

## Study of Activities Components of Industrial Products and Performed Catalysts under Conditions of Obtaining Ammofos

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### Abstract

The article presents data on the study of the behavior of the components of intermediate products and spent catalysts for the purpose of physicochemical substantiation and development of technology for copper and zinc-containing ammophos using cheap and affordable sources - recycled non-ferrous metallurgy (dust from electrostatic precipitators of the KFP furnace, zinc sublimates) and spent catalysts (NTK-4, zinc acetate). The results of a chemical analysis of the products formed in the ammophos pulp upon neutralization with ammonia are presented. The effect of the degree of neutralization of phosphoric acid by gaseous ammonia on the composition of copper (II) and zinc (II) compounds has been established.

**Keywords:** intermediate products, spent catalysts, physicochemical substantiation, copper and zinc-containing ammophos, recycled non-ferrous metallurgy, dust from electrostatic precipitators of the KFP furnace, zinc sublimates.

**Biology and other natural sciences Subject Classification (2010):**80A32 (primary); 92C45 (secondary).

### INTRODUCTION

According to various researchers, plant nutrition requires between 68 and 84 elements of the periodic system. The role of not all of them has been thoroughly studied. Nevertheless, it is generally recognized that a certain part of the elements found in plants and soil is absolutely necessary for the normal growth and development of plants, obtaining good yields.

All elements involved in the mineral nutrition of plants are usually classified according to their content in plants and in the soil. Usually they are divided into macrocells and microelements. According to this classification, elements whose content in terms of dry matter is from hundredths of a percent to several tens of percent, are macroelements. Those elements whose content does not exceed thousandths of a percent are referred to trace elements.

This classification is currently being supplemented. Some of the elements are now attributed to mesoelements, i.e., in fact, they form a group intermediate between macro- and microelements. In addition, ultramicroelements are sometimes isolated. These are those elements whose content in plants is negligible, and the physiological role and influence are practically not studied.

Basic fertilizers, macro-fertilizers containing trace elements are important to increase agricultural production. Trace elements are necessary for plants in very small quantities - their content is thousandths and ten thousandths of a percent of the mass of plants. However, each of them performs strictly defined functions in metabolism, plant nutrition and cannot be replaced by another element.

When crops are grown on soils with insufficient, and in some biogeochemical provinces with an excessive content of available forms of trace elements, the yield decreases and the

quality of the products deteriorates. Deficiency or excess of certain trace elements in crop production and feed can cause disease in humans and farm animals.

### **PRELIMINARY RESULTS**

The stability of the chemical composition is one of the most important and indispensable conditions for the normal functioning of organisms [1]. Deficiency of vital trace elements (selenium, zinc, iodine, manganese, etc.) in the body and an increased concentration of toxic elements (mercury, lead, arsenic, antimony, cadmium, etc.) in the environment leads to adverse effects on human life [4, 5, 6], in particular, to an increase in the number of oncological, autoimmune, degenerative and other diseases [2], diseases of the lymphatic system, and infectious pathology [3].

Optimization of the mineral nutrition of plants and increasing the efficiency of fertilizers are associated with ensuring a balanced ratio of macro- and microelements. The need for micronutrient fertilizers is also growing due to the use of highly concentrated macronutrient fertilizers, which contain almost no trace elements [7].

It is considered proven that for normal growth and development, trace elements must enter the plants in active form. It has been established that their use in the form of complexonates is most effective. They are in a biologically active form, easily absorbed by plants, do not decompose in soil for a long time, etc. In agriculture, the most widely used are chelates of diethylenetriaminepentaacetic (DTPA), ethylenediaminetetraacetic (EDTA) and hydroxyethylidenediphosphonic (HEDP) acids [8].

ZHUSS preparations are liquid fertilizer compositions in which microelements are in chelated form in the form of an organometallic complex, including inorganic salts  $\text{CuSO}_4$  and  $(\text{NH}_4)_2\text{MoO}_4$  and organic lipid - monoethanolamine (MEA) not only serve as sources of microelements for plants, but also exhibit protective and stimulating effects. In the Republic of Tatarstan, ZhUSS preparations are used on large areas, since they not only increase yield, but also reduce (by 3-4 times) the pesticidal load on the crops of some crops [9].

Recently, one of the most popular innovations in crop production is the use of micronutrients in the form of micronutrients [10] for foliar feeding during the growing season of crops.

Various micronutrient composites for foliar top dressing of crops during the growing season were obtained, containing micronutrients in complex form (copper - 60, zinc - 61 and manganese - 53 g / l) and a polymer adhesive WSP (water-soluble polymer) [11] to prolong the action and uniform distribution of components on the surface of plants and their fixation. These composites are homogeneous solutions, which allows them to be applied to the surface of vegetative plants by spraying. This minimizes the loss of nutrients and necessary components during foliar treatment of plants, and also increases the activity of fungicides. This allows you to reduce consumption rates and get economic and environmental effects. The preparations are compatible with additives, do not exfoliate and do not lose their properties in a wide temperature range from -30 to + 45 ° C [12].

Trace elements play a significant role in the nutrition of plant organisms. In particular, Cu, Mo, Mn, Zn, B increase the activity of enzymes and enzymatic systems in the plant, allow more rational use of macro fertilizers, as well as various nutrients from the soil. Trace elements increase plant resistance to adverse environmental factors, some infectious and non-infectious diseases, increase the rate of development of plant organisms and the maturation of their seeds. Based on the "law of minimum", we can say that the microelement factor is crucial in realizing the potential of cultivated crops [13].

Studies conducted in various soil and climatic zones of Uzbekistan and southern Kazakhstan with different contents of available trace elements compounds in soils showed that when there is a lack of individual trace elements in the soil, the cotton crop does not increase even with high doses of nitrogen-phosphorus fertilizers and other agricultural practices [14.15].

In the conditions of cotton growing in Central Asia, the most effective are trace elements - copper, zinc, cobalt, molybdenum, manganese, which are closely associated with nitrogen-phosphorus metabolism. The use of these trace elements under cotton enhances the intake and conversion of nitrogen and phosphorus into high molecular weight organic compounds, thereby positively affecting the crop [16].

For example, the need for manganese is explained by its participation in redox reactions, during photosynthesis, the influence on the degree of activity of certain enzymes, and the biosynthesis of nucleic acids [17]. Deficiency of this microelement in plant organisms leads to chlorosis, spotting, burns. Manganese deficiency negatively affects growth. As manganese fertilizers, manganese sulfate, manganized superphosphate, manganized nitrophosphate, manganese sludge are used [18]. Manganese sulphate is a fine crystalline dry anhydrous salt, in the composition of which 32.5% of manganese is readily soluble in water. Manganized superphosphate - light gray granules, which contain 1.0-2.0% manganese and 18.7-19.2%  $P_2O_5$ . In addition to nitrogen, phosphorus and potassium, the manganized nitrophosphate contains about 0.9% Mg in its composition. Manganese sludge is a waste of manganese production, manganese is included in the amount of 10-17%. Carbonate blackzem, chestnut and brown semi-desert soils need manganese fertilizers [19]. Positive effects of manganese on calcareous soils are also noted.

Manganese fertilizers are used, introducing them into the soil, during pre-sowing treatment of seeds, as well as for foliar top dressing. Seeds are usually treated with dry dusting. For better adhesion to the seeds, manganese sulfate is dried, crushed and mixed with talc. Foliar top dressing is performed using a weak solution of manganese sulfate.

In crop production, the most commonly used fertilizers are granular borosuperphosphate, double borosuperphosphate, boric acid, magnesium fertilizer, and boracite flour [20]. Granular borosuperphosphate - light gray granules, which contain 1% boric acid. Double borosuperphosphate - 1.5% boric acid. Boric acid is a white powder, in the composition of which 17% of boron is readily soluble in water. Bormagnesium fertilizer is a fine crystalline gray powder, which contains 13% boric acid. Boracite flour (contains about 10% B) is obtained from ground boron ores without processing.

Field experiments have established a positive effect of copper, zinc and molybdenum in the composition of ammophos on the yield and incidence of cotton by wilt [21]. It is shown that trace elements affect the growth, development and accumulation of crops. At the same time, the percentage of sick plants with wilt decreased by 25-27%, and the yield increased to 10-13%. Sick plants found a greater need for zinc and molybdenum than healthy ones [22, 23].

Y.V. Peyve forms of copper contained in soils are divided into the following groups: 1) water-soluble copper in the composition of water-soluble salts 2) exchange copper, absorbed by organic and mineral colloids; 3) sparingly soluble copper salts; 4) copper-containing minerals; 5) organometallic copper compounds (mainly complex). The most mobile is water-soluble copper, the content of which in soils is small (less than 1% of the total), it is represented by chlorides and sulfates, complex organomineral and mineral compounds. Copper forms soluble complexes with humic acids. In addition to water-soluble compounds, copper available in plants is also in an exchange-sorbed state. Copper ions are adsorbed by humic substances, clay minerals, oxides and hydroxides of manganese and iron, as well as silica, most of which are hardly soluble. In this regard, copper is a relatively sedentary element in the soil. Many researchers believe that the largest amount of copper in the soil solution is presented in the form of organic chelate complexes, the value of which reaches 80% of the sum of all water-soluble forms. Many works provide data on the bonds of copper with organic matter. Humic acids and fulvic acids are able to bind from 48 to 160 mg / kg of copper [24-27].

The use of copper, zinc, cobalt and molybdenum with basic fertilizers increases the degree of assimilation by plants of nitrogen by 5-9% and phosphorus by 4-5%, which affects the yield. The increase in raw cotton is from 4.5 to 5 kg / ha. Trace elements under conditions of weak chloride-sulfate salinization contribute to the entry of calcium and magnesium into plants and reduce the intake of sodium and chlorine ions [28].

The possibility of using struvite (magnesium ammonium phosphate is a waste product) as an ameliorant stabilizer of  $\text{Cu}^{2+}$ ,  $\text{Pb}^{2+}$  и  $\text{Zn}^{2+}$  ions in contaminated soil was considered [29].

Today, complex processing of raw materials is very popular not only from a purely economic point of view, but also from the point of view of environmental protection. The relevance of the rational processing of natural reserves is dictated by the need for their economical use in view of their limited content in the bowels of the earth, as well as constantly increasing volumes of modern industrial production, which is associated with environmental pollution.

Recently, it has become increasingly cost-effective to use secondary raw materials and industrial wastes to produce complex fertilizers.

When micronutrients are introduced into the fertilizer as a result of chemical interaction, insoluble, non-digestible forms of nutrition elements can form. On the other hand, the added additives can affect the physicochemical properties of the products. An important problem is the production of trace elements fertilizers using recycled non-ferrous metallurgy, spent catalysts and other waste. For the theoretical justification and development of the technology, deep physical and chemical studies of the behavior of trace elements in the conditions of receipt, storage of fertilizers and its influence on the properties of the products are required.

In connection with the foregoing, the development of physico-chemical bases and technology of complex fertilizers with microelements based on ammophos using recycled non-ferrous metallurgy and spent catalysts is an urgent task.

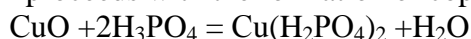
## MAIN RESULTS

We have developed methods for the extraction of trace elements using the main components of ammophos, from the dust of a copper smelter and sublimation of zinc production of JSC "Almalyk MMF". They are technogenic deposits of a unique composition of polymetallic raw materials, which are currently practically not used [30]. In the future, they are planned to be used to obtain nitrogen-phosphorus fertilizers, in particular ammophos. The composition of dust from electrostatic precipitators varies depending on the operating mode of the unit and the operating state of the recovery boiler. According to chemical analysis, dust from electrostatic precipitators contains %: Cu - 18.0; Fe - 17.1; S - 11.4;  $\text{SiO}_2$  2.4;  $\text{Al}_2\text{O}_3$  - 1.5; CaO -1,2; MgO - 0.3 - copper in the dust composition is in the main form of sulfate and oxide (the copper content associated with  $\text{S}^{2-}$  does not exceed 3-5%).

Zinc sublimates are formed in zinc factories in the process of purification of zinc oxide from impurities. Zinc sublimates contain %: Zn - 54.6; Fe - 0.4; Cl - 1.4; F is 0.08; Cd - 0.57. Zinc in the composition of zinc sublimates is in the form of sulfate and oxide.

The behavior of the main components of dust from electrostatic precipitators of an oxygen flare furnace (OFF), zinc sublimation of a low-temperature catalyst (LTC-4), and zinc acetate under conditions of obtaining ammophos was studied [31]. In this case, the effect of the degree of neutralization of phosphoric acid by gaseous ammonia on the composition of copper (II) compounds was established. The studies were carried out on the installation, which is a four-necked glass flask equipped with an electromechanical stirrer, reflux condenser, thermometer and holes for supplying the reaction mixture and sampling. For this, a calculated amount of 32%  $\text{P}_2\text{O}_5$ -phosphoric acid, copper oxide or sulfate (or copper-containing raw material) was introduced into the reaction vessel and stirred at a temperature of 90 ° C until the additive was dissolved. After the additive was dissolved in acid, the mixture was neutralized with gaseous ammonia to a predetermined pH value. After neutralization, the pulp was further mixed for 1 hour. The resulting precipitate was repeatedly washed with water, dried in air, analyzed for the content of nitrogen, phosphorus, copper and subjected to physicochemical methods of investigation.

When copper oxide and sulfate are introduced into thermal phosphoric acid [32], a reaction proceeds with the formation of copper dihydrogen phosphate:





**Table 1. The results of chemical analysis of the solid phase formed in the ammophosic pulp (copper oxide is introduced into phosphoric acid)**

pH pulp	$\text{HPO}_4^{2-}, \%$	$\text{Cu}^{2+}, \%$	$\text{NH}_3, \%$	Molar ratio $\text{HPO}_4^{2-} : \text{CuO} : \text{NH}_3$	Solidphase
1,6	54,3	35,9		1 : 1,00	[Cu(H <sub>2</sub> O)HPO <sub>4</sub> ]
1,8	54,0	35,5		1 : 0,99	
2,0	54,5	36,2		1 : 0,99	
2,5	54,0	36,9	2,1	1 : 1,03 : 0,22	[Cu(H <sub>2</sub> O)HPO <sub>4</sub> ]+ [Cu(NH <sub>3</sub> )(H <sub>2</sub> O)HPO <sub>4</sub> ]
4,3	52,2	35,7	3,4	1 : 1,03 : 0,37	
5,4	51,6	33,6	4,1	1 : 0,98 : 0,45	
6,0	50,2	33,2	6,2	1 : 1,00 : 0,70	
6,3	49,0	32,1	9,0	1 : 0,99 : 1,03	[Cu(NH <sub>3</sub> )(H <sub>2</sub> O)HPO <sub>4</sub> ]
6,8	49,5	32,0	8,8	1 : 0,98 : 1,00	
7,0	48,7	31,8	8,9	1 : 0,99 : 1,03	

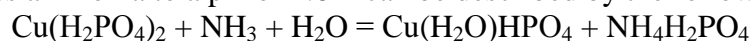
**Table 2. Results of chemical analysis of the solid phase formed in the ammophosic pulp (copper sulfate is introduced into phosphoric acid)**

pH pulp	$\text{HPO}_4^{2-}, \%$	$\text{Cu}^{2+}, \%$	$\text{NH}_3, \%$	Molar ratio $\text{HPO}_4^{2-} : \text{CuO} : \text{NH}_3$	Solidphase
1,7	54,5	35,4	1:0,98		[Cu(H <sub>2</sub> O)HPO <sub>4</sub> ]
2,0	54,1	35,7	1:1,00		
2,7	53,8	35,5	2,8	1:1,00:0,29	[Cu(H <sub>2</sub> O)HPO <sub>4</sub> ]+ [Cu(NH <sub>3</sub> )(H <sub>2</sub> O)HPO <sub>4</sub> ]
4,0	52,0	35,1	3,2	1:1,02:0,35	
4,5	51,2	34,7	3,9	1:1,02:0,43	
5,8	50,05	33,2	6,0	1:0,99:0,67	
6,3	49,8	32,5	9,0	1:0,98:1,02	[Cu(NH <sub>3</sub> )(H <sub>2</sub> O)HPO <sub>4</sub> ]
<b>6,7</b>	<b>49,3</b>	<b>32,5</b>	<b>8,7</b>	<b>1:1,00:1,00</b>	
<b>7,1</b>	<b>49,6</b>	<b>32,0</b>	<b>8,9</b>	<b>1:0,98:1,01</b>	

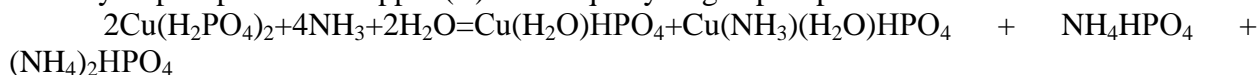
When neutralizing the resulting solution with gaseous ammonia to a pH of 1.6, a change in the phase composition of the solution does not occur.

At pH 1.6-2, the formation of a solid phase of a pale blue color is observed. Chemical analysis of the solid phase obtained at pH 1.6-2 showed the absence of nitrogen and the presence of 35,5-36,2%  $\text{Cu}^{2+}$  and 54,0-54,5%  $\text{HPO}_4^{2-}$ , which theoretically corresponds to the compound  $\text{CuHPO}_4$  (theoretical composition  $\text{Cu}^{2+}$  35,8%,  $\text{HPO}_4^{2-}$  54,1%). The results of chemical analysis are given in tables 1. and 2.

The process that occurs when neutralizing a copper-containing phosphate solution with gaseous ammonia to a pH of 1.6-2 can be described by the following equation:



In the pH range of 2.5-6.0, chemical analysis showed along with  $\text{Cu}^{2+}$  and  $\text{HPO}_4^{2-}$  also the presence of 2.1-6.2%  $\text{NH}_3$ , which indicates the joint precipitation of two salts: copper (II) monohydrophosphate and copper (II) monoammonium hydrogen phosphate:

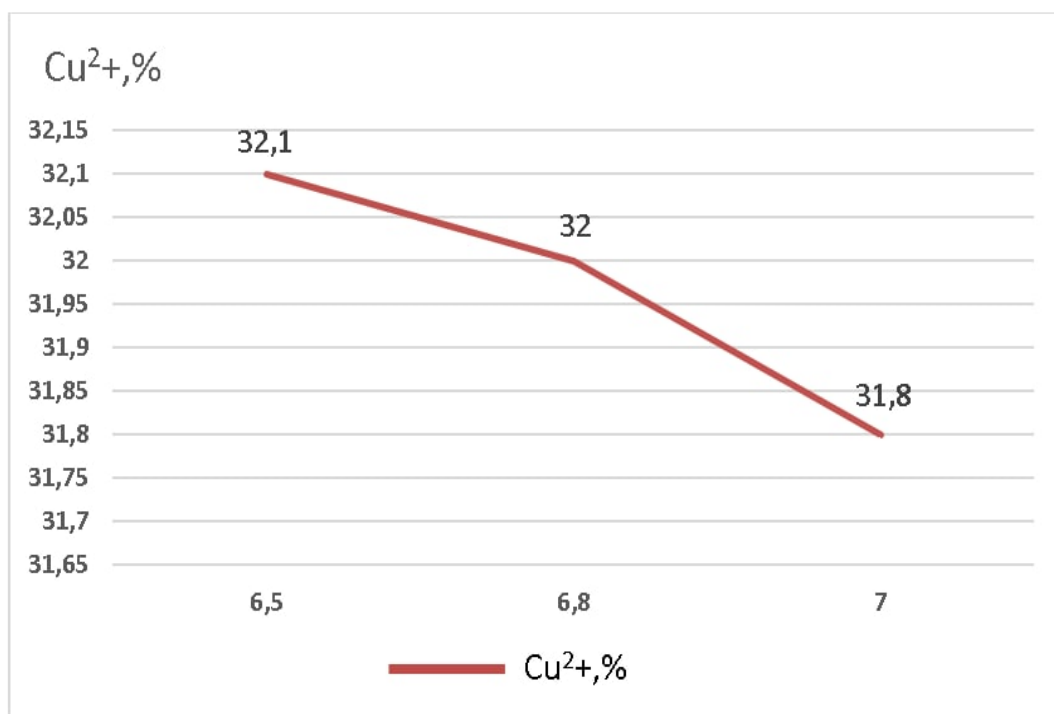


With further neutralization of the copper-containing phosphoric acid solution, the precipitate became intensely blue in color. Chemical analysis of the sediment showed the following components in it:

Ph	$\text{HPO}_4^{2-}$ , %	$\text{Cu}^{2+}$ , %	$\text{NH}_3$ , %
6,5	49,0	32,1	9,0
6,8	49,5	32,0	8,8
7,0	48,7	31,8	8,9

In a molar ratio, this was expressed 1: 1: 1. This indicates the formation with a high degree of neutralization of copper (II) monoammonium hydrophosphate. According to the calculation, this compound contains  $\text{Cu}^{2+}$  32,7%,  $\text{NH}_3$  8,7%,  $\text{HPO}_4^{2-}$  49,3%.

In Fig. 1. the dependence of the concentration of copper in the liquid phase on the pH of the ammonophosic pulp is shown (1% copper is introduced).

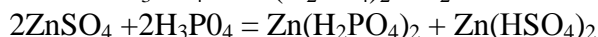
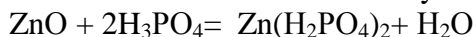


**Fig. 1. The effect of pH on the concentration of copper in the liquid phase ammonophos pulp at 90 ° C**

The behavior of zinc oxide and sulfate, the main components of zinc sublimation, under conditions of obtaining ammonophos, was studied [33–34]. The effect of the degree of neutralization of phosphoric acid by gaseous ammonia on the composition of zinc (II) compounds has been established. For this, a calculated amount of 32%  $\text{P}_2\text{O}_5$  phosphoric acid, oxide, zinc sulfate or zinc-containing raw material was introduced into the vessel and the mixture was stirred at 90 ° C until the additives were dissolved. After neutralizing the mixture with

gaseous ammonia, the precipitate formed was washed with water, dried in air, and analyzed for nitrogen, phosphorus, and zinc.

When zinc oxide and sulfate are dissolved in thermal phosphoric acid, a reaction proceeds with the formation of zinc dihydrogen phosphate:



When neutralizing the resulting solution with gaseous ammonia to pH 3.5, the formation of a precipitate is not observed. With further neutralization, a white precipitate forms. Chemical analysis of the solid phase formed during the neutralization of phosphoric acid containing zinc oxide and sulfate showed the following content of components (Tables 3 and 4):

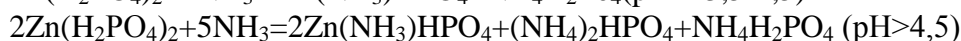
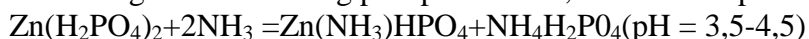
**Table 3. Results of chemical analysis of the solid phase formed in the ammophosic pulp**

pH pulp	HPO <sub>4</sub> <sup>2-</sup> , %	Zn <sup>2+</sup> , %	NH <sub>3</sub> , %	Molar ratio HPO <sub>4</sub> <sup>2-</sup> : ZnO : NH <sub>3</sub>	Solidphase
IntroducedZincoxide					
3,5	52,1	36,1	8,59	1:1,02:1,03	[Zn(NH <sub>3</sub> )HPO <sub>4</sub> ]
3,8	54,1	36,3	9,63	1:0,99:1,01	
4,0	53,2	37,0	9,63	1:1,02:1,02	
4,4	53,5	36,4	9,72	1:1,00:1,03	
5,2	53,6	36,8	9,92	1:1,01:1,05	
5.4	53,9	36,6	9,63	1:1,00:1,01	
Zinc sublimation introduced					
3,6	53,6	36,0	9,60	1:0,99:1,01	[Zn(NH <sub>3</sub> )HPO <sub>4</sub> ] + admixture of iron phosphates
4,2	54,0	35,6	9,52	1:0,97:1,00	
4,5	53,8	36,5	9,70	1:1,00:1,02	
5,8	54,3	36,8	9,92	1:0,94:1,03	

**Table 4. Results of chemical analysis of the solid phase formed in the ammophosic pulp (zinc sulfate is introduced into phosphoric acid)**

pH pulp	$\text{HPO}_4^{2-}$ , %	$\text{Zn}^{2+}$ , %		$\text{NH}_3$ , %	Molar ratio $\text{HPO}_4^{2-} : \text{ZnO} : \text{NH}_3$	Solidphase
3,6	52,8	36,8		9,10	1:1,02:0,97	[ $\text{Zn}(\text{NH}_3)\text{HPO}_4$ ]
4,2	53,4	36,2		9,45	1:1,00:1,00	
4,7	53,0	37,0		9,39	1:1,03:1,00	
5,0	53,6	36,9		9,58	1:1,01:1,01	
5,5	54,1	37,2		9,40	1:1,01:0,98	
6,0	53,8	36,6		9,52	1:0,99:1,00	

Based on the experiments performed and the results obtained, it can be assumed that when neutralizing zinc-containing phosphoric acid, the reaction proceeds:



As can be seen from the results, when zinc oxide and sulfate are introduced into phosphoric acid and the solution is neutralized with gaseous ammonia to a pH of 3.5 - 6.0, zinc monoamine hydrophosphate forms. When zinc sublimation is introduced, the solid phase is zinc monoamine hydrophosphate mixed with iron phosphates.

Ph	HPO <sub>4</sub> <sup>2-</sup> , %	Zn <sup>2+</sup> , %	NH <sub>3</sub> , %
4,4 - 4,7	53,0 - 53,5	36,4 - 37,0	9,39 - 9,72
5,4 - 5,5	53,9 - 54,1	36,6 - 37,2	9,40 - 9,63

In molar ratio, this was expressed as 1: 1.0: 1.03 and 1: 1.0: 1.02. This indicates the formation at pH 3.5-5.8 of zinc monoamine hydrophosphate. According to the calculation, this compound contains, %: Zn<sup>2+</sup> 36,7; HPO<sub>4</sub><sup>2-</sup> 53,8; NH<sub>3</sub> 9,5.

### CONCLUSIONS

Based on the comprehensive physicochemical studies, it was found that in the interaction of the main components of dust with electrostatic precipitators, zinc catalyst and spent LTC -4 catalyst with ammophos pulp at a pH of 1.6-2, copper (II) monohydrophosphate is formed  $[Cu(H_2O)HPO_4]$ . At pH > 2.5, the ammonia molecule is a part of the compound with the formation of copper (II) monoquaaaminehydrophosphate  $[Cu(H_2O)(NH_3)HPO_4]$ . When zinc oxide and sulfate are introduced into phosphoric acid and the solution is neutralized with gaseous ammonia to a pH of 3.5 - 6.0, zinc monoamine hydrophosphate  $[Zn(NH_3)HPO_4]$  is formed. When zinc sublimation is introduced, the solid phase is zinc monoamine hydrophosphate mixed with iron phosphates.

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