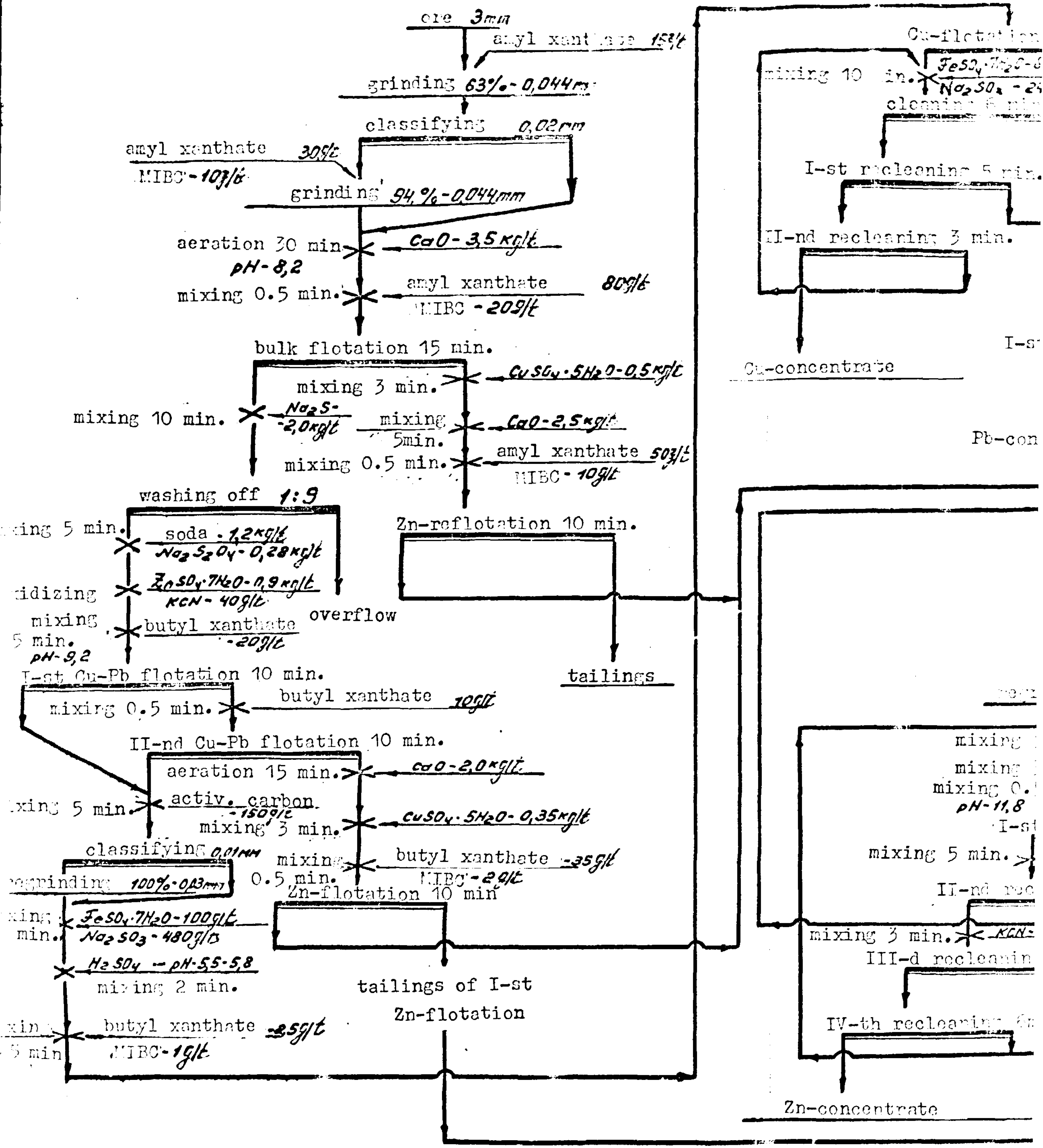


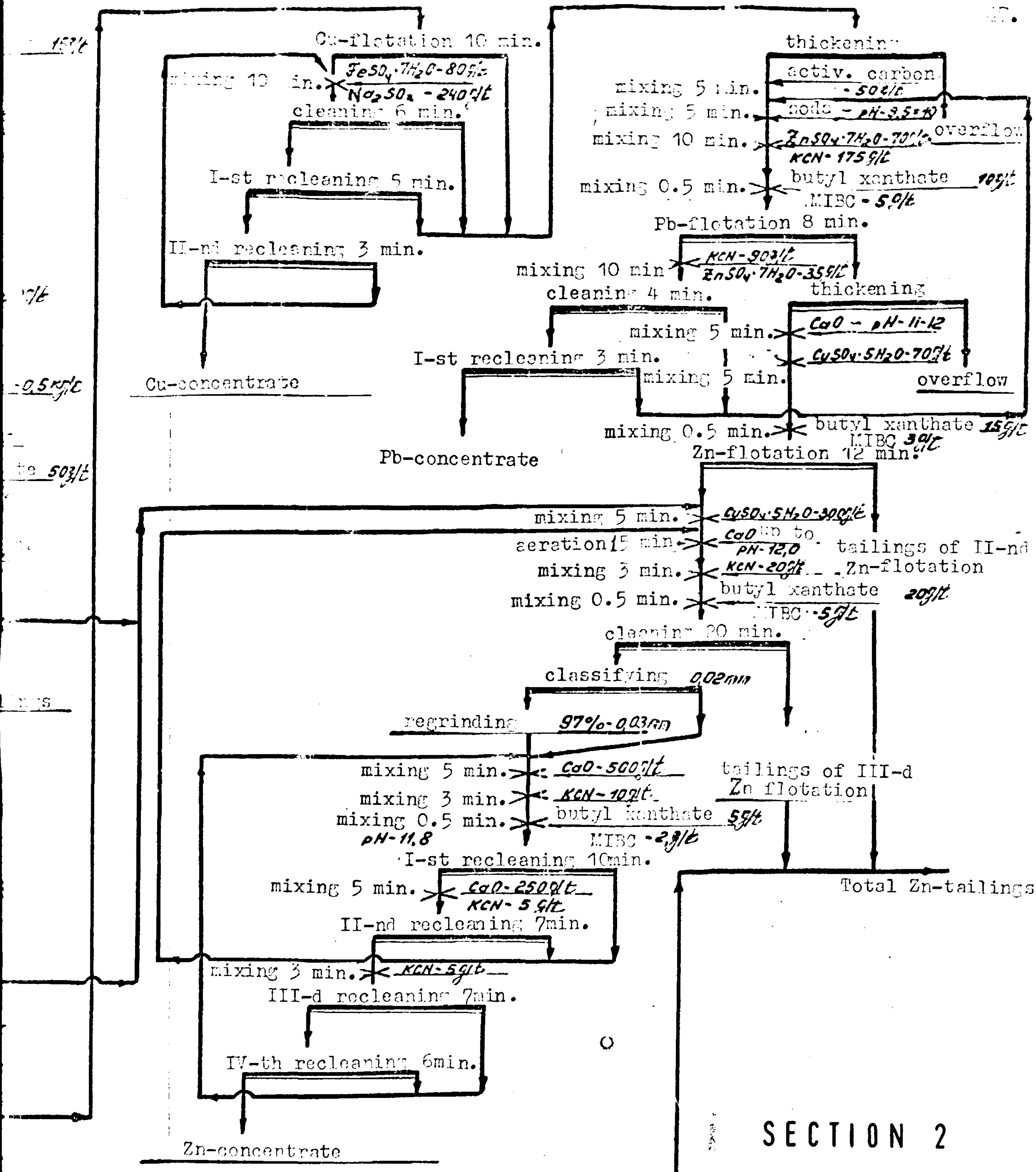
Fig.3.10. Flowsheet of test 1519, conducted on ore sample 1 on the basis of continuous operation


mixing 5 min.
KCN - 55g/t
ZnSO$_4$·7H$_2$O - 17g/t
mixing 0.5 min.
MIBC - 2g/t
pH - 9,5
Pb-flotation 15 min.
mixing 5 min.
KCN-250g/t
ZnSO$_4$·7H$_2$O-70g/t
cleaning 6 min.
mixing 5 min.
KCN - 100g/t
ZnSO$_4$·7H$_2$O-35g/t
I-st recleaning 4 min.
II-nd recleaning 3 min
Pb-concentrate
thickening
overflow
mixing 5 min.
activ.carbon 50g/t
mixing 10 min.
FeSO$_4$·7H$_2$O-200g/t
Na$_2$SO$_3$ - 600g/t
mixing 2 min.
H$_2$SO$_4$. pH-5.5-5.8
aeration 5 min.
butyl xanthate 30g/t
mixing 0.5 min.
Cu-flotation 5 min.
Cu-flotation tailings
cleaning 4 min.
Cu-concentrate
tailings
aeration 15 min.
CaO - pH-11,8-12,0
KCN-20g/t
mixing 0.5 min.
butyl xanthate 20g/t
MIBC - 5g/t
cleaning 14 min.
mixing 5 min.
CaO-300g/t
KCN-5g/t
I-st recleaning 10 min
mixing 5 min.
CaO-100g/t
KCN-5g/t
II-nd recleaning 6 min
mixing 5 min.
CaO-100g/t
KCN-2g/t
III-d recleaning 6 min.
mixing 3 min.
KCN-2g/t
IV-th recleaning 6 min
V-th recleaning 5 min
Zn-concentrate
Total Zn-flot. tailings
tailings of II-nd Zn-flotation
thickening
mixing 5 min.
activated carbon 50g/t
classifying 0,01mm.
overflow
CaO-250g/t
KCN - 20g/t
regrinding 98% - 0,03mm.
pH-11,7
mixing 3 min.
CuSO$_4$·5H$_2$O-50g/t
mixing 0.5 min.
butyl xanthate 5g/t
MIBC - 3g/t
middling flot.10min.
recleaning 10 min.
tailings of III-d Zn-flotation

SECTION 2

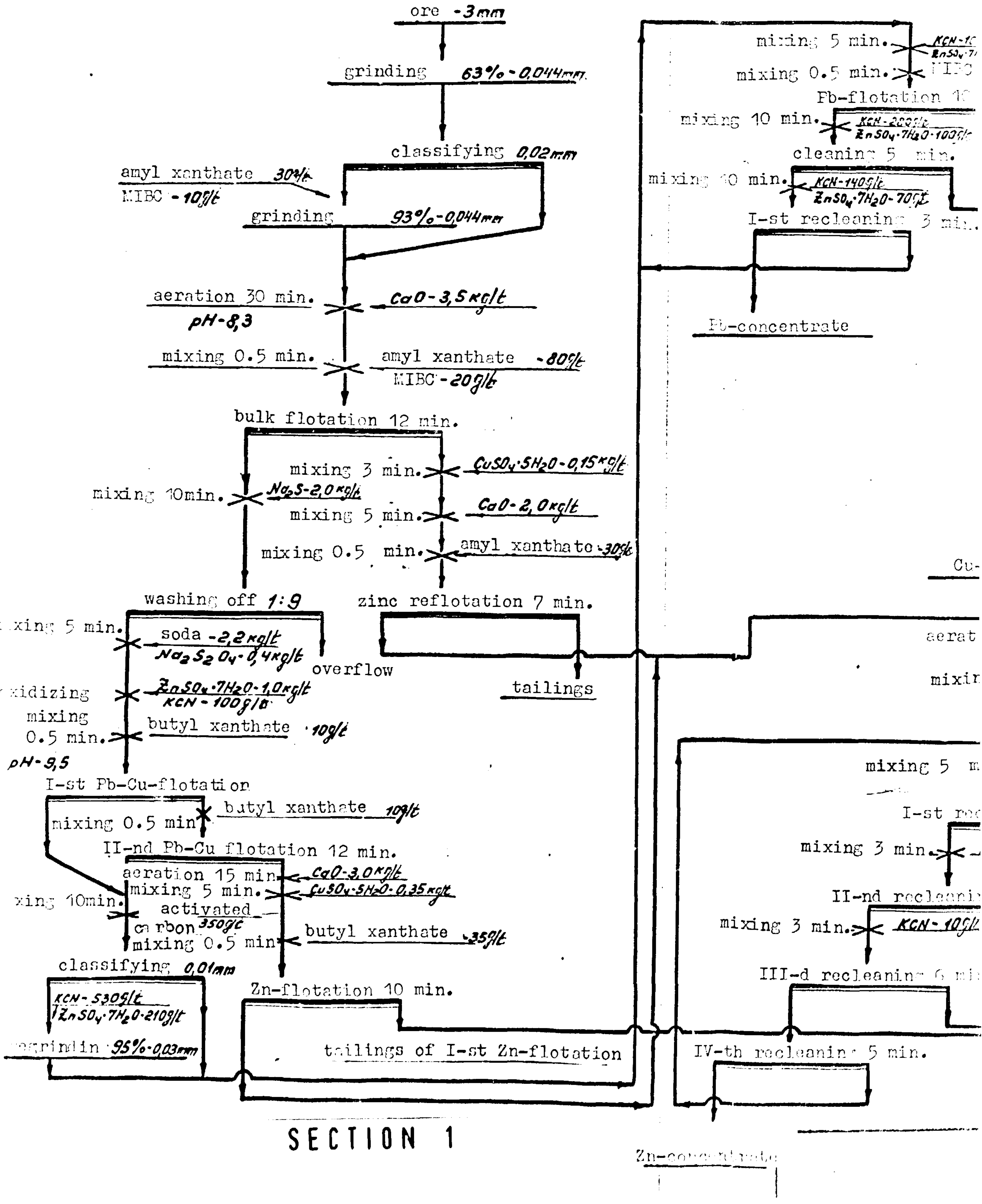

ore 3mm
amyl xanthate 15g/t
grinding 63%-0,044mm
classifying 0,02mm
amyl xanthate 30g/t
MIBC-10g/t
grinding 94%-0,044mm
aeration 30 min.
pH-8,2
CaO-3,5kg/t
mixing 0.5 min.
amyl xanthate 80g/t
MIBC-20g/t
bulk flotation 15 min.
mixing 3 min.
CuSO4·5H2O-0,5kg/t
mixing 10 min.
Na2S-2,0kg/t
mixing 5min.
CaO-2,5kg/t
mixing 0.5 min.
amyl xanthate 50g/t
MIBC-10g/t
washing off 1:9
ing 5 min.
soda 1,2kg/t
Na2S2O4-0,28kg/t
idizing
ZnSO4·7H2O-0,9kg/t
KCN-40g/t
mixing
5 min.
pH-9,2
butyl xanthate 20g/t
overflow
Zn-reflotation 10 min.
tailings
I-st Cu-Pb flotation 10 min.
mixing 0.5 min.
butyl xanthate 10g/t
II-nd Cu-Pb flotation 10 min.
aeration 15 min.
CaO-2,0kg/t
xing 5 min.
activ. carbon 150g/t
mixing 3 min.
CuSO4·5H2O-0,35kg/t
classifying 0,01mm
egrinding 100%-0,03mm
mixing 0.5 min.
butyl xanthate 35g/t
MIBC-2g/t
Zn-flotation 10 min
xing
min.
FeSO4·7H2O-100g/t
Na2SO3-480g/t
H2SO4 - pH-5,5-5,8
mixing 2 min.
tailings of I-st Zn-flotation
xin
5 min
butyl xanthate 25g/t
MIBC-1g/t
Cu-flotation
mixing 10 min.
FeSO4·7H2O
Na2SO3
cleaning 6 min
I-st recleaning 5 min.
II-nd recleaning 3 min.
Cu-concentrate
I-s
Pb-con
mixing
mixing
mixing 0.
pH-11,8
I-s
mixing 5 min.
II-nd rec
mixing 3 min.
KCN
III-d recleanin
IV-th recleaning
Zn-concentrate

SECTION 1

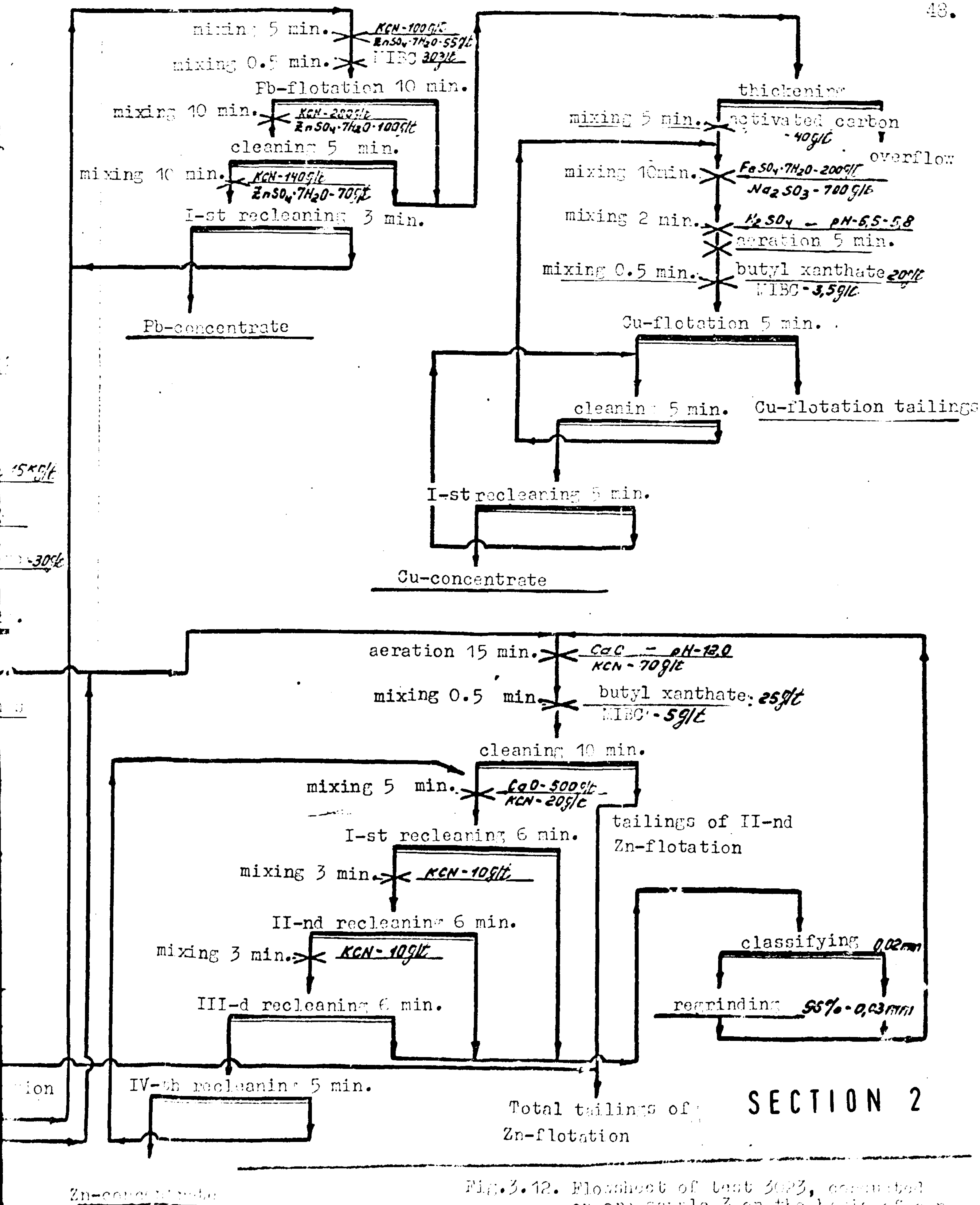


SECTION 2

Fig.3.11. Flowsheet of test 2028, conducted on ore sample^2 on the basis of continuous operation


ore -3mm
grinding 63% - 0,044mm
classifying 0,02mm
amyl xanthate 30g/t
MIBC - 10g/t
grinding 93% - 0,044mm
aeration 30 min.
pH - 8,3
CaO - 3,5kg/t
mixing 0.5 min.
amyl xanthate - 80g/t
MIBC - 20g/t
bulk flotation 12 min.
mixing 3 min.
CuSO4·5H2O - 0,15kg/t
mixing 10min.
Na2S - 2,0kg/t
mixing 5 min.
CaO - 2,0kg/t
mixing 0.5 min.
amyl xanthate - 30g/t
washing off 1:9
zinc reflotation 7 min.
xing 5 min.
soda - 2,2kg/t
Na2S2O4 - 0,4kg/t
overflow
xidizing
ZnSO4·7H2O - 1,0kg/t
KCN - 100g/t
tailings
mixing
0.5 min.
butyl xanthate 10g/t
pH - 9,5
I-st Pb-Cu-flotation
butyl xanthate 10g/t
mixing 0.5 min
II-nd Pb-Cu flotation 12 min.
aeration 15 min.
CaO - 3,0kg/t
mixing 5 min.
CuSO4·5H2O - 0,35kg/t
xing 10min.
activated carbon 350g/t
mixing 0.5 min
butyl xanthate 35g/t
classifying 0,01mm
KCN - 530g/t
ZnSO4·7H2O - 210g/t
grindin 95% - 0,03mm
Zn-flotation 10 min.
tailings of I-st Zn-flotation
mixing 5 min.
KCN
ZnSO4·7
mixing 0.5 min.
MIBC
Pb-flotation 1
mixing 10 min.
KCN - 200g/t
ZnSO4·7H2O - 100g/t
cleaning 5 min.
mixing 10 min.
KCN - 140g/t
ZnSO4·7H2O - 70g/t
I-st recleaning 3 min.
Pb-concentrate
Cu-
aerat
mixin
mixing 5 m
I-st rec
mixing 3 min.
II-nd recleani
mixing 3 min.
KCN - 10g/t
III-d recleaning 6 mi
IV-th recleaning 5 min.
Zn-concentrate

SECTION 1



SECTION 2

Fig.3.12. Flowsheet of test 3023, conducted on ore sample 3 on the basis of continuous operation

Table 3.12

Results of tests carried out under continuous process conditions to obtain bulk concentrates

Sample No.	Product	Yield, %	Content, %			Recovery, %		
			lead	zinc	copper	lead	zinc	copper
	bulk concentrate	7.0	8.13	33.05	3.30	60.0	86.1	70.8
2	tailings 1	73.2	0.35	0.39	0.10	27.0	10.6	22.4
	tailings 2	19.8	0.62	0.44	0.11	13.0	3.3	6.8
	total tailings	93.0	0.41	0.40	0.102	40.0	13.9	29.2
	ore	100.0	0.95	2.68	0.32	100.0	100.0	100.0
	bulk concentrate	9.7	4.27	15.10	10.06	51.8	74.0	80.7
	tailings 1	54.5	0.30	0.45	0.19	20.6	12.4	8.5
	tailings 2	35.8	0.62	0.75	0.37	27.6	13.6	10.8
	total tailings	90.3	0.43	0.57	0.26	48.2	26.0	19.3
	ore	100.0	0.80	1.98	1.21	100.0	100.0	100.0

NOTE: Tests have been carried out according to flowsheet in fig. 3.13.

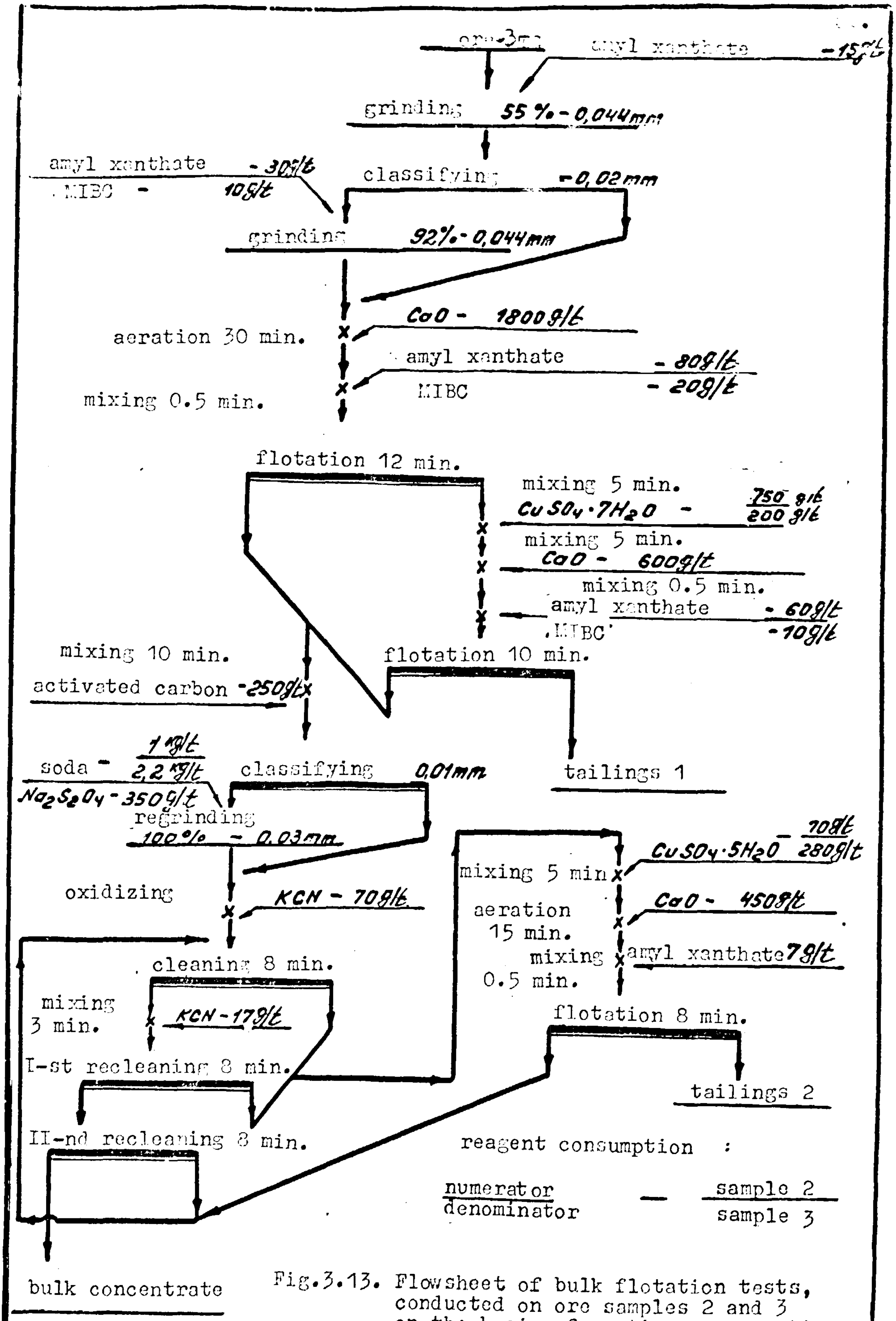


Fig.3.13. Flowsheet of bulk flotation tests, conducted on ore samples 2 and 3 on the basis of continuous operation

and a recovery 33-85.6%; zinc concentrates with 45.38 - 49% zinc (samples 1 and 2), and a zinc recovery into them 58.0 - - 56.9 %; from ore sample 3 a zinc product with 40.3% zinc and a recovery 50.4% was obtained.

As a whole, one may conclude that the results as to the lead content and its recovery into the concentrate are significantly lower in comparison with those obtained with the use of the bulk-selective flotation flowsheet. As to the results obtained for the other two concentrates, they differ to a lesser degree, but to obtain a zinc concentrate of the indicated grade, the pyrite was removed from the process in several steps (Fig. 3.10 - 3.12), as into the separation process is fed an unrecleaned lead-copper-zinc concentrate.

As to the second direction, according to the Customer's wish the tests were carried out only on ore samples 2 and 3. Pyrite depression in recleaning operations was achieved by sodium dithionite and cyanide addition in an alkaline (soda) medium at pH = 8.5. In the tests, carried out according to the flowsheet of Fig. 3.13 under continuous process conditions, bulk concentrates were obtained: with 8.13 % lead, 33% zinc and 3.3 % copper for ore sample 2; with 4.27% lead, 15.10% zinc and 10.06% copper for ore sample 3. Metal recovery figures are shown in Table 3.12. Accompanying metals content in the bulk concentrates is evidenced from Table 3.13.

Table 3.13.

Accompanying metals content in bulk concentrates

Components	Content in concentrates, %	
	Sample 2	Sample 3
Sulphur	36.84	40.06
Iron	19.25	30.45
Arsenic	0.66	0.63
Bismuth	0.0125	0.033
Antimony	1.02	0.20
Mercury	0.095	0.028
Silicon dioxide	1.15	1.15
Silver, g/t	483.3	88.3

3.4. Evaluation of the grade of semi-products as possible feed for "KIVZET"- process.

Apart from lead, copper and zinc concentrates produced by bulk-selective flotation and suitable for treatment by conventional metallurgical methods, there have been obtained semi-products (mostly these are cell products of the lead-copper separation - Figs.3.7--3.9), with cumulative metal recoveries 45.3 %, 40.8% and 43.4% for samples 1.2 and 3 respectively (metals recovered into zinc products containing 35-40 % zinc are out of consideration).

The characteristics of the semi-products are seen from Table 3.14.

The semi-products are rich in sulphur and iron and rather poor in valuable metals.

The grade of these intermediate products as a feed for the "KIVZET" - process was evaluated by experts of the VNIITSVETMET on the basis of the assays delivered. The results of an approximate estimation of possible variants of KIVZET-smelting testify the feasibility of the KIVZET- technique for treating the semi-products with a high sulphur recovery into gases and comparatively low metal recoveries into a copper-lead matte and sublimation products. Lead-copper product treatment (pos. 4, Table 3.14) is feasible only with sulphur recovered into gases, since at the indicated composition of the product practically all the metals will be lost with slags. The most expedient variant is treatment of a mixture of semi-products, composed on the basis of calculations, taking into account their yields in beneficiation processes (with the exception of lead-copper middlings).By using such a treatment, it is possible to produce: a lead--copper matte containing 10% copper and 15% lead, oxidized sublimation products containing 63% zinc and 7% lead and gases containing 65-80% disulphide. Hypothetical metal and sulphur recoveries into products of KIVZET - smelting from a semi-product mixture are given in Table 3.15. Accompanying metals (cadmium, bismuth, etc.) may be recovered into oxidized zinc sublimation products, as well as into blister copper and lead-laden dust when converting copper-lead matte.

CHARACTERISTICS OF MIDDLINGS PRODUCED DURING C
BENEFICIATION IN THE BULK-SELECTIVE FLOTAT
SYSTEM

t. o.	Samples No.	Product	Yield %	Content %											
				lead	zinc	copper	sulphur	iron	arsenic	cadmium	bismuth	antimony	cobalt	silica	gol
1	1	Copper flotation tailings	4.2	6.5	5.43	2.86	46.44	38.64	0.71	0.014	0.035	0.065	0.031	2-3[x/]	1.0
2	3	do	2.1	3.95	6.14	4.31	45.66	40.96	0.71	0.008	0.017	0.045	0.068	1-2	0.52
3	2	Zinc flotation tailings	1.9	2.39	6.0	0.70	44.6	39.0	0.66	0.012	0.030	0.040	0.079	2-3	0.77
4	2	lead-copper middlings	7.9	1.30	3.64	0.20	47.34	42.44	0.61	0.009	0.0074	0.085	0.011	1.0	0.85
5	3	zinc concentrate of middlings flotation	2.8	0.72	14.2	1.74	40.85	33.28	0.92	0.028	0.011	0.026	0.041	2.0	0.41
6		combined middlings (positions 1.2.3.5)		3.83	7.90	2.48	44.54	37.78	0.78	0.016	0.024	0.046	0.048	2.17	0.7

NOTE: Other ore constituents are not cited because

Table 3.14.

IDDLINGS PRODUCED DURING ORE SAMPLES
THE BULK-SELECTIVE FLOTATION
SYSTEM

						Recovery, %											
				g/t													
is-muth	antimony	cobalt	silica	gold	silver	lead	zinc	copper	sulphur	iron	arsenic	cadmium	bismuth	antimony	cobalt	gold	silver
.035	0.055	0.031	2-3x/	1.0	33.8	23.1	8.3	13.9	4.0	4.0	3.7	8.9	5.6	4.8	3.5	4.8	5.2
.017	0.045	0.068	1-2	0.52	10.1	9.9	6.3	7.7	2.2	2.2	2.0	2.5	27.6	1.5	3.5	2.4	1.0
.030	0.040	0.079	2-3	0.77	13.0	4.8	4.2	4.6	1.8	1.8	1.8	2.6	8.6	0.7	12.5	1.8	0.7
.004	0.085	0.011	1.0	0.85	15.0	10.9	10.8	5.5	7.9	8.3	6.7	7.9	8.9	6.3	7.2	8.1	2.7
.011	0.026	0.041	2.0	0.41	38.0	2.4	19.5	4.1	2.6	2.4	3.5	11.6	1.5	1.1	2.8	3.3	5.4
.024	0.046	0.048	2.17	0.72	26.75												

uents are not cited because of their low content in ore.

SECTION 2

Table 3.15

Hypothetical metal and sulphur recoveries into KIVZET-smelting products from a mixture of semi-products

Recovery,%		lead	zinc	copper	sulphur
into:	matte	72.5	4.4	87.0	8.0
	sublimation products	15.4	61.0	-	-
	slag	12.0	34.5	10.0	-
	gases	-	-		89.0

3.5. Conclusions.

, As a result of the investigations carried out in view to determine ore samples amenability to beneficiation, it was stated the following:

1) The bulk-selective flowsheet envisaging simultaneous flotation of lead and copper minerals with depression of pyrite and sphalerite, subsequent activation of sphalerite and separation of lead-copper concentrate provided for higher metallurgical data as compared with the bulk flotation flowsheet for lead, copper, and zinc minerals with pyrite depression and their subsequent separation into selective concentrates, though in both cases the results obtained were not high.

2) Selective concentrates can be treated by using conventional metallurgical processes. As to the zinc product of sample 3, due to a low zinc content in it (40%), it was studied additionally with a view to determine a feasibility of its treatment by a hydrometallurgical technique.

3) Due to a complicated nature of the ore mineral and chemical composition, besides selective concentrates there have been produced semi-products with a 40-45% metal recovery into them. Most of them, as is the opinion of experts, may be treated by using the KIVZET-process (Section 3.4 , Table 3.15);

4) To conduct pioneer investigations on hydrometallurgical treatment of the beneficiation products of samples 2 and 3, there have been produced bulk concentrates containing 4-8% lead, 15-33% zinc, and 3-10% copper, with respective metal recoveries 51.8-60%, 74-86% and 70.8-80.7%.

4. Pioneer investigations on hydrometallurgical treatment of beneficiation products.

The following products have been investigated:

- a zinc product, produced as a result of beneficiation of ore sample 3;

- a bulk concentrate, recovered as a result of beneficiation of ore sample 2;

and

- a bulk concentrate, produced as a result of beneficiation of ore sample 3.

To carry out the above-mentioned investigations, the products have been produced in tests under continuous process conditions according to the flowsheets, represented in figs. 3.9 and 3.13 - (Section 3.3). The weight of each product was 0.5 - 1.5 kg. The composition of bulk concentrates of ore samples 2 and 3 is seen from Tables 3.12 and 3.13 (Section 3.3).

Since the zinc product, used for the purpose of the present investigations, has been produced from the ore, that had been stored for rather a long period of time, the proportion of main constituents in it differs from that in products, obtained earlier. (See Section 3.2.5, Table 3.8).

4.1. Zinc product treatment tests

The zinc product contained: 37.42% Zn, 1.75% Cu, 0.76% Pb, 25.04% Fe, and 37% S.

Due to the fact, that the content of the main constituent in the product under research approached the lowest limit of grades for concentrates, treated in zinc plants, a conventional technology has been tested for its treatment. This technology provides for preliminary oxidation roasting of initial product with subsequent two-stage calcine leaching in sulphuric acid solutions. Zinc is extracted from the solution, after purification of the latter from admixtures, by electrolyses.

At first, the sample under research was tested to precise roasting conditions. With this aim in view, several weighted samples were roasted in a muffle at 650°C for different periods of time, and then the calcine was analysed for total sulphur, as well as for sulphur in sulphide and sulphate forms.

On the basis of the results obtained (Table 4.1) it was found out, that after a 4-hour roasting the sulphur content in the calcine is reduced to 5.6%. It is worth noting, that major amounts of sulphur turn out to be in the sulphate form. A proportion of sulphide sulphur does not exceed 0.1%. Calcine yield as produced from the initial product, is about 93%.

Table 4.1.

Results of zinc product roasting

Durability of roasting at 650°C,hr	Sulphur content in calcine, %		
	in sulphate form	in sulphide form	total sulphur
2	4.3	3.54	7.84
3	6.47	0.43	6.9
4	5.5	0.1	5.6

On the basis of the results obtained, in the tests that followed, the zinc product, before leaching, was roasted during 4 hours at 650°C.

With a view to adjust the leaching conditions, the calcine was processed in a solution, containing 130 g/l sulphur (this concentration is close to that used in practice) at different solid-liquid ratios. The temperature of the leach slurry was maintained at 60-80°C, the durability being 4 hours.

The test results were evaluated for metal content in leach insoluble residues.

The solutions were analysed for iron, because the latter is governing the efficiency of subsequent hydrolytic purification of the electrolytic solution before zinc is recovered out of it. It is known that at iron content in a solution being more than 6-7 g/l , its purification up to 0.03 g/l (this value is permissible in electrolysis) becomes quite a problem due to precipitation of voluminious iron compounds (that are difficult for filtering) out of the solution. The results of these tests are shown in Table 4.2.

Table 4.2.

Results of tests on calcine leaching

Test Nos	Solid-liquid ratio	Yield, non-soluble residue, %	Content in insoluble residue,%		Iron content in solution,g/l	Recovery into solution,%		
			Zn	Cu		Zn	Cu	Fe
5	1:5	28.0	10.8	2.3	5.8	92.5	65.7	14.2
69	1:6	21.0	11.25	2.2	15.1	94.1	76.0	37.0
70	1:7	16.3	10.48	1.94	20.0	95.8	83.2	49.0

On the basis of the results obtained, it followed, that the most acceptable is calcine processing in a sulphuric acid solution with a solid-liquid ration being 1 : 5. Under these conditions about 92.5% Zn is extracted into the solution at a relatively low degree of iron transition into it.

An increase of solvent consumption had a minor effect on the growth of zinc recovery into a solution, but to a great extent makes an iron solubility higher, thus preventing purification of the solution before zinc precipitation from it occurs.

The next tests have been carried out on the basis of continuous operation according to a flowsheet, comprising a two--stage calcine leaching, solution purification from copper, zinc electrolysis, addition of sulphuric acid into the spent electrolyte and recirculation of the latter into the head of the process (flowsheet in Fig. 4.1).

The calcine was introduced into the neutral leach stage, where it was treated by a solution from the oxidizing stage with 67-72 g/l sulphuric acid. Pyrolusite in the proportion equalling about 10 % of the calcine weight was also charged into this stage. And during the process of neutral leaching, simultaneously with a partial zinc dissolution, oxidizing of ferrous iron and its precipitation from the solution occurred. It was made possible because by the moment, when the neutral leaching was finished, which had been carried out for one hour, the solution turned out to have been completely neutralized. Its pH approached 4.5-5.0.

Upon completion of the neutral leaching the leach slurry was filtered without any difficulties, since the residue of major iron sulphates was mixed with granular material, which in itself represented a part of the calcine, that had not been decomposed during the neutral step of leaching.

The mother liquor, purified from iron by the above-indicated technique, was directed into the process of purification from copper, and the insoluble residue - sent for acid leaching. The latter process was performed in a solution, containing sulphuric acid approximately as much as 130 g/l. The acid leaching continued for 3 hr.

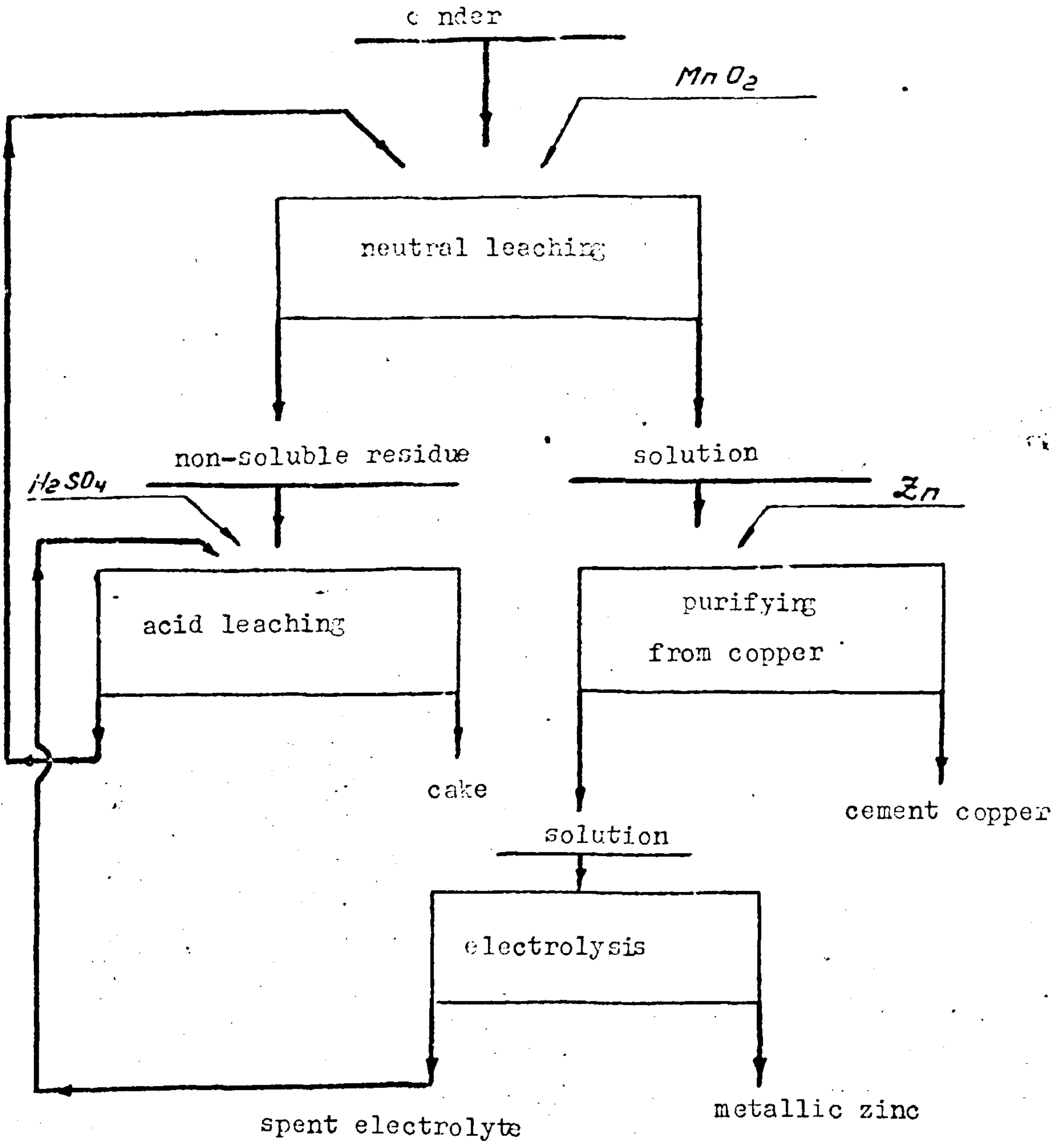


Fig.4.1. Principle flowsheet for treating the cinder produced when roasting the zinc product

The acid leaching was conducted at a solid-liquid ratio in the slurry 1 : 5. The slurry temperature was maintained at 60-70°C.

Table 4.3 illustrates the results of two tests, conducted under conditions of a two-stage calcine leaching.

The results obtained show, that with the use of the two-stage leaching flowsheet, from the calcine into a solution are extracted 90-95% zinc and 62.8 - 67.1 % copper.

The yield of the cakes is 24-28%, these latter contain 15-10 % zinc and 2.3-2.4% copper.

These cakes, as to their composition, are nearly similar to those, that are produced by treating conventional zinc concentrates. Such cakes are subjected to the Velz-process aimed at additional recovery of metal from them.

As to the solutions, obtained after filtering of the neutral leach cakes, they turned out to be purified from iron and required, before electrolysis, only purification from copper.

This operation was carried out by treating the solutions with granular zinc for 1 hr at 40°C. Zinc consumption was about 1.5% as regards the weight of the initial calcine.

The **cement** copper residue contained 81-86% copper.

The electrolysis of zinc was carried out in a vat, made of plexiglas. The anodes were cast of lead, the cathode was made of sheet aluminium.

As an electrolyte served a solution obtained after neutral calcine leaching and additionally purified from copper. It contained zinc in the region of 110 g/l.

The electric density on the cathode was equal approximately to 700 A/m^2. The voltage on the vat was 3.8-4.0 V. The electrolyte temperature was maintained in the range of 30-40° by way of cooling the vat walls with cold water.

Under these conditions, on the cathode was precipitated a dense zinc residue, which is easily separated from a matrix. The current efficiency was 89-91%, and the spent electrolyte contained zinc (39-41 g/l) and sulphuric acid (105-110g/l).

Table 4.3

Results of two-stage zinc product calcine leaching tests

Tests Nos.	Stage	Solution	Content in solution, g/l				Cake yield %	Content in cake, %		Recovery into solution, %	
			sulphur. acid	zinc	copper	iron		zinc	copper	zinc	copper
9	acid	fed	133.0	40.0	-	-					
		produced	72.0	73.0	1.5	6.1					
	neutral	fed	72.0	73.0	1.5	6.1					
		produced	pH=4.5	114.2	2.2	0.03	28.0	10.2	2.3	93.0	62.8
10	acid	fed	133.0	40.0							
		produced	69.0	74.5	1.6	7.2					
	neutral	fed	69.0	74.5	1.6	7.2					
		produced	pH=4.8	110.6	2.36	0.03	24.1	14.9	2.4	90.6	67.1

4.2. Results of treating the bulk concentrate, obtained in beneficiation of sample 2

This product was subjected to preliminary roasting under conditions similar to those practiced for the zinc product of sample 3, i.e. the process of roasting continued for 4 hours at 650°C.

The calcine obtained had 4-5% sulphur, its bulk being represented by sulphate compounds. Sulphide sulphur content did not exceed 0.1 - 0.2 %.

The phase analysis of the calcine for zinc showed, that 70% of the latter was represented by sulphate and oxide and 30% - by ferrite.

Taking into consideration a high amount of ferrite in the calcine, calcine leaching tests were carried out in sulphuric acid of different concentration with a constant solid-liquid ratio of the slurry 1 : 7,5 . The leaching process continued for 4 hours. The slurry temperature was 60-80°C.

The results of these tests, listed in Table 4.4, showed, that the leaching of the calcine, obtained from the bulk concentrate of sample 2, was less effective, than that, which had been conducted on the zinc product, produced from sample 3. To obtain high results it was necessary to apply sulphuric acid solutions of higher concentration, capable to decompose ferrites. However, the main disadvantage of the process lied in the fact that under conditions of a more complete transition of zinc into a solution, an intensive solubilization of iron took place. The content of zinc in the solution reached 12-25 g/l, that is considerably higher than it is permissible.

With an objective to increase the selectivity of the process, an oxidizing - autoclave technique was tested to treat the product. In conformity with this technique sulphide polymetallic beneficiation products are treated with sulphuric acid solutions in an autoclave at an elevated temperature, with the use of pressurized oxygen or compressed air to oxidize sulphides.

In this operation, zinc sulphide is conversed into a soluble sulphate according to the following reaction:

$$ZnS + H_2SO_4 + \frac{1}{2} O_2 \rightarrow Zn\,SO_4 + H_2O + S^0$$

accompanied by an isolation of elemental sulphur.

It is known, that under conditions, providing for a high degree of sphalerite decomposition, primary copper sulphides and pyrite react but relatively little. As to lead sulphide, it forms a sulphate of this metal.

The tests were conducted in an autoclave of 1 l capacity, made of titanium.

Into the autoclave were charged a weighted portion of the concentrate and a solution, containing sulphuric acid (100g/l) and zinc (40 g/l). The solid-liquid ratio in the slurry was 1 : 5.

The amount of the sulphuric acid charged was 115% with respect to a stoichiometric quantity, taking into account the zinc content in the weighted portion.

The contents of the autoclave were heated for 4 hours at a temperature about 105°C. During this period of time pressurized oxygen at 4 atm. was fed into the autoclave. During this leaching process the slurry was vigorously mixed with a turbine stirrer at 2600 r.p.m.

After the indicated lapse of time the autoclave was cooled, the oxygen released and the slurry in the autoclave - subjected to heating up to 150°C, the slurry being retained at this temperature for an hour. It is common knowledge, that as a result of slurry heating up to a temperature higher than that of sulphur melting, the latter envelops grains of underdecomposed sulphides and forms granules of 4-10 mm size, easily separated from the rest part of the insoluble residue by screening.

This operation made it possible, after the filtration of the autoclave slurry, to separate the cakes thus produced into two parts, from which one part happened to be lead-beneficiated to such an extent, that it could be defined as a lead-

Table 4.4.

Leaching results of calcine obtained after roasting of sample 2 bulk concentrate.

Tests Nos.	Sulphuric acid solution concentration, g/l	Acid consumption, % of estimated	Yield of insoluble residue, %	Content in insoluble residue, %				Iron concentration in solution, g/l	Recovery into solution, %		
				zinc	copper	iron	lead		zinc	copper	iron
56	100	110	53.0	16.25	2.46	32.81	14.9	6.0	75.8	69.3	18.0
60	129	165	40.0	14.0	2.44	30.6	20.8	12.2	82.5	74.6	42.9
75	300	380	20.0	1.12	0.33	13.8	39.5	25.1	99.3	98.3	86.5

-concentrate.

In Tables 4.5 and 4.6 are listed the results of one of the tests.

Table 4.5.

Results of the autoclave leach tests, conducted on the bulk concentrate, obtained as a result of sample 2 beneficiation

Total yield of insoluble residue, %	Content in insolubee residue, %			Content in solution, g/l		Recovery into solution, %		
	Zn	Cu	Pb	Sulphuric acid	Fe	Zn	Cu	Fe
62.3	2.16	40.7	12.2	pH=1.2	1.34	95.30	26.5	4.6

Table 4.6

Results of screening the insoluble residue, obtained after the autoclave leaching of the bulk concentrate, produced as a result of sample 2 beneficiation.

Products of insoluble residue screening	Yield from insoluble residue,%	Content, %			Recovery, %		
		Zn	Cu	Pb	Zn	Cu	Pb
Oversize	71.8	2.45	5.22	2.8	81.0	92.5	16.5
Undersize	28.2	1.42	1.13	36.1	19.0	7.5	83.5
Initial product	100.0	2.16	4.07	12.2	100.0	100.0	100.0

On the basis of the above-indicated results it was possible to conclude, that the autoclave process is effective enough for treating the bulk concentrate.

Its application provides for a high degree of zinc recovery into a solution (more than 95%). At the same time it turns out to be feasable to extract lead from an insoluble leach residue into a lead concentrate with zinc recovery being 83.5% as regards the initial product. Alongside with zinc, 26.5% Cu and about 5% Fe as regards these metals content in the initial product are transferred into the solution.

A comparatively low Fe-content in an autoclave solution (1.84 g/l) facilitates the solution purifying (before extracting zinc out of it) that is highly important for process steps that follow. Of interest are the results of the x-ray analysis of the products obtained by screening the insoluble leach residue.

These results show, that with pyrite being dominated, in the granular part of the product are present elemental sulphur, chalcopyrite, a small amount of gray copper ore, anglesite and negligible sphalerite.

As to the undersize product, its major phases are anglesite and plumbojarosite /$PbFe_6(SO_4)_4(OH)_2$/ (70% Pb are represented by anglesite and 30% Pb - by plumbojarosite). In this product there were small amounts of pyrite and sphalerite, in still less amounts were galena and chalcopyrite.

The undersize product of the insoluble residue was subjected to processing in a 25% - solution of sodium chloride for 1 an hour, at 100°C. After that the insoluble residue was filtered out, in which plumbojarosite as the major phase was detected by way of X-ray analysis. The anglesite was completely transferred into the solution according to the reaction

$$PbSO_4 + NaCL \longrightarrow PbCL_2 + NO_2SO_4$$

Since plumbojarosite was absent in the initial concentrate, it is possible to conclude that the finely ground plumbojarosite (- 1 µk), detected in the cakes of the autoclave leaching, is a newly formed compound, that is obtained as a result of interaction of lead compounds with Fe-sulfate, that are contained in the autoclave solution. As a result of this interaction, iron precipitation from the solution seems to occur, and its content decreases up to the indicated value.

To bring the content of this admixture up to 0.02-0.03 g/l, a hydrolytic technique of purifying solutions from iron in the autoclave has been applied. The solution was neutralized by lime up to pH = 4.5 and treated in an autoclave for an hour at 140°C and 4 atm oxygen pressure. During the process a precipitate was formed, that contained 6.48% Zn and 2.83% Cu. The yield of the precipitate was 2.3% as regards the weighted portion of the initial concentrate; 0.5% Zn and 1.8% Cu, as regards the initial content, have been extracted into it. The solution was then directed for copper cementation that was accomplished by its treating with granular zinc, as described earlier.

To speed up the processes, zinc was introduced in excess. Zinc precipitation out of the solution, that had been purified from admixtures, was performed by electrolysis.

Electrolytic zinc precipitation was carried out at current density being 700 A /m^2. This was accompanied by a formation of a dense zinc residue, that is easily separated from a matrix, at current efficiency ~ 95%.

In the spent electrolyte there has been left about 40 g/l zinc and 50 g/l sulphuric acid.

4.3. Tests on processing bulk concentrate obtained in beneficiation of sample 3.

The results of the tests according with the flowsheet, including preliminary concentrate roasting and subsequent calcine leaching in sulphuric acid solutions of various concentration, indicated that this flowsheet due to the lack of process selectivity, is also unfit for sample 2 bulk concentrate treatment.

As shown in Table 4.7, a large quantity of copper and iron simultaneously transfers into a sulphuric acid solution under conditions providing for high zinc recovery from calcine.

Iron content in the solution reaches dozens of grams per littre, which makes extraction of a high grade zinc product from it practically impossible.

Owing to all this, tests for pressure leaching of this product were conducted.

Leaching was carried out under conditions analogous to those accepted during the treatment of sample 2 bulk concentrate.

Leaching temperature was 105-110°C, duration - 4 hours. During the whole period of treatment, oxygen from an oxygen container was fed to an autoclave under 4 atm pressure. Total pressure in the autoclave was maintained at the level of 5.2 -5.5 atm. Solids content in the slurry was 20 %. At the beginning of the operation, the solution fed to the autoclave contained about 40 g/l Zn and various quantities of sulphuric acid, in accordance with its desired surplus, in comparison with its stoichiometric quantity, necessary for zinc sulphate formation. The results of these tests are given in Table 4.8.

The results obtained indicated, that the effect of sulphuric acid dosage on the results of metal leaching in an oxidizing-autoclave process was essential, as visible dissolving of copper and iron takes place simultaneously with that of zinc.

However, a zinc dissolving rate is apparently higher. Therefore, with a comparatively small solvent surplus, transferring of most of the metal into a solution is observed. When the surplus is noticeably increased, the process selectivity is disturbed . Thus, at sulphuric acid surplus being 150% relative to its stoichiometric quantity, 89.1 % Zn, 32% Cu and only 10.8% Fe were recovered into the solution. However, when a sulphuric acid surplus was 275%, with a 10% increase in zinc recovery, copper recovery increased by 23.3 %, and iron recovery - - by 50%.

Table 4.7

Results of sulphuric acid leaching of calcine obtained after sample 3 concentrate roasting

(Test conditions: concentrate roasting for 4 hours at 650°C;
leaching for 4 hours at 60-80°C;
at a solid-liquid ratio 1 : 5).

Test No.	Sulphuric acid, content, g/l	Yield of insoluble residue, %	Content in insoluble residue, %				Iron content, g/l	Recovery into solution,%		
			Zn	Cu	Pb	Fe		Zn	Cu	Fe
72	130	40.0	1.55	1.33	11.5	36.7	26.3	95.6	95.1	53.5
75	300	31.5	0.25	0.22	14.7	27.1	40.2	99.5	99.35	71.2

Table 4.8

Results of pressure leaching tests on bulk concentrate obtained from sample 3.

Nos.	Sulphuric acid dosage		Yield of insoluble residue, %	Content in insoluble residue, %			Iron content in solution	Recovery into solution, %		
	g/kg	% as regards stoichiometric quantity		Zn	Cu	Pb	g/l	Zn	Cu	Fe
42	225	100	93.8	6.40	8.99	4.90	1.12	60.0	21.6	2.0
91	340	150	80.7	2.04	9.06	5.6	6.1	89.1	32.0	10.8
77	600	275	43.3	0.26	11.1	10.6	34.4	99.2	55.3	61.0

Thus, it was found out, that an oxidizing - autoclave process can also be used for sample 3 bulk concentrate treatment. It is only necessary to adjust carefully the conditions of autoclave leaching and, in particular, to specify a sulphuric acid dosage.

In our tests, with sulphuric acid consumption 150% of the amount required for binding zinc into a sulphate, iron proportion in the autoclave solution was 6.1 g/l, which allowed to obtain a solution, purified from iron (iron content was 0.035 g/l), as a result of its neutralization by lime up to pH ~ 4.0 and subsequent treatment in an autoclave at 140°C and 4 atm oxygen pressure.

The solution purification from copper was carried out by copper cementation with granulated zinc. After cementation copper traces remained in the solution, and the cement residue contained 84% metal. A cement residue yield made up 4.3% of the concentrate.

Zinc was precipitated from the solution purified from iron and copper by electrolysis. The electrolytic precipitation of zinc was carried out from a solution, containing 83.2 g/l zinc. The electrolysis was conducted under conditions, similar to those described in the previous Section, i.e. at current density 700 A/m^2 and voltage 3.5 - 3.8 V, with a dense metal residue being deposited on the cathode at current efficiency 94.6%. The spent electrolyte contained 29.2 g/l zinc and 80.3g/l H_2SO_4.

As to the insoluble residues after bulk concentrate leaching, they were kept during an hour in a 20% - sodium chloride solution containing 20 g/l muriatic acid, in order to recover lead. Solids to liquid ratio was 1 : 1, slurry temperature - 100°C. The results of this test are shown in Table 4.9.

Lead was precipitated from the solution by heating the latter up to 60°C with soda solution added to reach pH ~ 8.

Table 4.9

The results of lead leaching out of autoclave process cakes.

Insoluble residue yield of lead leaching,%	Content in insoluble residue, %			Lead recovery into solution, %
	lead	zinc	copper	
90.8	1.2	2.2	9.9	80.7

Apparently, a residue with metal content 43.7% consisting of a lead carbonate and hydrate mixture was precipitated. The mother liquor contained traces of lead. In the flotation tests conducted with a view to increase copper content in the insoluble residues which remained after lead leaching, an attempt to rise its content higher than 12% was not successful, apparently,due to intimate association of copper minerals with pyrite.

4.4. Conclusions.

On the basis of the research investigations it was found out, that, in pirnciple, there is a feasibility to treat the three products, produced as a result of beneficiation of the ore samples tested, with the use of hydrometallurgical processes.

As final products there have been produced: cathodic zinc, lead as sulphate and carbonate, and copper in the cement form, and as a 10 % - concentrate.

Cathodic zinc was precipitated out of the zinc product, that had been treated by the standard technology, with recovery, on the ore basis, being 43%. It is worth noting, that there is a possibility to increase zinc recovery by way of retreating cakes (produced in zinc concentrate leaching) by existing methods.

The leaching of bulk concentrates, obtained in the process of beneficiating samples 2 and 3, was carried out by means of an oxidizing - autoclave technique. About 79% Zn (as considered from the ore content) was extracted from the bulk concentrate of sample 2 into cathodic zinc.

Besides, out of this product there has been produced a lead concentrate with 36.0% lead and a 50% recovery (on the ore basis).

As to copper, one can expect to obtain a 16% copper concentrate with recovery being about 50% (on the ore basis), by way of flotation of the insoluble residues, produced in leaching the product.

About 20-22% copper in the form of a cement precipitate is deposited out of an autoclave solution.

Treatment of product 3 provides for recovery (on the ore basis) about 64% zinc into cathodic metal, 41% lead into a mixture of carbonate and hydrate of this metal with a lead content 43.7%, about 55% copper into a 10%-concentrate and nearly 26% copper as a cement precipitate.

5. GENERAL CONCLUSIONS

Three samples of polymetallic pyrite ore of complex composition from Portugal have been studied.

In spite of the commercial content of lead, copper and zinc,the ore of these samples is amenable to beneficiation but with difficulty. This can be explained by very fine and non-uniform dissemination of all valuable minerals in the pyrite, the content of which is 81-88% and by the presence of a variety of lead and copper minerals of complex composition, accompanied by an isomorphic iron admixture in sphalerite.

During the studies, oxidation of the stored ore was observed, that resulted in a decrease of flotation activity of chalcopyrite, and especially galena.

As a result of the studies, it was revealed that:

1. In case of flotation for the purpose of obtaining selective concentrates, it is reasonable to concentrate the ore samples according to a bulk-selective flotation flowsheet providing for successive recoveries of a lead-copper concentrate (and its subsequent separation into lead and copper concentrates), as well as of zinc and pyrite concentrates from the ore. In this connection, lead, copper and zinc concentrates were obtained (the latter ones are produced from samples 1 and 2). A zinc product with zinc content 40.3% and recovery 49.5% was obtained from ore sample 3 (Fig. 3.7 - 3.9).

The corresponding concentrates contain 40.3 - 50.8% lead, 16-24% copper, and 50.31-51.88% zinc. Recoveries into the concentrates are as follows: 30-42.6% lead into a lead concentrate, 50.4-67% copper into a copper concentrate, and 55.5 -59.8% zinc into a zinc concentrate. The concentrates are suitable for usual methods of metallurgical treatment.

2. The tailing of the ore flotation circuit containing 45.5 - 50% sulphur at recovery 77.9 - 81.4 % is a pyrite concentrate that can be used in sulphuric acid production.

3. According to the developed technology, apart from the concentrates, there were obtained some semiproducts, into which, at low metal content (2-6%), 40-45% metals were recovered (total for each sample).

According to the estimate of VNIITZVETMET specialists, there is, in principle, a possibility to treat mixtures of these products by the "KIVZET" - process with a high recovery of sulphur (89%) into gases and a comparatively low metal recovery into a copper-lead matte (82% copper and 72.5% lead) and sublimates (61% zinc).

Thus, there can be obtained: copper-lead matte with copper content 10%, lead content 15%; oxidized sublimates with zinc content 63% and lead content 7%; gases containing sulphurous anhydride- 65-80%.

4. The research investigations of hydrometallurgical treatment of the beneficiation products showed that:

a) In principle, there is a possibility of treating the zinc product obtained from ore sample 3, according to conventional technology for zinc concentrates treatment.

Evidently, similar conclusion would be justified with respect to the zinc concentrate of the middlings flotation, containing 35.94% zinc, obtained from ore sample 1 (Table 3.8).

b) The combined ore treatment flowsheet is competitive with a flotation one. The flowsheet includes: obtaining of a bulk concentrate by flotation (Fig. 3.13), its leaching in an oxidizing - autoclave process with subsequent extraction of metals from leaching products. In this case, there can be recovered: zinc into cathodic zinc - 64-79%; lead in the form of sulphate, carbonate, or lead concentrate - 41-50%; copper in the form of a 10-16% concentrate - 50-55%, and about 26% copper in the form of cement copper.

5. The above indicated bulk concentrates obtained as a result of samples 2 and 3 processing containing 4-8% lead, 15--33% zinc, 3-10% copper with recoveries 51.8-60% lead, 74-86% zinc and 70.8-80.6% copper cannot be effectively treated by the "KIVZET"- process.

6. The principal combined flowsheets for processing the ore samples tested are shown in Figs. 5.1 and 5.2.

7. The conditions, practiced for pyrite and sphalerite depression, as well as those applied for a non-cyanide separation of the lead-copper concentrate are protected by a licensing law in the USSR.

8. In spite of low metal recoveries, the data obtained are, as a whole, satisfactory taking into consideration a complex nature of mineral and chemical composition of the ores.

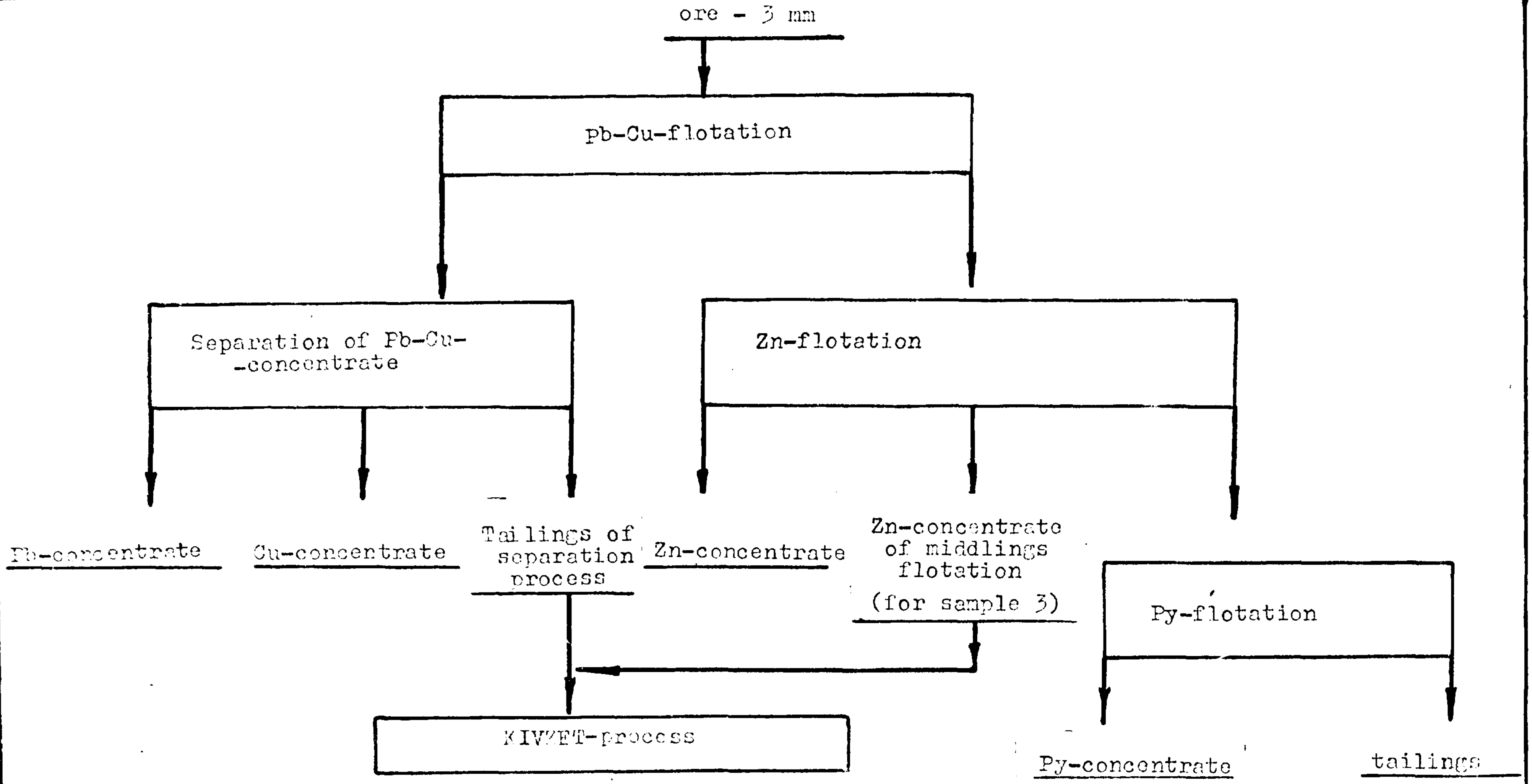


Fig.5.1. Principal flowsheet for ore treatment by means of flotation and KIVZET-process

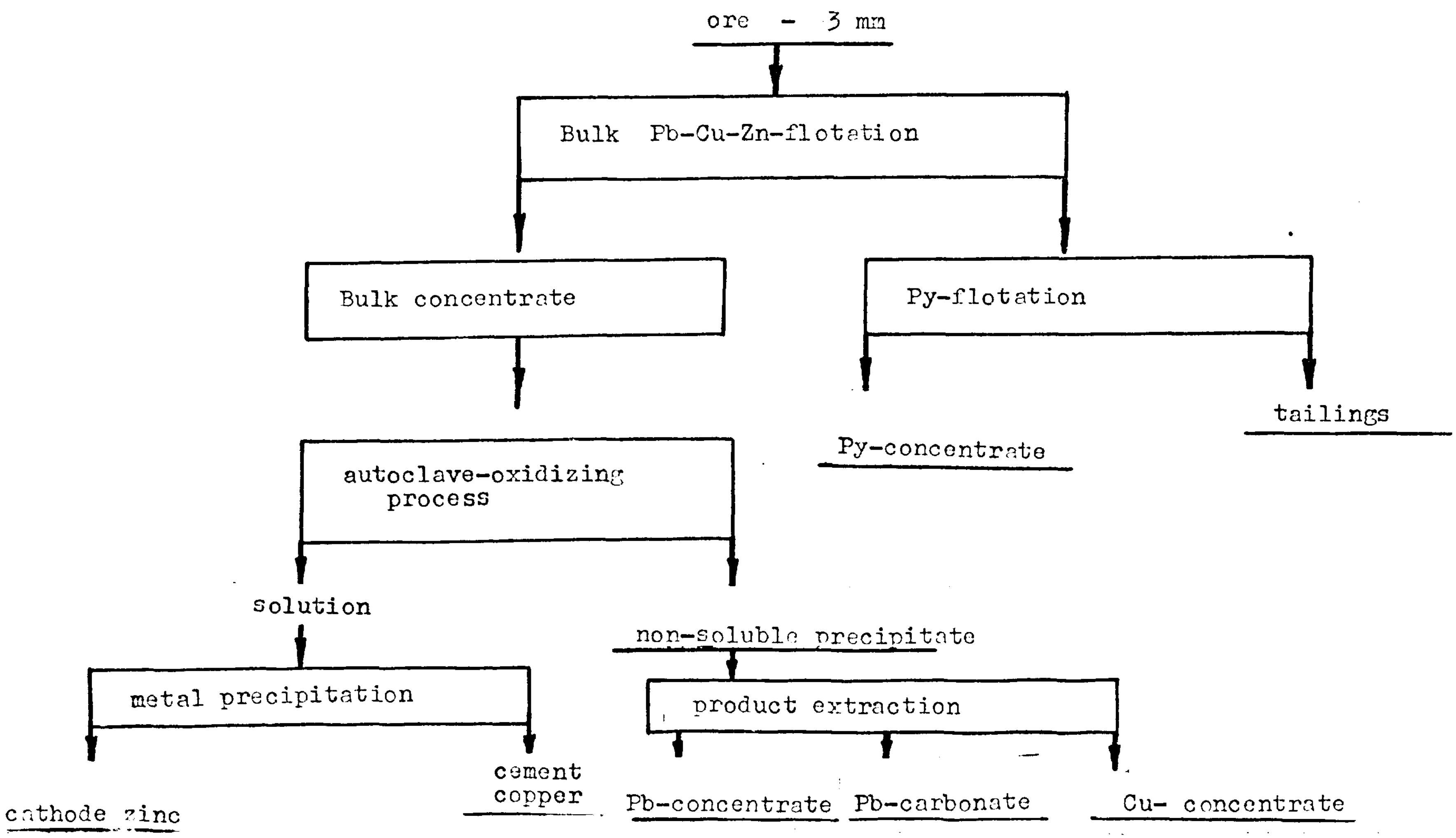


Fig.5.2. Principal flowsheet for ore treatment by means of flotation and hydrometallurgical processes.

6. Recommendations for using the laboratory testwork results

Based on the data resulted from the investigation of three samples of pyrite polymetallic ore, the following should be recommended:

1. Carry-out the feasibility study of both process flowsheets given in Figs 5.1 and 5.2 , with a view to determine the optimum one for dressing Portugal pyrites.

2. Discuss with the UNIDO and Portuguese party the possibility of conducting a pilot-plant flotation testwork based on a selected process flowsheet.

3. Carry-out, using specialized methods and in conformity with an adopted process flowsheet, a scaled-up laboratory testwork on refining the products and semi-products recovered during the pilot-plant ore dressing.

4. Determine the subsequent work resulting from the data of testwork envisaged in items 2 and 3.