

# Distribution of non-ferrous metals, arsenic and antimony during plumbous slags sulfidizing impoverishment by copper-zinc concentrate

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The existing technology of semiproducts and reverts processing is characterized by a number of disadvantages, the main of which are low non-ferrous metals recovery into target products and considerable accumulation of arsenic and antimony in the primary production process because of their low recovery into dust. Along with the raw materials, a considerable proportion of arsenic and antimony is received with converter slag, which constitutes 26% in the total volume of the raw material. With converter slag, up to 30% of the total arsenic volume in the charge comes to the process.

The present paper is dedicated to solving a problem of arsenic and antimony deep sublimation from slag to dust and withdrawing them out of the process chain in the form of non-toxic sulfide compounds.

Results of the converter slag recovery-sulfiding depletion by the sour copper-zinc concentrate are presented in the paper. Experimentally is proved a possibility of the slag deep non-ferrous metals, arsenic and antimony depletion with matte and metal lead obtaining in condensed phases. It is established that on charging a fresh portion of the copper-zinc concentrate, excess sulfur arising as a result of the principal sulfides dissociation is completely spent for arsenic oxide and antimony sulfidizing. In conditions of high temperature (1573 K), the Gibbs free energy of arsenic oxide and antimony sulfidizing is as follows:  $\Delta G_{1573K} = -862.40$  kJ/mol and  $\Delta G_{1573K} = -746.9$  kJ/mol respectively, that is almost 2 times greater than the values of the Gibbs free energy of the interaction reactions of non-ferrous metals and ferric oxides with elemental sulfur. High values of the Gibbs free energy testify an arsenic and antimony deep sublimation to dust as non-toxic sulfides  $As_2S_3$  and  $Sb_2S_3$ . The impurities sublimation completeness depends on consumption of the principal sulfides (excess sulfur) fed with concentrate: the greater is the introduced excess sulfur quantity, the greater is an arsenic and antimony sublimation to dust. It is shown that on the converter slag with high lead content treatment with different sour concentrate consumption, the highest possible lead extraction to lead bullion and copper extraction to matte are achieved. It was determined that at temperature of 1573 K and optimum concentrate consumption of 20%, extraction of copper to matte is 96%, extraction of lead to lead bullion is 92% and extraction of zinc to slag is 87%. Besides, high arsenic and antimony extraction to dust is achieved with amounts of 82% and 78% correspondingly. It is demonstrated that the copper content in the matte is decreasing as the concentrate consumption rises above 20%. It is determined an equilibrium composition of the obtained products. Listed are the results of copper, lead, zinc, arsenic and antimony distribution among the melting products.

The obtained results are applied to the calculation of initial charge with new composition in case of semiproducts and reverts of the lead industry processing in combination with the copper-zinc concentrate under the shaft concentrating conditions. Use of the new technology will significantly reduces coke consumption in the smelting process and guarantees straight processing of a collective difficult-to-process copper-zinc concentrate for one stage. As this take place, high results of both copper extraction to matter and zinc extraction to slag are achieved. Also, as a result of arsenic and antimony deep sublimation to dust, it is provided their maximum withdrawing out of the process, thereby essentially rising quality of the obtained products.

**Key words:** slag, copper-zinc concentrate, impoverishment, non-ferrous metals, arsenic, antimony, recovery, matte, lead bullion, distribution.

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## Introduction

Typical technological and economic peculiarity of the lead industry enterprises is a great arsenic accumulation at the main lead production stage. The existing technologies of lead manufacturing are no longer cope with processing of the complex-composition

raw materials, which contains a fair arsenic and antimony amount (growth of arsenic accumulation, rising of material expenditures, increase of amounts of accompanying semiproducts and revert, low process characteristics, the obtained products' quality deterioration, etc.). As an analysis shows, the present day none of the processes included to the general lead manufacturing technological

flow sheet allows achieve deep enough arsenic and antimony sublimation to dust, which leads to their significant accumulation in the primary process of lead production. Deterioration of the quality of the obtained smelting products results in drastic increase of both material expenditures on consequent operations of their treatment and ecological load on the environment and human health. In view of the rigid requirements to the environment condition, the problem of the integrated waste gas and sewage water treatment of arsenic compounds has become much more pressing [1].

Development and application of effective means of arsenic and its compounds withdrawal out of the metal industry manufacturing methods by converting them to non-toxic, easy-kept products now have become a challenge which demands an urgent decision. Relevant theoretical investigations have been especially intensive during last years. An arsenic behavior in different processes of copper smelting and copper mattes converting are the subject of a considerable technical literature [1–8]. Nevertheless, the questions of arsenic and other impurities distribution among the smelting products, even in the case of traditional lead melts, are still open and require an additional research. Study of this question is currently central for an alternative blast concentrating smelting, in which products with the heightened arsenic and antimony contents there are processed.

The blast concentrating smelting is considered as the most-risk unit in the general technological chain of lead manufacturing on Kazzinc LLC. There is processed almost complete assortment of semiproducts and reverts of the lead industry. In the absence of efficient treatment mode for the mentioned products, the existing forced separate processing of the complex-composition raw materials, consisting of a mix of poor mattes, converter slag, alkaline fluxes, leaden cake etc., with heightened arsenic content doesn't solve but strengthen problem of arsenic and antimony accumulation in the lead manufacturing. Technological indexes of the process are also unfavorable. Non-ferrous metals extraction to target products is situated on a low level with the following amounts: copper to matte up to 85%, lead to lead bullion of ~60%, zinc to slag of ~65%. Arsenic and antimony sublimation into dust amounts to ~60% and 55%, respectively.

In [1] it is shown that the main arsenic proportion of its total quantity in the smelting charge comes with converter slag (up to 30%). It is an evidence of necessity to make provision for additional efforts on process organization aimed to both cutting down losses of non-ferrous metals along with slag and arsenic and antimony deep sublimation into dust.

Depletion of non-ferrous metal slags has been the objective of much research [9–17]. Each method has been developed with reference to conditions of specific engineering process and has been first of all aimed at improvement of in-process figures: non-ferrous met-

als yield enhancement due to lowering their losses with slag. However, questions of arsenic, antimony and other accompanying impurities behavior have not been adequately and system examined by the authors of the above mentioned methods. Each separately developed method of slag depletion should be stipulated not only by economic reasons, such as an enhancement of non-ferrous metals yield out of slag, but an ecological expediency of the taken decision as well, for example, the concomitant impurities removal out of the engineering process as non-toxic, easily-buried and/or utilizable compounds.

The objective of the present paper is an investigation of arsenic, antimony and non-ferrous metals distribution during converter slag sulfiding depletion by a sour copper-zinc concentrate as applied to conditions of semi-products and reverts of the lead industry treatment.

### Research procedure

Scheme of the unit for realization of experiments is shown in Fig. 1.

Procedure of pursuance of experiments in all the meltings has been identical and has come to co-smelting of converter slag with different sour copper-zinc concentrate consumption in presence of quartz. To avoid slag overoxidation by air, the experiments have been carried out in atmosphere of a rare gas (Ar), fed out of cylinder (1). The main point of the procedure has been as follows. Crucible with initial test charge (5), consisting of converter slag (0.30 kg) and quartz (in amounts depending of the concentrate consumption), has been plugged and charged to a silit shaft furnace (4).

Three holes in the plug are provided for alundum tubes placing: a rare gas has been introduced through the first tube (6), waste gas extraction out of the reaction zone has been fulfilled through the second one (8). The third tube (7) has been placed in the middle and stopped by a plug which has been pulled out on concentrate charging.

After concentrate charging tube has been again stopped by a plug. That technique has allowed an initial concentrate charging to the melt without capsulation failure. Stack gases has been absorbed in the special vessel with water (9). To avoid water saturation by sulphur dioxides, vessel with water has been periodically replaced. Temperature has been kept equal ot 1573 K and has been controlled by a platinum thermocouple (10). Temperature has been regulated by the use of metering equipment (11). As the required temperature achieved, sour copper-zinc concentrate has been poured to the melt surface through the tube (7). Concentrate consumption has accounted 5%, 10%, 15%, 20% and 25% of the converter slag weight. After concentrate charging, tube (6) has been submerged in the melt for its intensive blowing by argon for 10 min. Argon consumption has been controlled using gas valve (2) and rotameter (3). In all the

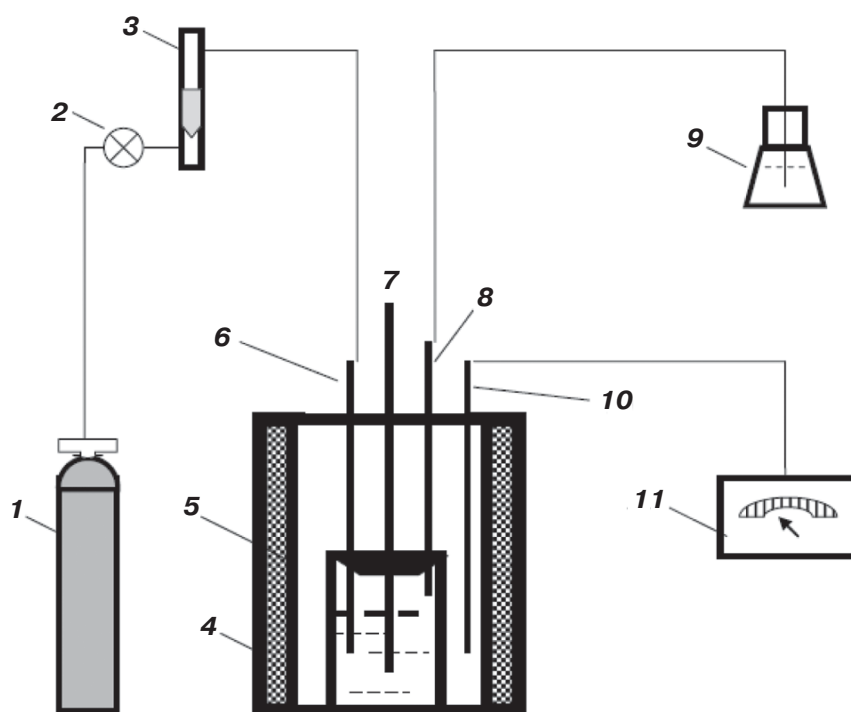


Fig. 1. Scheme of the laboratory-scale plant:

1 — argon cylinder; 2 — gas valve; 3 — rotameter; 4 — silit shaft furnace; 5 — crucible with melt; 6 — blow tube (Ar); 7 — tube for concentrate loading; 8 — tube for gas output; 9 — vessel for gas absorption; 10 — thermocouple; 11 — metering equipment

experiments argon consumption has been constant and was equaled to 0.3 l/min.

When the required blowing time is up, tube (6) has been switched to the “above the melt” and the melt settling has been carried out in argon stream for 15 min.

During the preliminary experiments, it has been found out that the settling time duration (5, 15, 30 min) exerts little influence on the final content of non-ferrous metals and impurities in the melting products. As this take place, formation of slag, matte and lead bullion phases and their clear-cut separation has been achieved even in insignificant settling time.

When the required settling time was up, a crucible has been taken out of the furnace for air cooling. After cooling down crucible has been broken up. The obtained smelting products has been separated and weighed and then they have been subject of a chemical analysis to detect copper, lead, iron, arsenic and antimony presence.

### The results of the experiments and their discussion

Composition of source materials is shown in the Table 1.

Converter slag of the 1<sup>st</sup> period is obtained as a result of converting copper-lead mattes of the shaft concentrating smelting. Character of these mattes is an increased content of lead and low values of iron and quartz. Technological specifics of the process and distribution of metals among concentrating products are elaborately covered in [3]. Sour collective copper-zinc concentrate of Nikolayevskoye deposit of the Eastern Kazakhstan region has been chosen as a sulfidizing agent for slag depletion. Taking into account complexity of treatment of such a type of concentrate, in experiments there were pursued the objective of a complex task solution, namely, using concentrate both as a raw material for extracting non-ferrous metals into target products by direct smelting in one stage and simultaneously as a sulfidizing agent

for non-ferrous metals slag depletion and arsenic and antimony deep sublimation.

Fig. 2 shows a dependence of the residual non-ferrous metals, arsenic, antimony content and smelting products yield on the concentrate consumption. One can see that increase of concentrate consumption from 5% to 25% is accompanied by decrease of copper and lead content in slag and by increase of zinc content in it as well (Fig. 2, a)). In the conditions under consideration, it is also ascertained an essential reduction of arsenic and antimony content in slag: from 2.4% to 0.32% and from 0.92% to 0.25%, correspondingly (Fig. 2, b)). Depending on the concentrate consumption against a background of the matte, lead bullion and dust quantity increasing, it is observed slag volume lowering with its further smooth growth at the concentrate consumption above 20% (Fig. 2, c)).

The established mechanism is perfectly explainable based on the general concept of thermodynamical

Table 1

Chemical composition of the source materials

Product	Content of components, % (wt.)								
	Cu	Zn	Pb	Fe	S	As	Sb	SiO <sub>2</sub>	CaO
Converter slag	3.83	4.54	33.50	12.70	0.60	2.80	0.95	15.7	1.24
Copper-zinc concentrate	18.57	12.50	2.30	24.59	33.40	0.22	0.25	—	—

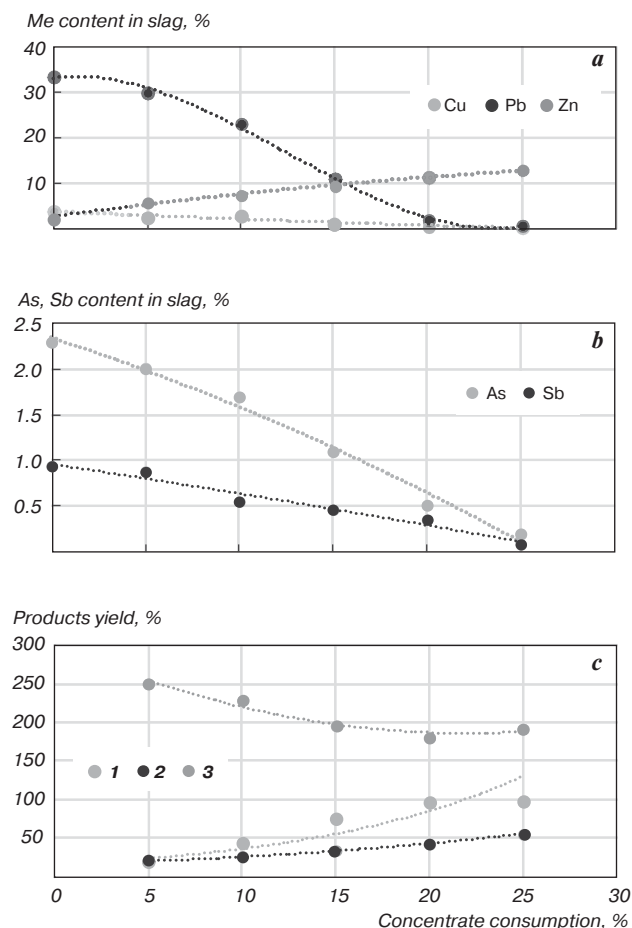
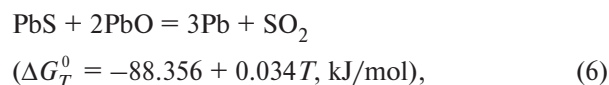
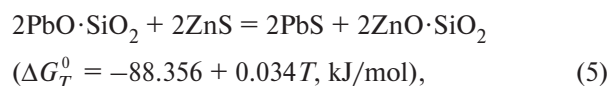
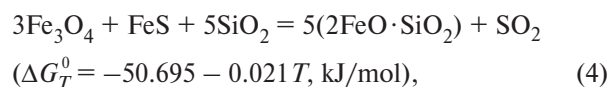
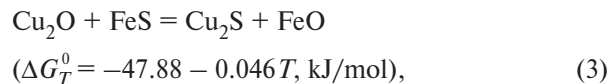
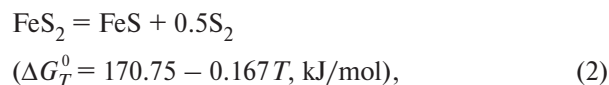
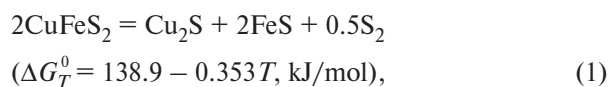


Fig. 2. Content of non-ferrous metals (a), arsenic and antimony (b) in slag and smelt products yield (c) dependence of the concentrate consumption:  
1 — lead bullion; 2 — matte; 3 — slag

analysis of interaction reactions between the components of converter slag and copper-zinc concentrates.

Results of mineralogical investigations and X-ray diffraction analysis of assays of converter slags of the 1<sup>st</sup> period of converting process has showed that copper in slags is for the most part represented in oxide form [1]. Lead and zinc in slag is found out in the silicate part as their oxides bound with silica. The most part of iron is represented by fayalite ( $2\text{FeO} \cdot \text{SiO}_2$ ). Marginal iron rate (under 4%) is detected in the form of magnetite. Arsenic and antimony present in slag in a pentavalent form  $\text{As}^{5+}$ ,  $\text{Sb}^{5+}$  or  $\text{As}_2\text{O}_5$  and  $\text{Sb}_2\text{O}_5$ . The obtained results are strengthened by the data of [18, 19].

Taking into consideration the established forms of non-ferrous metals, arsenic and antimony presence, chemism of the converter slag and copper-zinc concentrate components interaction can be described by the system of the following reactions:



On charging a fresh portion of concentrate, the matte forming and its separation into a self-dependent phase takes place at the expense of chalcocite ( $\text{Cu}_2\text{S}$ ) and copper sulfide, arising as a result of chalcopyrite dissociation by the reaction (1). An addition volume of copper sulfide obtained as a result of reaction (3), converts to a matte phase thus increasing its yield. Iron sulfide forming as a result of dissociation of the principal sulfides, which enter with the concentrate according to the reactions (1), (2), is additionally spent for magnetite reduction in accordance with the reaction (4). Due to the reactions (3), (4) the copper content in slag is reducing, which increases total copper recovery to matte. As this takes place, an excessive amount of iron sulfide which doesn't participate in the reactions under consideration, is transiting to matte. Such mechanism is confirmed by results of the fulfilled experiments. The Fig. 3 illustrates how content of metals in matte depends on concentrate consumption. As indicated in the diagram, it is possible to obtain the mattes reach in copper (up to 50%) and with minimal content of iron sulfide in the process of reducing and sulfidizing slag depletion with different concentrate consumption.

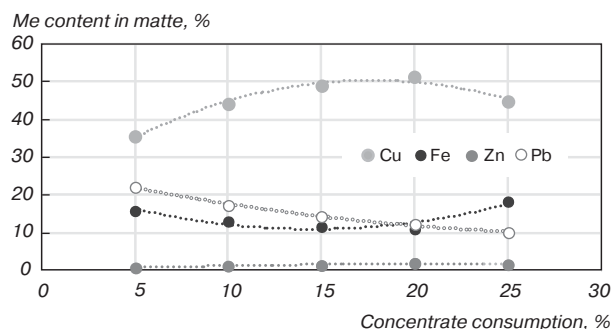


Fig. 3. The non-ferrous metals content in matte dependence of the concentrate consumption:  
1 — Cu; 2 — Fe; 3 — Zn; 4 — Pb



The following established fact seems to be of a certain interest. In an interval of the concentrate consumption growth from 5% to 20% copper content in matte is growing and is at the high at the concentrate consumption of 20%. Further increase of the concentrate consumption leads to a smooth lowering of the copper content in matte (Fig. 3). Such effect can be interpreted based on the following considerations. Copper dilution in lead bullion should be expected as matte is contacting with lead bullion [2, 20]; at that, copper transition from matte to lead bullion is growing with increase of the copper content in slag. One can easily see it in Fig. 4, which illustrates the copper content in lead bullion dependence of the concentrate consumption. Character of the curve with a slight growth of the copper content in matter at the segment of the concentrate consumption from 5% to ~20%, repeats path of the copper line represented in Fig. 3. At concentrate consumption above 20%, growth of the copper content in lead bullion is much more pronounced, which is in complete concordance with the above stated reasoning.

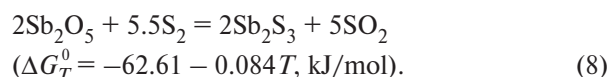
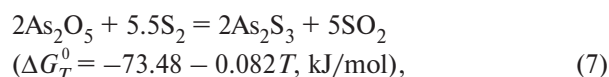
Lead bullion formation may be interpreted in terms of the sequence of reactions (5) и (6). In an initial moment of concentrate charging, the direction of the exchange reaction (5) is shifting towards a lead sulfide formation due to feeding a fresh portion of zinc sulfide to the slag smelting. The resulting lead sulfide interacts with the plumbic ochre according to reaction (6), thus assisting the metal lead bottom phase formation. Rise of concentrate consumption increases volume of a charging zinc sulfide fresh portion; course and shift of the reaction (5) towards a lead sulfide formation will activate the reaction (6). A metallic lead yield will consequently grow, which can be observed in Fig. 2. The described chemism is confirmed by the results of experimental study.

Fig. 5 shows how change of the lead and zinc content in slag depends on quantity of the sulfur bond to zinc sulfide. One can see that raising a zinc sulfide charging to slag leads to increase of zinc oxide content in it with decrease of lead oxide content at the same time. Optimum concentrate consumption corresponds to the value of ~17%, at which the maximum zinc oxide content in slag along with the minimum lead content in it are achieved (point of lines intersection in Fig. 5). Further increase of a lead sulfide ratio leads to a slag yield growth due to the additional iron bringing in with slag. Quantity of a lead sulfide required for the maximum lead oxide sulfidizing may be calculated according to the stehiometry of reactions (5) and (6). In that case, one should expect rise of lead sulfide content in matte and sulfurous anhydride essential excretion, which is undesirable in the view of practice.

Slag content should correspond to the conditions of ZnO maximum solubility along with its fusibility keeping [21]. In case of an excessive ZnO accumulation in slags, it is necessary to correct their content for providing fusibility and fluidity, correspondingly. Under

conditions of co-smelting of converter slag and copper-zinc concentrate, slag content adjustment is carried out by trial of the optimum copper-zinc concentrate consumption. Choice of the optimum slag content  $\text{PbO} - \text{ZnO} - \text{FeO} - \text{CaO} - \text{SiO}_2$  is very important with relation to both guaranteeing the minimal copper concentration in slag [22] and the easily melted zinc-containing slag with the maximum ZnO solubility in it [21]. Execution of these conditions during sulfidizing depletion of converter slag is reached by charging optimum concentrate volume.

Study of arsenic and antimony behavior is of great interest with relation to a process-ecological aspect. On charging a fresh portion of the copper-zinc concentrate, an excess sulfur arising in accordance with reactions (1) and (2) will be completely used up for arsenic oxide and antimony sulfidizing:



In experiments under the conditions of high temperature (1573 K) the Gibbs free energy values for reactions (7) and (8) amount to :  $\Delta G_{1573\text{K}} = -862.40 \text{ kJ/mol}$  and  $\Delta G_{1573\text{K}} = -746.9 \text{ kJ/mol}$  respectively, that is

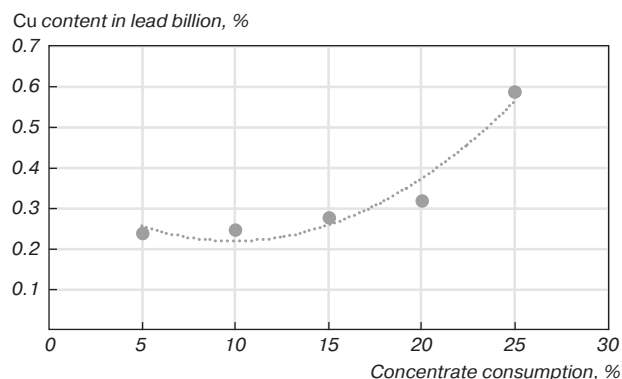


Fig. 4. The copper content in lead bullion dependence of the concentrate consumption

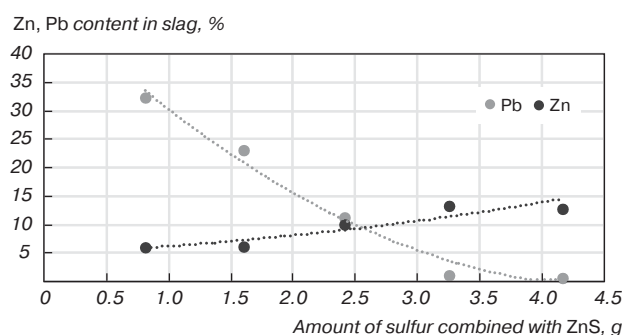


Fig. 5. Pb and Zn content in slag dependence of zinc sulfide

Table 2

**Equilibrium composition of the smelting products determined in the process of slag sulfidizing by concentrate (concentrate consumption 20%)**

Product	Content of components, % (wt.)								
	Cu	Zn	Pb	Fe	S	As	Sb	SiO <sub>2</sub>	CaO
Depleted slag	0.31	11.35	1.15	28.70	N.d.*	0.51	0.32	29.90	3.33
Matte	51.35	1.93	12.26	10.95	22.03	0.92	0.09	–	–
Lead bullion	0.32	–	98.50	–	N.d.	0.13	0.09	–	–
Dust	0.03	8.24	0.27	0.62	N.d.	22.7	9.28	0.58	0.24

\* N.d. — not defined.

Table 3

**Arsenic, antimony and non-ferrous metals distribution between smelting products (concentrate consumption 20%)**

Product	Distribution of metals, %				
	As	Sb	Cu	Pb	Zn
Slag	11.52	17.17	2.47	1.81	86.32
Matte	5.55	1.35	96.42	5.11	3.88
Lead bullion	1.71	2.69	1.06	93.01	–
Dust	81.22	78.79	0.04	0.07	9.80

almost 2 times greater than the values of the Gibbs free energy of the possible interaction reactions of non-ferrous metals and ferric oxides with elemental sulfur. High values of the Gibbs free energy for reactions (7) and (8) evidence deep arsenic and antimony sublimation to dust in the form of non-toxic sulfides  $As_2S_3$  and  $Sb_2S_3$ . That in its turn guarantees significant arsenic and antimony withdrawal out of the engineering process.

Table 2 contains resulting equilibrium compositions of smelting products, determined in the process of slag sulfidizing with the optimum consumption of copper-zinc concentrate.

Dust quantity was determined by the difference between the sums of non-ferrous metals, arsenic and antimony in an origin charge and obtained melting products.

Cu, Pb, Zn, As and Sb distribution among the melting products is shown in Table 3.

Non-ferrous metals extraction to the target products amounts to 96% of copper to matte, 91.8% of lead to lead billion, 86.5% of zinc to slag. Arsenic and antimony extraction to dust is 82.3% and 78.5%, respectively.

Hence, the fundamental difference of such an approach from the preceding ones lies in the fact that the proposed process of slag depletion with a copper-zinc concentrate guarantees high non-ferrous metals extraction to target products out of a collective difficult-to-process copper-zinc concentrate by its direct smelting at one stage. On the other hand, simultaneous use of the sour concentrate as a sulfidizing agent provides favorable

conditions for significant arsenic and antimony withdrawing out of the engineering process due to their maximum extraction to dust in the form of non-toxic volatile sulfides  $As_2S_3$  and  $Sb_2S_3$ .

## Conclusions

1. On the grounds of the experimental study, it has been shown the feasibility of the converter slag depletion by the sour copper-zinc concentrate and the regularities of arsenic, antimony and non-ferrous metals distribution among the smelting products has been determined.

2. It has been established that at the optimum concentrate consumption of 20%, high impurities extraction to dust can be achieved: up to 83% of arsenic and up to 80% of antimony. As this take place, the non-ferrous metals extraction to the target products amounts to 96% of copper to matte, 92% of lead to lead billion, 87% of zinc to slag.

3. The obtained data are used for selecting an optimum charge composition for the blast concentrating smelting in case of semiproducts and reverts processing in combination with the sour copper-zinc concentrate.

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