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COMPARATIVE ANALYSIS OF ZINC AND TIN OXIDATION
WITH ACIDS AT ROOM TEMPERATURES

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The paper analyses the parameters of deep oxidation of zinc and tin by copper (II) compounds and molecular iodine in the presence of molecular oxygen and hydrogen peroxide, as second oxidizers working synchronously with them in various media (aqueous, aqueous-organic and organic) with the participation of mineral and carboxylic acids close to room temperature. The contribution of the reactivity of the metal to its rate of consumption, especially the average, is often much less than the contribution associated with the release characteristics of the working surface, determined by the rates of accumulation of surface deposits of metal oxidation products, their strength and adhesion characteristics their destruction and shifting into the bulk phase.
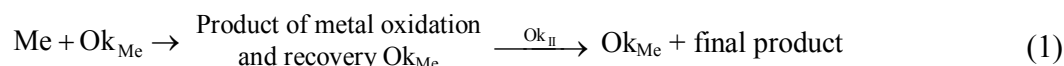
Key words: zinc; tin; oxidation; process characteristics; comparison; reactivity; manifestation; surface blocking; transformation products; reasons for commensurate speeds

Introduction. Zinc and tin are among the most important coating metals for protecting iron from corrosion [3, 5, 6, 9, 12, 13, 15-20, 23-28]. At the same time, in terms of electrochemistry zinc is a more active reducing agent (for zinc, $E^0 = +0.7628$V, and for tin, $E^0 = -0.136$ V [10-12, 14]). In galvanic series zinc is to the left of tin. Zinc belongs to metals of constant valence, and tin, on the contrary, to metals of variable valence. Zinc is quite easily oxidized in humid air to form an oxide film. With respect to air and water, tin at a normal temperature is more stable, which does not exclude the formation of an oxide film [7, 8]. Therefore, when using the considered metals as reducing agents, the presence of oxide films on their surfaces should be considered, which can affect the characteristics of the redox processes carried out [7, 26, 28]. It could be expected that the degree of manifestation of the effects indicated will depend on such factors as the nature of oxidizers and metals in contact with the metal, the nature and content of the latter acids, the presence of salt additives, concentration ratios and other conditions of the processes. This work is devoted to assessing the influence of a number of these factors.

To obtain comparative characteristics, the following processes were chosen as model ones: the oxidation of metals with copper compounds (II) in water-salt solutions and practically non-watered organic media, as well as oxidation with hydrogen peroxide in watered organic media with and without the presence of an independent liquid phase the initial reaction mixture based on water-insoluble (and practically water-free) organic solvent with aqueous solutions of hydrogen peroxide and monobasic mineral acid.

The selected processes fall into the category of rapid and large-scale, even quantitative, destruction of the metal. They proceed with the participation of two oxidizers (Ok_{Me} – metal oxidant, Ok_{II} – the second oxidizer for regeneration) according to the following scheme

$$Me + Ok_{Me} \rightarrow \begin{matrix} \text{Product of metal oxidation} \\ \text{and recovery } Ok_{Me} \end{matrix} \xrightarrow{Ok_{II}} Ok_{Me} + \text{final product} \qquad (1)$$

with the participation of acids (mineral and carboxylic) and other acid reagents in the macrocyclic stage with 10-30 cycles and many cycles during the process [21]. The strength of the acid is of no fundamental importance. In many cases, with the participation of weak and even very weak acidic reagents, the indicated processes proceed more quickly and more deeply in comparison with analogues with strong and even the most powerful mineral acids [22].

Results and its discussion. The processes of the comparative experiment were carried out in accordance with the process flowchart (Fig. 1).
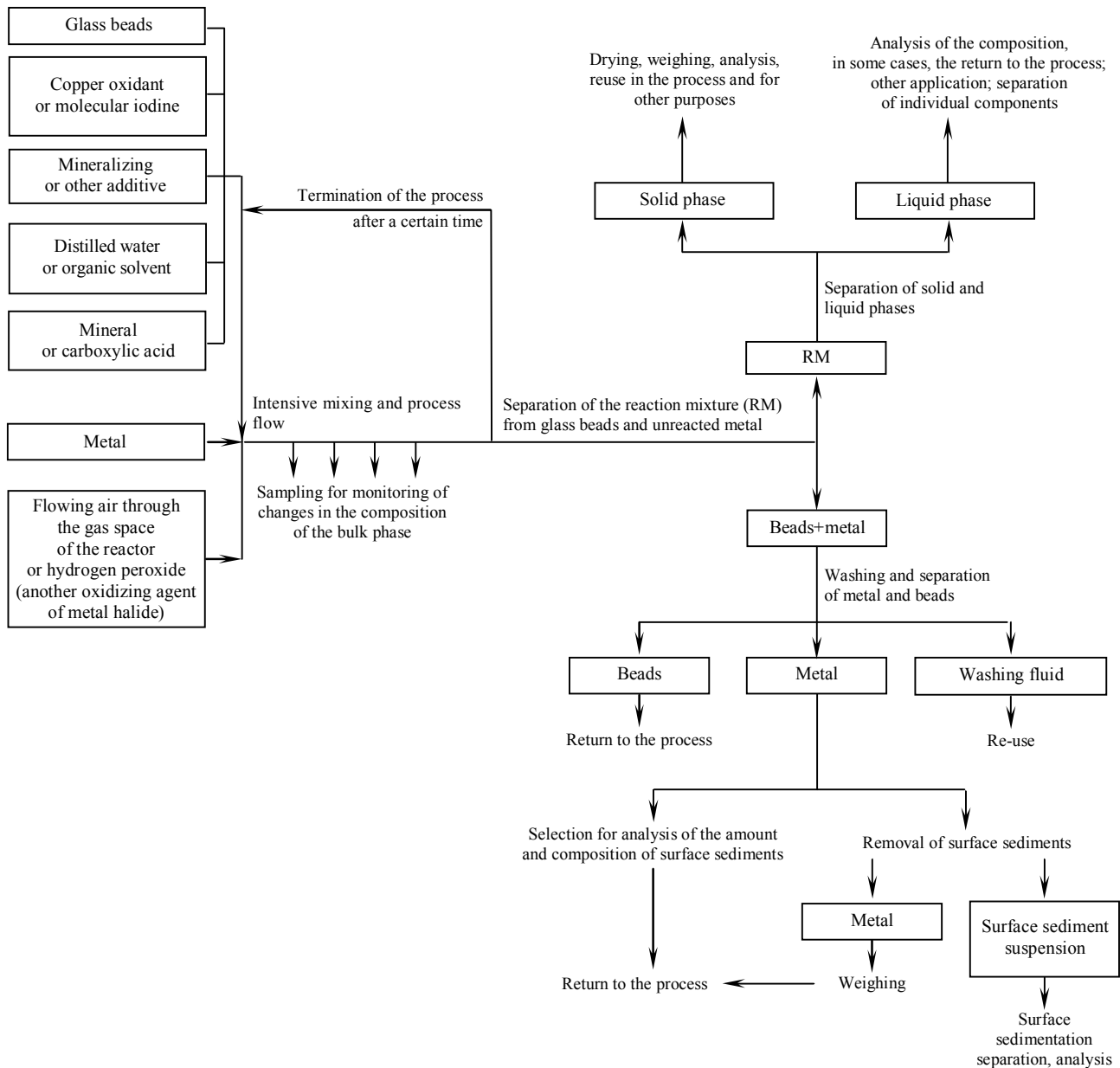
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Fig.1. General flowchart of oxidation of metals in various media

As a base reactor we used a vertical-type bead mill with glass or plastic housing with an inlet of 45-60 mm and a ratio of height to diameter of 2: 2.5 per charge of the grinding agent and the reaction mixture in a mass ratio of 1: 1 (100-150 g without metal mass). In the course of the process, sampling of the reaction mixture is provided without stopping intensive mechanical stirring and analyzing them for the content of the reagent, product and oxidant in the reaction mixture. The mixing was carried out using a paddle-type mechanical stirrer (1440-3000 rp/m). The width of the blade is 2-4 mm less than the inlet of the reactor, and the lower edge did not reach the bottom of the case by 0.3-0.8 mm.

As the reactor, we used a tank with a flat bottom with a diameter of up to 100 mm and more, a ratio of height to diameter of 0.3: 0.5. Mixing was carried out with a mechanical shaker, and the gas space is connected to the measuring system of a volumetric unit. According to the characteristics of the process carried out, it was often (somewhat noticeably) inferior to a bead mill, but it allowed tracking the accumulation of gaseous product along the way, as well as during breaks, which varied not only for hours but also for days, weeks and more. In this case, in such a reactor, you can use a milling agent, which is very important for the indicated processes.

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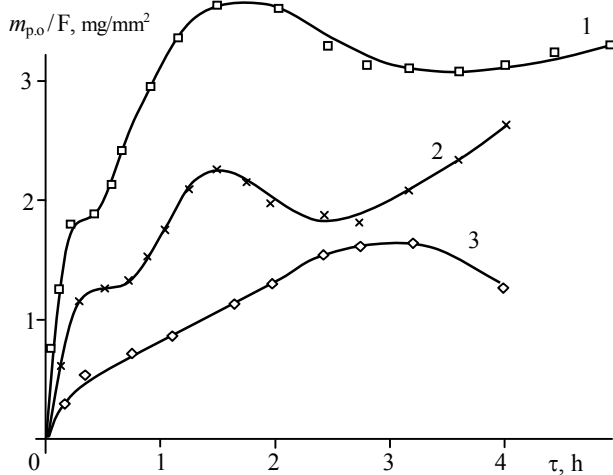
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Fig.2. Dynamics of sediment products accumulation on zinc (1,2) and tin (3) with the participation of formic acid without malachite (1) and with malachite as an oxidizing agent in the amount of 10^{-1} mol/kg (2, 3) at room temperature

It was experimentally confirmed that neither zinc nor tin were crushed under the influence of used grinding agents, and the agents were not destroyed or consumed. Consequently, their main role was reduced to mechanical impacts on the surface of solid reagents in the form of impacts, scraping, abrasion, scratching, etc.

The separation of the grinding agent and unreacted metal from the reaction mixture took place on a grid with 0.3-0.3 mm cells, which served as a filter diaphragm. It did not filter the solid phase of the product, which greatly facilitated the separation of the metal. The solid phase of the sedimented product was subsequently separated by filtration, the precipitate was washed on a filter and dried in air. The filtrate and washing solvent were analyzed for the content of oxidizing agents, acids, products, accumulated and used in downloads of repeated processes, including special-purpose ones.

In liquid and substantially watered organic media, the consumption of metal must take place both with and without the oxidizing agent. The first is the direct interaction of the metal with the acid to form hydrogen as the product. Analyzing the selection of the latter, one can judge how the indicated interaction proceeds. In the overwhelming majority of cases, it is ahead of the development of metal consumption with the participation of the oxidizer and at the initial stage proceeds at higher speeds, but not too long, since the mode of such flow is a rapidly progressing self-stifling, terminated either by self-stopping of this process by a certain (sometimes rather long) time with the subsequent renewal and flow at very low speeds, or going to such speeds immediately, bypassing the self-stopping stage.

It was experimentally confirmed that these phenomena are associated with the blocking of the metal surface by sedimentation of products (Fig.2).

Indeed, if during the period of self-stopping the metal with surface sediments is removed from the reaction mixture, cleaned of them, then returned to the same reaction mixture and the process continues, the kinetics of hydrogen accumulation will repeat not only qualitatively but also almost quantitatively. At the same time, the degree of metal conversion to self-stopping, even in the case of zinc, never exceeded 5 %.

Volumetric measurements made it possible to evaluate the characteristics of the indicated interaction as parallel with the oxidation of the metal in acids. Interestingly, in this combination, it often proceeded even more quickly. This is because the blocking of metals by surface sediments of the products of their interaction with an oxidizing agent is weaker, which ensures a freer access of the acid to the metal surface.

With an oxidizing agent, the density of surface sediments on zinc is less than without it, which is due to some degree to the change in their nature. Without an oxidizing agent, the first product was a medium salt, which underwent hydrolysis, the equilibrium of which was constantly shifted due to the expenditure of the formed acid with the metal, i.e. without the release of the latter from the near-surface layer into the bulk phase with subsequent adsorption on the free surface. With the participation of the oxidizing agent, the main salt immediately dominated, which predetermined significant differences in the composition of surface sediments and, consequently, in their properties.

With a similar oxidation of tin, its molecular weight is almost 2 times the molecular weight of zinc. Consequently, with commensurate mass amounts of sediments per unit of surface, in the case of tin, when converted to moles, they will be significantly less. Most likely, this is the reason

why oxidation of tin proceeds not only with comparable average speeds, but often significantly higher, and with simpler kinetics. In any case, the process is accompanied by a smaller number of self-stopping and its almost complete absence, with metal conversion levels up to 50-60 % of the oxidation process achieved under specific conditions.

In a strongly stifled process, these interactions occur even during periods of forced and deliberate interruptions, even if they last for weeks or months, and without any mixing whatsoever. The amount of reacted metal in such periods can reach tens of percent (in both processes, with and without an oxidizing agent). In general, it can be stated that the contribution of direct interaction of zinc with acid in the quantitative expenditure of the metal by reagent in the deficiency is 15 %, and tin – 3-5 %. These data correlate well with the higher reactivity of zinc as a reducing agent noted above.

The consumption of metal with the participation of the oxidizer occurs according to the stoichiometric equations:
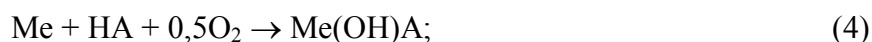
1) with a stoichiometric amount and an excess of acid on the reacted metal

$$\text{Me} + 2\text{HA} + 0{,}5\text{O}_2 \rightarrow \text{MeA}_2 + \text{H}_2\text{O} \tag{2}$$
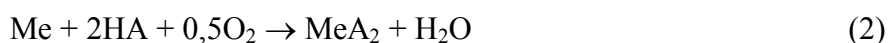
followed by hydrolysis of the middle salt and partial return of the consumed acid

$$\text{MeA}_2 + \text{H}_2\text{O} \rightarrow \text{Me(OH)A} + \text{HA}; \tag{3}$$
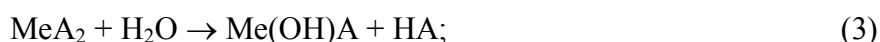
2) with a moderate lack of acid introduced at the beginning to the metal that actually reacted

$$\text{Me} + \text{HA} + 0{,}5\text{O}_2 \rightarrow \text{Me(OH)A}; \tag{4}$$

3) with a very large (5-30 times or more) lack of acid in the initial load

$$\text{Me} + \text{H}_2\text{O} + 0{,}5\text{O}_2 \rightarrow \text{Me(OH)}_2. \tag{5}$$
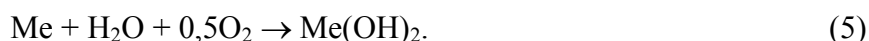
In the oxidation of zinc with copper (II) compounds, all three variants are realized. Moreover, a characteristic feature of the third variant is the transition of the pH in the process zone to the alkaline region (see table).

The table shows that such a transition is observed when using a rather large range of oxidizing agents with different acids, their disadvantages on the reacted metal, at different metal loads with respect to the remaining mass of the reaction mixture and the degree of metal consumption. However, the obligatory presence of ammonium nitrate as a salt additive, which in selected conditions was destroyed:

$$\text{NH}_4\text{NO}_3 + \text{H}_2\text{O} \rightleftarrows \text{NH}_4\text{OH} + \text{HNO}_3,$$
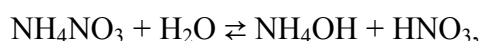
supplying a certain amount of acid as a reagent for the main process and transferring the pH of the bulk phase to an alkaline region with a pronounced ammonia odor [1]. Other ammonium salts in the selected conditions did not possess this ability. But with other metals, it manifested itself clearly [4].

Near the transition of the bulk phase to the alkaline region, close to the maximum metal consumption rate was observed, which indicated the most favorable conditions for the process under consideration during this period. The type of kinetic curve $\Delta m_{zn}/m_0 = f(\tau)$ up to this point was different: from monotonously increasing to wavy with steps and with a clearly defined induction period.

When tin is oxidized under similar conditions, the pH of the reaction mixture grows more slowly and never appears in the alkaline region under the studied conditions. Therefore, the main direction in the lack of acid is the interaction (4) with the formation of the main salt as a product. Tin hydroxide and oxide are most likely the products of hydrolysis of the basic salt in $\text{SnO} \cdot x\text{H}_2\text{O}$ and the loss of water last in the presence of the basic salt [2].

Copper plating of zinc under certain conditions of oxidation is associated with a higher reactivity of the metal, which is not typical of tin. The functioning of the oxidizing agent in the conventional mode corresponds to the scheme (HA – acid)

$$\text{Cu}^{2+} \underset{+\text{O}_2,\ \text{HA}}{\overset{+\text{Me}}{\rightleftarrows}} \text{Cu}^{1+}. \tag{6}$$

**Variants of movement of the final reaction mixture to the alkaline region during oxidation of zinc
(33 % of the rest of the load) in an aqueous solution of 0.04 mol/kg acid and 0.145 mol/kg ammonium nitrate with copper
compounds (II) (2×10^{-2} mol/kg) at room temperature**

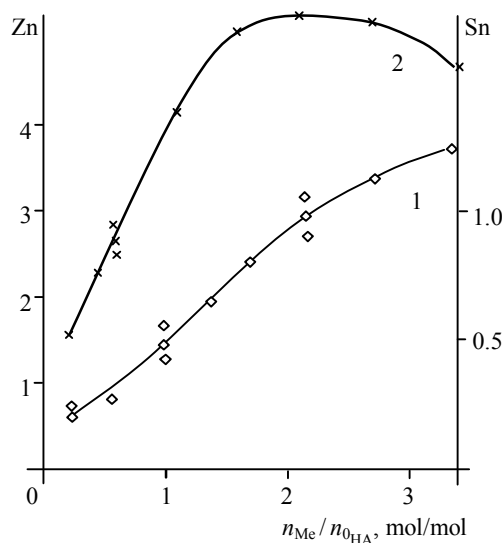| Metal oxidation with copper compounds	HA – acid	Degree of conversion of zinc, %	$\Delta n_{Zn} / n_{0_{Cu^{2+}}}$, mol/mol	$\Delta n_{Zn} / n_{0_{HA}}$, mol/mol	pH of the final reaction mixture	$m_{p.o}$ (g/kg) on the remaining metal
$CuCO_3 \cdot Cu(OH)_2$	HF	45.2	10.6	5.3	10.13	63.3
	HCOOH	48.3	11.4	5.6	9.93	45.7
	CH_3COOH	33.1	7.8	3.9	9.41	32.3
	NH_2CH_2COOH	37.8	8.9	4.4	9.65	40.1
CuF_2	HCOOH	27.3	6.4	3.2	9.33	28.7
$Cu(OCOCH_3)_2$		44.7	10.5	5.2	9.78	19.3
$Cu(OCOCH_2NH_2)_2$		39.3	9.2	4.6	10.03	17.7
$Cu(OH)Br$	HF	55.4	13.0	6.5	10.25	59.3
	HCOOH	61.2	14.4	7.2	10.16	64.7
K_2CuBr_4	HF	60.3	14.2	7.1	10.44	72.8
	HCOOH	69.4	16.3	8.2	10.53	79.6
$Cu(OH)NO_3$	HCOOH	27.3	6.4	3.2	9.12	8.8
$CuCl_2 \cdot 3Cu(OH)_2$		37.4	8.8	4.4	9.05	33.7
$Cu(OH)_2$		31.5	7.4	3.7	9.14	16.7
CuO		28.4	6.7	3.3	8.59	10.2
Cu_2O		32.3	7.3	3.6	8.71	14.2
$CuCl$		34.7	8.2	4.1	9.81	48.6
$Cu(NO_3)_2$		31.8	7.5	3.7	8.84	34.5
$CuSO_4$		26.3	6.2	3.1	8.53	23.7
Acid copper oxidation products:	HCOOH					
hydrochloric		29.8	7.0	3.5	8.9	19.7
benzoic		44.6	10.5	5.2	9.6	38.9
salicylic		39.9	9.4	4.7	9.7	61.2
n-aminobenzoic		47.7	11.2	5.6	9.4	54.5
cinnamic		43.5	10.2	5.6	9.9	47.3
hydrocinnamic		32.8	7.7	3.8	8.5	23.8

Fig.3. Dependency of relation $n_{Me} / n_{0_{HA}}$ of reacted zinc (1) and tin (2) on from the molar ratio of metal to formic acid in the initial charge during the oxidation of metals by copper (II) compounds (0.01 mol/kg in the water-salt bulk phase $(X_{0_{NH_4NO_3}} = 0.17$ mol/kg)

1 – copper nitrate (II); τ = 180 min; 2 – malachite; τ = 150 min

In the case of zinc as an oxidizing agent, the reduction of Cu^{2+} to Cu^{1+} could take place on a large scale, i.e. up to metal, which led to the actual loss of part of the oxidant at some stage of the development process. However, the process is not irreversible. Copper can oxidize Cu^{2+} to Cu^{1+} compounds and further, according to the above scheme, return Cu^{2+} compounds as the most effective oxidizing agents [21].

Thus, in the oxidation of selected metals in the liquid phase at room temperature in a vertical-type bead mill with high-speed (1560 rp/m) paddle stirrer (glass beads as a milling agent) copper compounds (II) with a significant lack of acid composition of products for zinc and tin will be different: $Zn(OH)_2$ dominates for zinc, Sn(OH)A for tin. Consequently, the oxidation of zinc allows a greater lack of acid in the initial charge, unlike tin. When the maximum of the acid deficiency is exceeded, the tin is oxidized to a certain limit. For zinc (for a fixed time per mole of acid loaded), the amount of metal reacted to the acid loaded at first will increase (Fig. 3). The time spent on this will also increase, but to a much lesser extent than when tin is used up to an expediently acceptable acid deficiency.

The process under consideration proceeds with a certain Ok_{Me} deficiency in stoichiometry 10-25 times less than the estimated metal consumption, bringing the dosage of this component to catalytic amounts. This directly follows from the regeneration of this oxidizing agent according to flowchart (1). In the case of copper (II) compounds, the latter process is to some extent accompanied by the replacement of the anion of the initial variant with the anion of the acid present in the reaction mixture. However, this may be accompanied by a substantial increase in the efficiency of such an oxidizing agent (with the initial CuO; $Cu(OH)_2$; $CuCO_3 \cdot Cu(OH)_2$ etc. always occurs). At the same time, the loss of such efficiency in the course of further process no longer occurs. This is confirmed by the fact that copper-containing products released from the reaction mixtures are strong metal oxidizers and can be used both during the same process and in the oxidation of its products to tin (IV) compounds (Fig.4, the oxidation products of the metal with malachite as the primary oxidizer of the metal), and in much smaller quantities than during the initial use of individual copper (II) compounds.

In case of oxidation of metals in liquid media with the participation of acids and oxidizing agents associated with the position of metals in galvanic series, no advantages were found. On the contrary, the processes with zinc and in terms of average speed and duration are quite often inferior to similar proc-
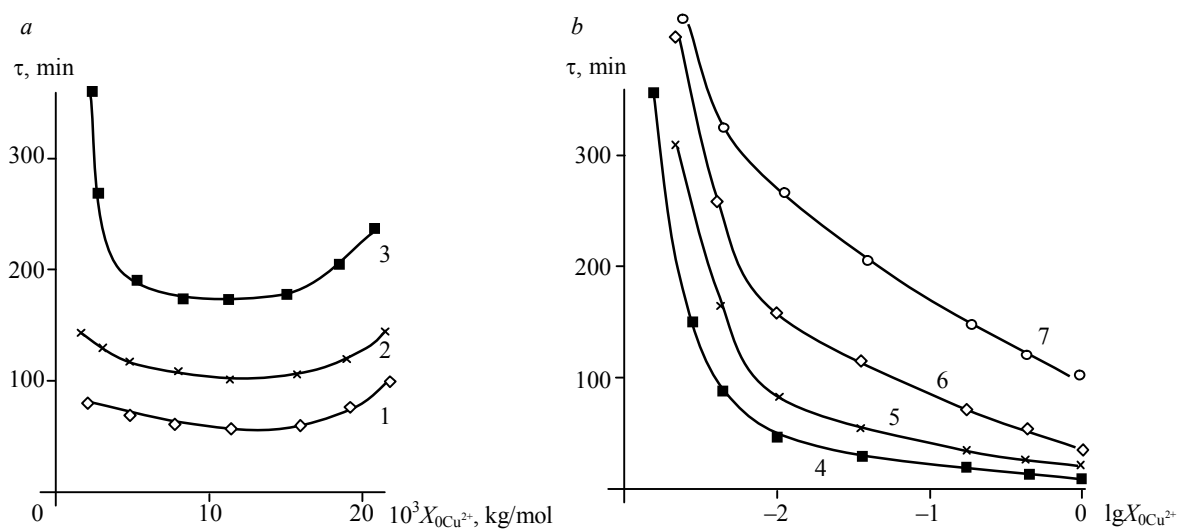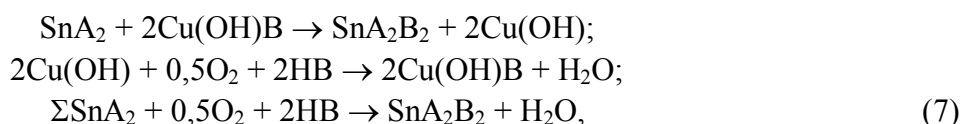

Fig.4. Passage time to reach 0.25(1); 0.50(2) and 0.90(3) degree of transformation of tin in water-salt ($X_{0_{NH_4NO_3}}$ = 0.2 mol/kg)

in solution of formic acid (*a*) and 0.25(4); 0.50(5), 0.75(6) and 0.98(7) degree of transformation of tin hydroxide (II) in white spirit (*b*) when they are oxidized with copper (II) compounds, depending on the content of copper compounds (II) in the initial loading process

esses with tin. This is largely because the blocking of the zinc surface was much stronger, more often it was complete, the yield was longer, and during the process these phenomena began in the early stages and repeated with a high frequency. Even though local rates of zinc oxidation were often higher (sometimes significantly), they were leveled by a longer duration and number of intermediate self-stopping. In other words, blocking the surface of tin with deposits of products turned out to be significantly more favorable than in the case of zinc.

The blocking of the surface correlates well with the fact that the interrupted oxidation of tin by stopping the stirring and mechanical effects on the metal surface continues to proceed, although at a slower (50-200 times) rate than before the stopping of the stirring, but several times and sometimes tens of times more than the rate of zinc consumption in similar situations. Moreover, in the absence of mixing, the self-terminating process can resume and proceed with the achievement of metal consumption in quantities not only commensurate, but also exceeding those achieved until the process is interrupted. It was not possible to identify similar phenomena with zinc.

With the exclusion of water from the reaction mixture, the oxidation of metals with copper (II) compounds proceeded almost entirely by reaction (2). This required an increase in the initial acid loading to stoichiometric amounts. In other words, the selectivity of the redox process increased. In this case, it is necessary to consider the fact that in the case of tin, SnA_2 was not a final but an intermediate product, i.e. can oxidize to tin (IV) salt:

$$SnA_2 + 2Cu(OH)B \rightarrow SnA_2B_2 + 2Cu(OH);$$
$$2Cu(OH) + 0,5O_2 + 2HB \rightarrow 2Cu(OH)B + H_2O;$$
$$\Sigma SnA_2 + 0,5O_2 + 2HB \rightarrow SnA_2B_2 + H_2O, \tag{7}$$

where A^- and B^- – anions of acids HA and HB. In this case, the content of tin (II) and (IV) salts in the final reaction mixture will substantially depend on the nature of the acid and solvent of the liquid phase.

The use of molecular iodine as Ok_{Me} has significantly expanded the range of Ok_{II} (these are metal oxides with higher valence, metal peroxides, organic peroxides, etc., all oxidizing agents that can react quickly, quantitatively and in strict accordance with stoichiometric equations with MeJ_2, for tin also with SnJ_4). In this range, hydrogen peroxide is of particular interest, the presence of which predetermines the introduction of a certain amount of water into the system, which can be increased by using hydrofluoric, hydrobromic or nitric acid in the form of concentrated solutions. The injected water may be in the form of a solution (for example, in ethyl cellosolve), or it may form an independent liquid phase. In the latter case, it is important that it is not in the dead or stagnant zone of the reactor, which leads to a significant decrease in the process rate (the liquid phase will contain the bulk of the acid and oxidant, there is very weak mixing and transfer of the dissolved components into the organic phase).

In the oxidation of tin, the presence of water in solution or as an independent liquid phase is not of fundamental importance. The kinetic accumulation curve of Sn^{2+} has a typical S-shaped character. The only product is Sn^{2+} compounds. Their oxidation occurs in Sn^{4+}. But under these conditions, Sn^{4+} compounds easily and quickly oxidize the metal to Sn^{2+} compounds:

$$Sn + SnA_4 \xrightarrow{J_2} 2SnA_2, \tag{8}$$

predetermining that Sn^{4+} is an intermediate product, and Sn^{2+} compounds are in fact the final product. Complications in the form of self-stifling and self-stopping are observed only at deep stages and can be overcome when the process is terminated, and the reaction mixture stays for a long time.

For zinc the situation is different (Fig.5). The process begins with a maximum speed, but initially proceeds with a rapidly progressing inhibition, up to its cessation for a long, often longer, elapsed time. This can be repeated several times, which ultimately predetermines a lower average metal consumption rate than in the analogous variant with tin.
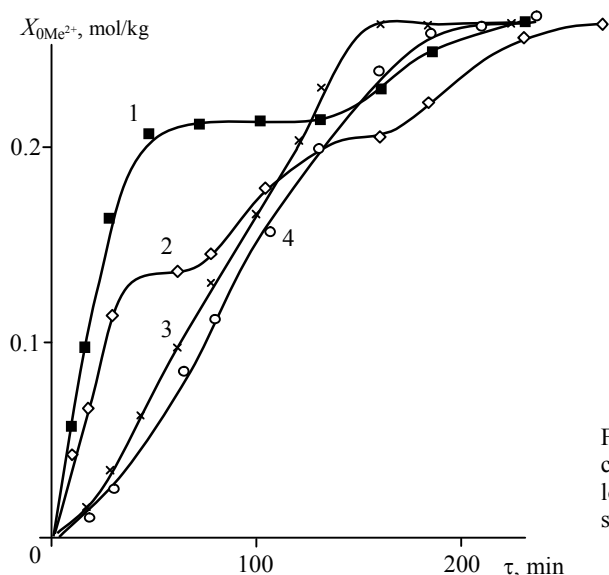
Fig.5. Kinetic curves of the accumulation of zinc (1, 2) and tin (II) (3, 4) compounds in the oxidation of metals (12.5 % of the rest of the initial load) with hydrogen peroxide at 0.05 mol/kg of molecular iodine and stoichiometric amount of nitrogen (1, 3) and benzoic (2, 4) acids in bulk phase based on white spirit at room temperature

Conclusion. In practice, it is possible to detect the manifestation of a higher reactivity of zinc only in certain local variants. In most cases, it is leveled by the specifics of the formation, consumption, and properties of surface deposits of products on the metal, which predetermines the high degrees of blocking of the working surface in time. Therefore, there are quite often cases when the average metal consumption rates and the duration of tin oxidation processes are less than in similar characteristics for zinc.

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