

**MINISTRY HIGH AND MIDDLING
SPECIALLY FORMING OF THE
REPUBLIC OF UZBEKSITAN**

(on right manual sample of writing)

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**Improvement physic-chemical and mechanical characteristic of nitric and
complex fertilizers**

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INTRODUCTION

The problem of maintenance of preservation of various granulose materials at their transportation and storage is one of the major in chemical engineering. It is known that separate grains of firm chemical products in most cases represent the is capillary-porous bodies absorbing at storage significant amounts of water from the surrounding aerosphere. It leads, as a rule, essential change of physical and chemical structure of grains and accordingly consumer properties of products therefore they can become unsuitable to practical use.

From physical properties of the granules materials characterizing their stability to affecting of environment, the most important are hygroscopic, consolidation and strength of granules.

The chemical industry releases wide assortment various firm hygroscopic materials in the granules and powdery aspect: mineral fertilizers, soda, chemical products, etc.

Physical properties - hygroscopic, consolidation and strength of granules of mineral fertilizers have great value not only from the point of view of their preservation at transportation and storage, but also for agro chemistry as they define also possibility of uniform application of fertilizers in soil, and their ability with this or that intensity to enter reactions of an ionic exchange with soil components.

The problem of quality of mineral fertilizers has exclusively great value, especially taking into account great volumes and seasonal prevalence of their application in agriculture. At the first stage of development тыковой the industries for improvement of a quality of product first of all were solved problems of reception of fertilizers with the improved chemical compound.

Now there is a necessity of working out of new aspects of fertilizers, creations of production engineering of rehash of new aspects of phosphate raw

materials. At the same time, the problem of mar tempering of physical properties of fertilizers not always dares till now well.

Improvement of physical and chemical and physic mechanical properties of fertilizers is attained usually by a product graining, its deep drying, cooling and inoculation - a covering of granules bath tonic and keeper of moisture additives.

A graining of chemical materials usually fathom such stage of a process which allows to gain a product in the form of grains of certain sizes, forms and strengths. Existing methods of a graining small disperse materials can be divided:

- granulose a way through volatilization in granular tower to a tower;
- granulose a way to pack up on a moving surface in a drum-type grain mill;
- granulose by pressing, graining or an extrusion in wave to a press;
- granulose a way volatilization liquid mashes on a surface of corpuscles in drum-type to a grain mill-dryer or in a fluidized bed.

Making smarm - crystallization and hardening of drops of the melt spluttered in high hollow towers towards to a stream of chilling air. In such a way grain an ammonium nitrate, lime-ammoniacal saltpeter, a carbamide, the calcium nitrate, some aspects of difficult fertilizers.

Graining a method of nebulizing enough widespread fertilizers in production engineering. It use in manufacture of superphosphate, an ammophos and a nitroammophos. Essence of this process that larger corpuscles of a material being rolled on a surface of a powder and sticking together to its corpuscles, constantly grow in volume.

Graining a pressing method use usually in need of reception in the granules aspect of dry powdery materials. Grain potassium chloride, ammonium sulphate, ammonium chloride, phosphor-potash fertilizers.

Formation of granules occurs by spraying by the air-atomizing burner of the mash containing 10-30 % of a moisture, on the dense curtain of a grained material. Mash drops partially parch in flight to a layer of the curtain and adhere to a surface

of corpuscles retouch. Thus granules coarsen and get the spherical form. Further at traffic of granules in a drum there is their final drying.

In Uzbekistan the large industry making mineral fertilizers and chemical means to protection of plants is created. At three production associations of Open Society "Maves-chirchik", "Navoiazot" and "Ferganaazot" make the nitrogenous fertilizers which assortment develops of an ammonium nitrate, a carbamide and ammonium sulphate. Raises quality of mineral fertilizers - to give to agriculture not slumping firm mineral fertilizers in the grained aspect, suitable for a bulk handling in specialized self-unloading carriages, storages without container and preparation dry a solid mineral fertilizer of mixes.

Among principal views of the mineral fertilizers applied in agriculture, the ammonium nitrate possesses mainly propensity to caking. The reason of it is its high hygroscopic and solubility in water, and also the modification transformations proceeding at 32,4°C.

Now Open Society "SAMARQANDKIMYO" releases difficult fertilizer under name NCPHF (nitrofos)-nitro calcium phosphate fertilizer on a basis azotic sourness rehashes high phosphorites Central Kyzylkum. Fertilizer in the core consists from nitrate of calcium and calcium phosphate.

In the literature there is a great number of the published works devoted to improvement of physical and chemical and commodity properties of fertilizers. These works in the core are directed on decrease in hygroscopic of a product, raise of strength of granules, elimination of consolidation of saltpeter, change of temperature of modification transitions IV - III.

Researches on the further improvement of quality of nitrogenous and difficult fertilizers by selection of new aspects and increase in efficiency of known additives are rather actual. Among additives applied now the most economic and perspective for saltpeter improvement of quality is the phosphate additive. It is known that at hanging of quantity of this additive of odes of 1,5-2,0 % in

conversion on P_2O_5 consolidation is completely eliminated and sharply strength of granules raises. But, additive raise at existing production engineering leads to a reinforcement of insolubilize components on internal apparatuses and department communications.

The research objective the Purpose of the present dissertation is agency research high carbonate phosphoric flour on physic mechanical properties of granules of an ammonium nitrate and nitrophos, and working out of the recommendation about creation of new production engineering of reception of products.

1. LITERARY PART

1.1 Physical and chemical and physic mechanical properties of mineral fertilizers

The ammonium nitrate - technical nitrate of ammonium, NH_4NO_3 represents achromatic or *беле* the crystal substance containing 35 % of nitrogen in ammoniacal and nitrate forms. Molecular weight - 80,05 density of 1,69-1,725 g/sm^3 , temperature of fusion of 169,6 wasps. Depending on strength of granules the bulk weight is in limits 0,82 - 1,16 tn/m^3 .

The ammonium nitrate well dissolves in water, ethyl and methyl spirits, acetone and in liquid ammonia. Ammonium nitrate dissolution in water is accompanied with sorption of a considerable quantity of heat. An ammonium nitrate strong oxidizing agent of some inorganic and organic joints.

For needs of agriculture an ammonium nitrate release in the grained aspect in various additives and should contain not less than 34 % of nitrogen [1].

Polymorphic transformations nitrate of the ammonium, reflecting its ability to change a crystal structure is specific property. Depending on temperature ammonium nitrate can exist in five crystal forms (modifications). Transition of one form of crystals in another is accompanied by change of volume, density of crystals, refractive index, a specific heat and other properties, and also allocation or sorption of certain quantity of heat (tab. 1.1.) [5-15].

Table 1.1

Modification transitions of an ammonium nitrate

Type to symmetries (modification)	Warm-up intervals of existence crystal. forms, °C	Temperature of the conversions, °C	Parameters of the lattice, A			Factor of the linear expansion			Density crystal, g/sm ³	Change ud. volume	Heats of the conversions, excrement/g
			a	B	c	A	b	c			
Cubic(1)	169,6-125,2	169,6	4,40	4,40	4,40	1,9	1,9	1,9	-	0,054	16,75
Tetragonal(II)	125,2-84,5	125,2	5,75	5,75	5,50	1,1	1,1	0,5	1,690	-0,013	12,24
Rhombic (monocline) (III)	84,5-32,4	84,5	7,06	7,66	5,80	0,4	1,3	0,5	1,660	0,008	4,75
Rhombic (pyramidal) (IV)	32,4-18,0	32,4	5,75	5,45	4,96	0,2	3,1	0,5	1,725	-0,022	4,99
Hexagonal (V)	under-18,0	-18,0	5,72	5,72	5,90	0,8	0,8	0,0	1,725	0,017	1,60

Besides the specified 1.1 transformations of an ammonium nitrate VI modification is detected at temperature above 170 about and under pressure above 9000 kg/sm² [16]. Existence of VII modification at temperature below -170 wasps is installed by S.I.Volkovich with employees [24] and confirmed by A.Teore and S.Sandorvi [31].

Many researchers [19-22] underline that at heating and ammonium nitrate cooling, along with consecutive transitions I → II → III → IV → V, it is observed at 47-50 wasps metastable transition II → IV, passing modifications III, and at introduction in nitrate of ammonium of some additives - metastable transformation II → V [10,23,24].

In the literature there is a considerable quantity of works on studying of agency of various additives and a moisture on temperature and kinetics of modification transformations.

One of the major parameters characterizing physical and chemical and commodity properties of mineral fertilizers is strength of its granules. Granules should possess sufficient strength of granules and without destruction to stand certain loadings in transit, storage and entering into soil [6].

Strength of granules largely defines such properties of an ammonium nitrate and other fertilizers, as consolidation and hygroscopic [10,25,26].

Initial strength of granules and stability of strength at storage depends on many factors: the mechanism of Crystallization, sizes and density of packing of crystals, moisture content, quantity and an aspect of special additives, a leakage of modification transformations, a refrigerating duty and other processes, and also

from storage conditions, oscillation of temperature and a relative humidity of ambient air [27,28].

With increase in humidity and porosity of a product mechanical stability of granules of an ammonium nitrate decreases. It increases with decrease of sizes of separate crystals of salt [6,29] and excitedly density of their packing [30]. At studying of the transverse spellings of granules under a microscope it has been installed that the density of packing a crystal in the center of granules is less, to that their surface [4]. All granules of an ammonium nitrate in the center have the spherical cavity, incorporating the narrow channel with an outdoor surface. It is formed as a result of substance density at its crystallization from swum. The general porosity of a granule developing of actually porosity of crystal weight and volume of a pipe, fluctuates for commercial machines within 3,4-10 % from volume of granules [31,32].

Ammonium nitrate granules are characterized by the big irregularity of strength: maximum and minimum ν лечены are discriminated in 2-3 times. It follows from this that formation and cooling of different granules proceed in unequal conditions [32].

Till the end of 1960th years when saltpeter was released with humidity by of 1,0 and more percent, strength of granules made 100-300 gr/granule. As a result of decrease in humidity of a product and application of effective aspects of additives strength of granules of an ammonium nitrate increased odes of 400-900 gr/granules [33,35]. However, these parameters yet do not meet demands of agriculture

increasing every year. In-process [36] it is shown that for reception of an ammonium nitrate not slumping at long storage strength of its granules should be not less than 1200 gr/granules. Features of material composition Kyzylkum's phosphorites, consisting in their high carbonateness (maintenance CO₂ attains in some samples of 27 % and more), their rehash complicating process demanded correction of the standard technological circuit designs of manufacture phosphorus inclusive fertilizers.

Developed technological circuit designs provide also mar tempering of quality of released products (an ammonium nitrate, nitrophose, superphosphate, etc.) by a graining of fertilizer and consolidation decrease. These problems cause necessity of performance of a complex of provisions: optimization of a chemical compound of fertilizer, rational sampling of ways of a grain, application of the protective substances inducted into a blanket of granules or in their volume (inoculation) and other.

Conducting of the specified measures precede transpiring of features of process of structuration at mass crystallization of the given product (the single and difficult fertilizer), definition of character of defective structure of crystal blocks (crystallite), composing granules of fertilizer and diffusion of watery-salt components (WSC), carried in crystals. The diffusion stream has the sources and flows as which any cracks serve, pores, dints, including - points of targeting of granules. These factors "control" migration of equated ions and water to fertilizer blankets, causing its consolidation.

The thermodynamics of the superficial phenomena connects these factors with passage of process of diffusion to a direction of leveling of a landform of a surface of the firm phase, accompanying decrease of magnitude of free superficial energy.

Such model of process of consolidation served basic for working out of two directions in the solution of its elimination. According to first from them, it is

recommended to conduct a product graining so that to suppress traffic of dislocations and their outcropping of crystals, to brake a volume diffusion of ions to a surface. It can achieve by introduction no coherent on crystal lattice parameters crystallite some fertilizer small dispersion impurity (limestone, dolomite, etc.) Dislocations braking traffic not only in the course of formation of structure of granules, but also at the further mechanical affecting on a product. The second direction in elimination of consolidation of granules consists in application of one of known ways of a retardation of a surface diffusion. Application of following methods thus is recommended: - drawing on a surface of granules high hygroscopic substances (for example, a diatomite) that leads to destruction WSC; - dusting of granules by hydrophobic substances which, to sorption on the active centers in places of an exit of dislocations on a surface, block them and prevent formation WSC; - drawing on granules of the surfactants giving to a surface water repellency and preventing process of surface diffusion WSC; - a covering of a surface of granules the polymeric films also interfering diffusion WSC in a zone of contact of granules.

At implementation of the above-stated ways of decrease in consolidation of the grained product it is necessary to consider agency of changes of environment also. It is installed that as a result of such affecting origination of various physical structures and diffusion the processes resulting, in the final reckoning, to new, more resistant to equilibrium state of system with a minimum of a free surface is possible. The reason of such phenomena consists in ability of mineral fertilizers in certain conditions to be formed even at rather small loadings.

The similar phenomena (formation of monoliths) can be called also recrystallization or reorganization of a surface of corpuscles in the field of local distortion where a bonding force responsible for stability of a crystal lattice, can relax, raising probability of a leakage of reactions.

Practical recommendations at sampling of a technological regime of a graining and the maximum decrease in consolidation are reduced to that it is necessary to set in advance certain type of structure and a size of granules, considering thus presence of inducted modifications which definitely influence formation of structure of a target product. Many additives in paucities render a great influence on properties of mineral fertilizers.

From resulted obviously that processes of a grain, elimination of consolidation of mineral fertilizers represent extremely a challenge. She demands cumulative studying of many factors accompanied by deformations of a crystal lattice of granules, diffusion WSC, changes of contacts and communications between structural elements (ions) of fertilizers. The big role is played thus by the chemical nature of the components which are a part of fertilizer: presence of them some them promotes reaction with a generating of anions and their migrations from one phase in another or from a boundary of granules in a homogeneous phase.

The experiments made by us on reception of difficult azotic-phosphoric fertilizers on a basis Kyzylkum phosphorites and approbation of various ways of a grain and consolidation elimination allow to recommend some of them, as the optimal storages providing possibility, product transportations in незатрапенном an aspect, and raise of positive agrochemical effect thanks to uniformity of entering into soil and presence at it of several nutritious elements (phosphorus, nitrogen, sulphur, etc.).

High hygroscopic of nitrate of ammonium and the fertilizers containing nitrate of calcium - ability with sorption of a moisture from a circumambient is one of the main reasons of its consolidation, decrease in strength of granules and looseness [30,37,38-41]. Process of sorption of a moisture of air dry soluble salt proceeds in two stages: at first salt adsorbs a moisture and on a surface of crystals the pregnant solution film is formed. From this time, under a condition if a partial pressure of steams of water in air above, than pressure of the water rights over salt

pregnant solution, process of sorption of a moisture by substance passes in absorption area. Thus sorption of steams of water will proceed until all substance pride in a condition of the solution having same dose pressure of steams in a condition of the solution having same dose pressure of steams in a condition of the solution having same dose pressure of steams, as in a condition of the solution having same dose pressure of steams, as in air. If dose pressure of steams in air more low, than vapor pressure over pregnant solution the substance dries up. There is a transpiration of a moisture from pregnant solution.

Relative humidity air at which the substance is not moistened and does not dry up, is called as a hygroscopic point which is function from temperature and is defined from the equation:

$$h = \frac{P_{p.r}}{P_{max}} \cdot 100 \%$$

Where: h - a hygroscopic point, in % . Moistures air

$P_{p.r}$ - pressure of steams of water over pregnant solution,

P_{max} - the maximum pressure of steams of water at the given temperature.

The hygroscopic point of an ammonium nitrate decreases with an increase of temperature:

t °C	10	15	20	25	30	40	50
h %	75,3	69,8	66,9	62,7	59,4	52,5	48,4

Theoretical bases of hygroscopic and its change depending on temperature, sizes and a condition of a surface of corpuscles, are stated solubility of a product, speed of diffusion and other factors in P.E.Pestov's monograph. It in detail studies agency of hygroscopic on physical and chemical and commodity properties of

various fertilizers and methods of definition of hygroscopic, a moisture capacity are developed.

I.M.Kuvshinnikov and Z.A.Tihonovich [23] consider that hygroscopic properties firm should be characterized substances isothermals sorption and kinetic regularity of process. These characteristics to gain by means of a dynamic method easier. In the capacity of criterion for an estimation of intensity of a moisture absorption of salts it is offered to use magnitude of factor of hygroscopic:

$$\gamma = K_x W_p$$

Where: K_x - a constant depending on humidity of air,

W_p - pressure of water vapors over pregnant solution of salts.

For the purpose of decrease in hygroscopic of an ammonium nitrate its mixture with ammonium sulphate, chloride potassium, mono calcium phosphate is offered. Thus formed double salts and solid solutions have pressure of steams over pregnant solution big, than the ammonium nitrate, i.e. they is less hygroscope [43]. The best result on hygroscopic decrease give additives to an ammonium nitrate surfactant active and hydrophobic substances which forms on a surface of granules the un molecular film, interfering a moisture in salt [44,45].

The basic deficiency of an ammonium nitrate and other mineral fertilizers is its consolidation - propensity of its corpuscles turns in certain conditions to the monolithic weight. Consolidation of an ammonium nitrate leads to the big losses and additional labor expenses for product crushing before application [46,47,48]. It is called by high solubility and hygroscopic of salt, polymorphic transformations, low mechanical strength of granules and other specific properties of a product, and also technological deficiencies [6,29,49-56].

The moisture containing in an ammonium nitrate, is in a mother liquor aspect. At decrease in temperature of salt, owing to solubility change, new crystals which link separate corpuscles of a product and as a result the weight of saltpeter turns to a continuous monolith fall out of a mother liquor. Hence, than the temperature is less than moisture in a finished stock and the more low at packing in container, the product with other things being equal less slumps.

At long storage the ammonium nitrate owing to oscillation to temperature and air relative humidity undergoes repeated dissolution and crystallization that spends to a deformation of granules and caking.

To N.E.Pestov [39] it has been shown that the product with equal corpuscles on a size slumps less, than a product with different sizes of corpuscles though average diameter last was equal with first. This results is from the fact that at non-uniform magnitude of corpuscles small corpuscles will occupy pores between large and to a point contact of corpuscles of this product will be more that causes the big consolidation.

On N.P.Kurin [32] at dry wet of nitrate of ammonium, as a result of exudation of moisture, porosity of salt increases. Thus strength of granules decreases that promotes consolidation increase. Act of polymorphic transitions of an ammonium nitrate on its consolidation many scientists.

A.M.Dubovitsky Klevek [65] notes that transition from one modification in another occurs to volume change that involves increase in consolidation of an ammonium nitrate.

N.P.Kurin installed that the changes of phase connected with infringement of a crystal lattice of saltpeter, facilitate migration uterine a solution in places of contacts between granules and consequently promote consolidation.

M.Louri and Himing [60], and also other researchers [59,60] consider that consolidation of an ammonium nitrate in case of modification transitions is not connected with change of character of crystals, and grows out of secondary process - their crushing.

V.A.Klevke of N.K.Whole , A.L.Shneerson, Century And Klevke and M.A.Miniovich [64] specify that consolidation of an ammonium nitrate caused by transformation of modifications III = IV, sharply increases with moisture raise in saltpeter. Authors express opinion that for a leakage of process of the consolidation connected with modification transition, all matters not a moisture which is in an ammonium nitrate, but only containing on a surface of its corpuscles during transformation.

F.Volf and V.Sharre [12] came to a conclusion that modification transitions IV = III do not play any role in consolidation of nitrate of ammonium.

In separate works [62,63] it is noted that altitude salting product storages, an angle of repose, the capillary pressure and creep of solutions of an ammonium nitrate also influence its consolidation.

For the purpose of elimination of consolidation, raise of strength of granules and improvement of other physical and chemical properties of nitrogenous and difficult fertilizers in the industry along with constant development of processes, various additives are applied: hydrophobic, powdering, inoculating and conditioning [64-92].

In the capacity of hydrophobic additives applied paraffin, the paraffin fuel oil, parching oils both other natural and synthetic products [6,7,46,53,60] earlier. However, because of raise of explosion hazard of a product this way has been prohibited.

Capsule ammonium nitrate granules high-molecular [62,93,96] also did not find the application because of high cost and complexity of this process.

During the last years the great attention is given to machining of granules of an ammonium nitrate of the PEAHENS, capable to form on a surface of granules a hydrophobic protective film [51,54,97].

Industrial application synthetic fatty acids WPCA found in our country (waste products cyanic acids) and dispersant «NF» - a product of condensation of formaldehyde with the two-sodium salt naphthalene acid [6,98,99]. Action hydrophobic the PEAHENS put on granules at the rate of formations unimolecular salting rather shortly [100]. Besides under production conditions it is difficult to avoid local accumulation dropping that sharply raises firefighting and explosion hazard of manufacture and the product [53,101,103].

Powdering of granules by pulverous inorganic materials [6,7,32,36,104-108] in our country applications in an aspect of their small efficiency did not find. Ways of the combined drawing powdering and film-forming materials [11] are known.

In works [26,37,101] are generalized result of long-term and regular researches of the mechanism of act of additives DPhF and leaches and their separate components on physical and chemical and commodity properties of an ammonium nitrate. It is shown that phosphate additives DPhF, LEACHES and EPhA from calculation of 1-2 % P_2O_5 in a finished stock eliminate consolidation of an ammonium nitrate. Differing difficult composition, these additives are present at all stages of manufacture of an ammonium nitrate and actively participate in crystallization process, having complex affecting on properties of nitrate of ammonium.

Phosphate additives raise an ammonium nitrate moisture capacity that reduces negative act of hygroscopic not its consolidation and friability. As a result

of decrease in capillary -diffusive mobility of nitrate of ammonium in the presence of phosphate additives possibility of formation of walkways between granules sharply decreases at dry out a product. This results not only in consolidation decrease, but also elimination of process of recrystallization of granules.

Insolubilize components of phosphate additives DPhF, LEACHES and EPhA promote raise of density and strength of granules of an ammonium nitrate.

Phosphate additives and their separate components sharply reduce kinetics of modification transformations that leads to an increase of temperature of transition IV = III, to decrease in temperature of return transformation III = IV and replacement of transformations II - III - IV on metastable transformation II - IV in the course of saltpeter reception. As a result of it the consolidation connected with modification transformations IV = III at storage of a product in the conditions of sharply continental and hot climate is eliminated.

In-process [56] researches of agency of one of components of phosphate additive DPHF - calcium nitrate on physical and chemical properties of an ammonium nitrate are conducted. It is thus installed that in system of alloys in the field of its stratification formation of hydrates of double salts of composition is observed:



Decrease of solubility of azotic-acid ammonium in the presence of calcium and magnesium nitrates is installed by authors work [27-28] at studying of solubility of systems:



Agency of an additive of LEACHES from the Vietnamese apatite and other component of this additive – tri calcium phosphate on physical and chemical properties of an ammonium nitrate is studied by Le Min Njan and

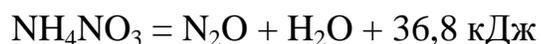
N.S.Torocheshnikov [41,50]. Them also that the additive of LEACHES and tri calcium phosphate increases hygroscopic and reduces consolidation of an ammonium nitrate.

Ammoniac saltpeter is an explosive material, but it is a little sensitive to pushes, a friction, blows and blows up under the influence of a strong detonator. However, from world practice many cases of fires and ammonium nitrate explosions in production process are known, at conveyances and storage [16].

In certain conditions, especially in the presence of some impurity, saltpeter, is inclined to spontaneous decomposition [13-15]. At long heating it gradually decomposes on ammonia on nitric acid, since 110 wasps and above on the equation:



And others installed B.Ju.Rozman that in the interval 473 - 543 to saltpeter decomposition proceeds autocatalytic with nitrous oxide and water formation:



In-process [61] results of research of dependence of speed of thermo decomposition of an ammonium nitrate from the nitric acid maintenance in the interval temperatures 170-280 °C are presented. It is shown that thermo decomposition process is autocatalytic, its speed increases directly proportional concentration of formed nitric acid. There is data on agency of additives of nitrogen dioxide and nitric acid on an initial stage of process of thermo decomposition of an ammonium nitrate at 170 wasps . It is installed that presence of these substances accordingly in 70 and 35 time increases speed of a build-up of pressure at decomposition in comparison with process for a pure ammonium nitrate.

E.B.Moshkovich, I.I.Strizhevsky and others [18], studying agency nitrogenous, sulfuric and phosphoric acid came to a conclusion that speed of

thermo decomposition of an ammonium nitrate depends on temperature and from the maintenance of acids. Introduction of 5,0 % of acid leads to increase in speed of thermo decomposition of an ammonium nitrate at 453 - 473 to in 6 - 63 times depending on an acid aspect.

On the basis of presented above works and other literary data it is possible to make that mineral acids and nitrogen dioxide speed up process of thermal decomposition of an ammonium nitrate, and water presence reduces catalytic nitric acid act.

Agency of other impurity on thermal decomposition of saltpeter is investigated by authors of works [19-22].

And others experimentally installed J.I.Kilman, what even at the insignificant maintenance of impurity of chloride and oils process of thermal decomposition of an ammonium nitrate is sped up and the temperature the beginning of this process on 20 wasps [67,68,69] decreases also.

In-process [69] agency of various aspects of container on explosive property of an ammonium nitrate is studied. By authors it is installed that insignificant impurity of a combustible material of container (the paper, polyethylene and polyvinylchloride) raise sensitivity of saltpeter to initiating affecting electro-genital in 3-4 times.

1.2 Characteristic of phosphorites of Central Kyzylkum

Republic of Uzbekistan - the developed agro industrial country. In agriculture 40 % of able-bodied population are occupied. More than 97 % of all agricultural products gain from 4,3 million in hectare of irrigated lands. Claps and grain cereal is the basic grown up crops. At a present stage of economic development of Uzbekistan the great attention is given to maintenance more than 25 million population by various agricultural products. Fertilizers is solving the factor in raise as productivity of plants, and quality of agricultural products. An important role in the solution of this problem it is taken away to manufacture and effective application of mineral fertilizers.

It is known that each ton of mineral fertilizers applied in farming industry provides annual requirement for foodstuff of 5-6 persons. Besides 40 - 50 % of a gain of a crop of agricultural crops gain from their application. Over forty chemical elements Mendeleev's tables are necessary for normal dew and developments of plants. From them nitrogen, phosphorus, potassium, calcium, sulphur and a magnesium is the cores nutritious. Phosphoric fertilizers takes a special place at raise of fertility of soil and productivity of agricultural crops.

For last years maintenance of agriculture of republic with mineral fertilizers, in particular, on phosphorus and potassium, was considerably divided out. So, the stated requirement for the mineral fertilizers, defined on the basis of scientifically well-founded optimum norm under various agricultural the crop grown up in republic, in 2001 made 997,5 thousand tons nitrogenous, 691,7 thousand tons phosphoric and 352,5 thousand tons potash in conversion on 100 % of nutrients. And security on nitrogenous fertilizers made 58,8 %, phosphoric - only 18 %.

The farming industry condition depends on supply by mineral fertilizers. If at reception of nitrogenous fertilizers by raw components air and the rock gas by which stocks the republic is provided the chemical industry of Uzbekistan on

manufacture phosphor inclusive fertilizers was based on imported mineral raw materials - phosphorites of a deposit to Carat from Republic Kazakhstan is.

In this connection the prompt basis of own deposits of phosphate raw materials, in particular, phosphorites Central Kyzylkumov is extremely important.

With a view of creation of a raw-material base for the chemical factories of Central Asia on manufacture phosphor inclusive since 1975 development and comprehensive studying of phosphorites of central-Kyzylkum basin began leading geologists, specialists and scientists. In which limits the granulose phosphorites are dated to adjournment Eocene age and passed round in them practically everywhere. However, phosphorites which represent economic interest for open mountain workings out, are concentrated mainly in two contiguous dints - Dzherojs and Syrdaryo, as is reflected in the name of a deposit and which is reference for all deposits and developing processes of the granulose phosphorites Eocene epoch sea sediment genesis, revealed in Central Kyzylkum. occurrence mucks on a deposit подоге, complicated by a neotectonics plicate character (a wavy landform of seams) and explosive (disjunctive) infringements quarter palaeogene age. Here, in thickness marl an average eocene power of 50 m and more, are concluded five seams of phosphorites, but practical interest is represented only by two seams, difficult the granulose differences, so-called first and second phosphor layer

The revealed phosphorites refer to to the widespread granulose carbonate to type and to composition is analogues of the largest deposits of phosphorites of the Afrikan-Arabian province, on differ rather low maintenance in ore of a useful component.

Worked overtime the acting factories (Morocco, Algeria, Tunis, Jordan, Israel, Egypt, Iran) phosphorites of this kind contain 20-30 % P_2O_5 commodity concentrate contain, a minimum, 27-29 % P_2O_5 , usually 30-35 % P_2O_5 .

Within the Dzheroj-Syrdaryo deposit eight are gated conditionally out in different extent of proved areas. From them stocks on sections of Tashkura, by Dzheroj Southern and Kurukkuduk are in details reconnoitered. The confirmed geological stocks on these sections at depth of working off to 50 m make: 223,9 million tons ores , with the average maintenance of phosphoric anhydride of 19,42 %; stocks P_2O_5 - 45,5 million tons.

Tashkura - one of three in details proved areas of the Dzheroj-Sirdaryo deposit, accepted in the capacity of prime to commercial operation.

Operational stocks phosphorite ores I and II phosphor layer a section of Tashkura at depth of working off by a quarry to 40 m make 79,6 million tn. (13,9 million tn. P_2O_5). Average maintenance P_2O_5 in operational stocks I phospho layer makes 14,83 % II phosphor layer - 19,28 %, average on the sum on I and II phospho layer - 17,43 %. Average power phosphor layer - 0,62 - 0,63 m. Quantity P_2O_5 on seams: I seam - 33 %, II seam - 67 %. Between seams of 15 m (internal stripping) lie down marl clayey and clay lime power from 8 odes. Mucks of external stripping have power from 5 to 30 m and are presented forestry by loams with prolayers of sandy-gravelitistoj mix, lime clay and weakly phosphate.

On mountain-technical parameters of extraction and qualitative composition кызылкумские phosphorites considerably concede to foreign analogue deposits of the granulose phosphorites in particular and that, extraction and which rehash is carried out as in deserted and anhydrous regions (Jordan, Israel, Egypt, Tunis, etc.). Expenses for manufacture from them phosphoritic concentrates on the traditional production engineering applied to world practice, by results repeatedly (from the moment of assertion of stocks in 1985) the executed technical and economic calculations, promised low profitableness, if not un profitableness of designs. Thus, the question on industrial development of the Dzheroj-Syrdaryo deposit of phosphorites did not find until recently univocal and an affirmative reply.

With the beginning of maintenance of a section of Tashkura in integrated works the program of research and trial works has been developed, on working out of new progressive and economic production engineering of extraction and enrichment of the phosphoritic ores, which heading additional manufacture of the enriched phosphoritic concentrates would allow to provide directions in short terms.

Kyzylkum phosphorites refer to the granulose type and the basic minerals is calcite and fluorine carbonate apatite. They on chemical and mineralogical compositions differ from known phosphorites. The chemical compound of an averaged sample of phosphorite contains mas. % P_2O_5 -19-19; CaO - 43-46; MgO - 0,6-0,8; CO_2 -14-16; R_2O_3 - 2,0-3,0; SO_3 -2,5-3,5; F-1,5-2,5, H_2O - 5-8. For these phosphorites traditional enrichment methods are inefficient. Rehash of finished phosphate raw materials as told I.A.Karimov's Republic of Uzbekistan Pezident during the visit in Zarafshan, during to go in three directions. First, it is necessary to organise to manufacture of fertilizers, commensurate require agriculture. Cost to this blow owe a life low, than equipments had possibility to buy them. The second direction - is necessary on mimic factories sentry raw materials with high containing P_2O_5 . The third direction of manufacture examiner products matching to the international standards.

We consider here pertinent, is short informs on extraction of Kyzylkum phosphoritic ore, about starting-up and a condition look through it on fertilizers. At extraction Kyzylkum фосфритной ores with helping somasvalno-radiometric quality monitoring on concentration of the basic component phosphorites share on some quality: 1 sort-20-21 % P_2O_5 ; 2 quality-19-19,5 of % P_2O_5 ; 3 quality-17-18 of % P_2O_5 ; 4 quality-14-16 of % P_2O_5 and the poorest phosphoritic ore, so-called mineralized the weight containing 10-12 % P_2O_5 , is thrown out in a sailing.

At Navoi mountain-metallurgical integrated works (NMMI) richer part of the phosphorite containing 19,5-20,5 % P_2O_5 , subject to drying to crushing to certain sizes of corpuscles, then classify. Further a method of dry enrichment small a flour milling less than 0,5 mm concentrate to 23-24 % P_2O_5 . The phosphoritic flour after dry enrichment goes to oven branch where under the influence of high temperature 800-850°C burn. 85-95 % диоксида carbon Here leave, and organic matters are completely burnt. After roasting in the gained thermo concentrate concentration of phosphorus attains 26-27 %, but a relationship of CaO: P_2O_5 remains high (1,90-1,95). Since April, 2001 on the present in Zarafshan of Navoi area the plant on roasting torture with productivity of 400 thousand tons of a phosphoritic concentrate.

The basic deficiencies of an existing method of thermal enrichment of phosphorites Kyzylkum, in our opinion, are complexity of the circuit design and high temperature application that prepares for essential rise in price of the cost price of a phosphoritic concentrate and an ammophos from it.

Ordinary phosphorite the flour with the maintenance of 18-19 % P_2O_5 arrives on Kokand superphosphate plant for reception of phosphate fertilizers.

Now 400 thousand tons of the burnt concentrate and a maximum of 100-120 thousand tons of an ordinary phosphoritic flour is worked overtime on an ammophos and superphosphate idle time accordingly. The phosphate fertilizers gained from such volume of phosphorite, it is naturally not enough for satisfaction of requirements of agriculture of Republic.

To today's for 40-45 % extracted phosphoritic ore Central Kyzylkum, containing 15-18 % P_2O_5 , is warehoused and does not find applications in production engineering of lecture of fertilizers. Huge quantities of poor phosphorites with the high maintenance of a marl and carbonates (mineralized the weight) in general is thrown out in a sailing. After enrichment mineralized weight

maintenance P_2O_5 in it attains 12-14 %. In the long term with expansion of integrated works the quantity of extracted ore increases to 3600 thousand tons. Thus naturally increases volume of off-grade phosphorites.

1.3 Condition of manufacture not slumping ammoniac saltpeter

For mark long a time an ammonium nitrate as fertilizer to be used in the form of the so-called lime -ammoniac saltpeter gained by mixture of an ammonium nitrate with ground limestone. Such product contained 21 - 28 % nitrogen.

The ammonium nitrate meeting demands of agriculture, owes a life without free moisture, is released in the form of homogeneous strong granules and to incorporate small quantity of the special additives softening an adverse effect on strength of granules of polymorphic transformations, proceeding at their formation.

For ammonium nitrate reception by initial components 58-60 % nitric acid and gaseous ammonia are. The production engineering consists of following basic stages: nitric acid neutralization by gaseous ammonia, reception high concentrated solution an ammonium nitrate, a graining solution, cooling of granules, packing and finished stock storage. For reception of a commodity ammonium nitrate of better quality with high stable strength, suitable the additive representing a solution of phosphate and sulphate of ammonium is applied to a bulk handling and a bulk storage – phosphor- sulphate.

In connection with sharp increase in manufacture and ammonium nitrate consumption there was a severe need of substantial increase of its quality. Therefore all factories of the country have been translated on exhaustion of an ammonium nitrate without internal additives with the downgraded humidity and cooling of granules below 30 wasps before casing. Application of dusting additives was provided. However, quality of an ammonium nitrate not only improved, on the contrary, worsened and losses of a product with departing air from granule tower,

thumb thereby a circumambient increased. Experiences on powdering of granules did not give desirable result. Therefore all factories have been translated again on use of internal additives.

Initial raw materials for reception of a magnesium additive is the dead-burnt magnesite grain gained as a result of trapping of a dust, formed at manufacture спеченного magnesite. This powder contains a significant amount of impurity of organic chemistry (black) and the sulphates which overshoot in system of manufacture of an ammonium nitrate is extremely dangerous in the composition. Besides, saltpeter with magnesia and dolomite additives it is necessary to store in a sealed container since it possesses higher hygroscopic properties, than saltpeter with other additives. Machining of granules by dispersant «NF» and WPCA increases fire and explosion hazard of the product.

As a result of decrease in humidity and application of conditioning additives considerably raised qualities of an ammonium nitrate, but it still ferly does not meet increasing demands of users and does not provide a bulk handling and product storage.

The further improvement of quality of an ammonium nitrate by decrease in humidity of a product and increase in quantity sulphate, magnesium, доломитной additives, and also dispersant «NF» and WPCA is not obviously possible, as it leads to raise of explosion hazard of manufacture and is economically not favorable.

At increase in quantity of a phosphate additive explosion hazard of manufacture decreases, consolidation is completely eliminated and sharply strength of granules of an ammonium nitrate raises. Besides, as a part of an ammonium nitrate with a phosphate additive additional phosphate fertilizer is produced.

The analysis available literary the data and a condition of manufacture of an ammonium nitrate and difficult fertilizers shows that for full prevention of consolidation, sharp raise of strength of granules of fertilizers and maintenance of stability of quality indicators at long storage in various environmental conditions, the most perspective, economic and providing safety of manufacture is introduction of an additive of products azotic acid and vitriolic decomposition of phosphate in number of more than 1,0 % in conversion on P_2O_5 .

- The Purpose of the present work was agency research high carbonate a phosphoritic flour on properties of an ammonium nitrate and difficult fertilizer - nitrophose.

Finding of the first part.

For the purpose of the further improvement of quality and development of production engineering of reception of an ammonium nitrate with a phosphoritic flour the new way of introduction of an additive is offered.

Research of physic mechanical and commodity properties of samples with various additives showed that the ammonium nitrate with high carbonate a phosphoritic flour does not slump and possesses high strength of granules and stability at repeated modification transformations and long storage.

2. EXPERIMENTAL PART

2.1 Methods of research of properties of mineral fertilizers. Definition of humidity of samples of an ammonium nitrate and difficult fertilizers

Humidity of samples defined methods of drying and culonometric titration by Fisher's chemical agent.

The Culonometry method is based definition of the electric charge spent on electrochemical oxidation of ions of iodine, formed at interacting of chemical agent of Fisher with a saltpeter moisture in anodic the electrolyze chamber on reactions:



After emersion on anode J_2 , the chemical agent containing excess SO_2 , becomes again active to water. Installation consists of an electrochemical mesh with generating and display electrodes, the power supply, devices for registration of a display current and the magnetic rabble. In the installation circuit design possibility of compensation of drift of the display current called by instability of chemical agent of Fisher is provided. Methyl spirit is an additional source of formation of water owing to reaction etherify in the presence of acids in the presence of acids in the analyzed sample or formations of acetyls and catalysis at presence carbonate joints [23]. Depending on quality of chemical agent of Fisher the compensatory current reaches 30 A. Labor toque generation (80 + 100) And. In the capacity of a material for display electrodes and the anode of generating pair platinum is used. In the capacity of the cathode of generating pair the graphite impregnated by paraffin

is used. The electrochemical mesh (electrolysis) consists of two chambers with a partition between them from simple glass.

In the anodic chamber of a mesh pour 60-70 ml of the dehydrated methanol and 15-20 ml of a solution in pyrites (a solution Fisher's 1 chemical agent). An electrolyte of the same composition pour in the cathode chamber. In a mesh haul down electrodes and put on the magnetic switches on for 1-2 minutes prior to the beginning of an electrolysis. Further flow in the anodic chamber of a mesh an iodine solution in methanol (a solution Fisher's 2 chemical agents) until the display current does not attain the chosen point of equivalence-5mk A and install the compensatory current fixing a display current near to a point of equivalence. After that into the anodic chamber of a mesh induct test of saltpeter 0,2-0,3 g and switch on generating system. An electrolysis conduct until then, the display current does not attain the initial value. On reaching an equivalence point generating system will be disconnected automatically. The quantity of the iodine oscillated on the anode, is proportional to the electric charge, passed through a mesh and is a moisture content measure in saltpeter test:

$$C_{H_2O} = K \frac{(i-i_0)\tau M_{H_2O}}{96500 np}$$

THERE ARE:

C_{H_2O} - A moisture content, % weight.;

P – exemplar saltpeter tests;

τ - A generating time, second;

i- A generating current, ampere;

i_0 - a current of compensation of drift of "null", ampere;

M - molecular weight;

n - Quantity of charges, $n J_2 = nH_2O = 2$

k- an exit (J_2) on a current, %.

The express-method of definition of consolidation of saltpeter applied now [24] at unitary modification transition IV-III cannot characterize quality at storage and various environmental conditions. Therefore consolidation of saltpeter we defined at repeated modification transitions III - IV with a stand-up from 50 wasps within 8 hours with the subsequent cooling to 15 wasps - 16 hours and at long storage under room conditions (15-28 wasps). In all ways of definition of consolidation set forth above the device applied in technique SINI has been used. Extent of consolidation of samples after a stand-up in the device it was defined crushing cakes in an electromechanical press «Zeus Schjhhez».

For elimination of agency of sizes of granules on consolidation of saltpeter we used an equal mechanical grading.

Static strength of granules of samples defined on the device of type MIP-10-1 by technique SINI [1] in which basis loading measurement is put, at which granules are destroyed. On 20 granules in diameter of each sample of 2 mm subjected crushing and computed arithmetic-mean value of strength of granules.

Agency moistening on strength of granules defined after a stand-up of samples in exactors with water at 20-25 wasps in a current of 8 hours.

2.2 Methods of chemical analysis of raw materials and mineral fertilizers

By working out of scientific bases of production engineering of reception NP - fertilizers applied chemical and physical and chemical methods of the analysis. By well-known techniques carried out the analysis of phosphate raw materials Central Kyzylkum, the sour and neutralized mashes, ready fertilizers on the maintenance of various components.

The maintenance of all forms of phosphorus (the general, comprehensibility, water-soluble) defined photolorimeter a method in the form of yellow phosphor vanadium molybdenum a complex on photolorimeter KFK - 3 (440nm).

Definitions of a mass fraction of the general phosphate (P_2O_5) it is based on test dissolution in hydrochloric or nitric acid. Used chemical agents and solutions: acid hydrochloric in accordance with GOST 3118, 20 % solution or acid nitrogenous in accordance with GOST 4461, diluted 1:2. Extraction spend as follows: 1,0 r fertilizers weigh (result of weighting in grams write down to within the fourth decimal digit), transfer to a glass and a conic flask capacity of 250-300 cm^3 , moisten 5-10 cm^3 of water and add 30 cm^3 of acid and water to volume of 50 cm^3 . A glass cover with hour glass and heat up at first slowly, and then lead up to boiling and slowly boil 30 minutes, for transfer of polyphosphate in orthophosphate - 60 minutes, from time to time mixing a glass stick, adding water in process of evaporation of a solution to volume about 50 cm^3 .

After boiling a solution dilute with water twice both transfer together and transfer rest with a deposit to a measured flask capacity of 250 or 500 cm^3 , carefully washing walls water. After cooling to room temperature solution volume lead up water to a label, mix and filtrate, kicking the first portions of a filtrate.

Extraction of water-soluble phosphate and free acid water. The method is based on extraction of water-soluble phosphate and free acids by water. Extraction conducting: 2 g tests (4-5 r tests at definition of water-soluble phosphate and free acid), weigh (result of weighting in grams write down to within the fourth decimal digit) and transfer to a flask of Shtohman (or in a measured flask) capacity of 250 cm^3 (is admitted to apply a measured flask capacity of 500 cm^3). Test 200 cm^3 (fill in 400 cm^3 according to taken exemplar tests 4-5) waters and at once mix to avoid formation of lumps. A flask close a stopper, install on the apparatus for a shaking and shake 30 мин. After this time flask contents lead up water to a mark and

carefully shake within several minutes. Then a solution I will strike after deposit sedimentation filtrate through the dry filter «a white tape» in dry ware.

The differential photometric method of definition of phosphate is based on formation is yellow colored phosphor vanadium molybdenum a complex and photometric measurement of optical density of this complex at wave length equal 430-450 nanometers concerning a solution of the comparison, containing quantity P_2O_5 is known. Used equipment, chemical agents and solutions: the spectrophotometer with the monochromatic or the filter with the photocell with ditches with a thickness absorbing light salting a solution not less than 10 mm, photo electro calorimeter type КФК (440 nanometers) or another analogous the device.

For construction cooler a drawing capacity of 100 cm induct serially measured volumes of working solutions into five measured flasks depending on concentration P_2O_5 in an analyzed solution so that this concentration was in limits between the least and greatest concentration P_2O_5 in working solutions.

Dilute with water of odes of volume about 30 sm^3 . After that flow 40 sm^3 of solution (is admitted to flow 25 sm^3 of a solution). Solutions refill water of odes of a label at temperature of 20 wasps (it is admitted at room temperature) and mix.

Through 15 mines (more than through 60МИН), measure optical densities or magnitudes light pass the colored working solutions concerning a solution of comparison with the least quantity P_2O_5 . It is admitted magnitudes light pass to convert on value of optical densities.

Working solutions prepare simultaneously with preparation of an analyzed solution, and optical density of working solutions measure in the beginning and in the end of the analysis, define average arithmetical value and троят cooler the schedule, postponing for axes of abscissae concentration P_2O_5 in 100 sm^3 of a solution in milligrams, and on axes of ordinates matching to them dimension optical density.

Analysis conducting: 25 sm³ of an analyzed solution select a pipette in a glass capacity of 200-400 sm³, add 20 sm³ of hydrochloric acid, boil 15-20 mines, transfer quantitatively to a measured flask capacity of 100 sm³, refill water to a mark at temperature about 20 wasps and mix.

At the analysis of a solution and definition orto - forms P₂O₅ of operation of addition of hydrochloric acid and boiling are expelled.

At application of a settlement way an analyzed solution scan photo metrically simultaneously with two solutions of comparison closest to it on optical densities (one - with mainly, second - with smaller optical density, than at an analyzed solution) and different from each other on weight of phosphate no more than on 0,5 mg P₂O₅, concerning a solution of the comparison containing 1 mg P₂O₅ in 100 sm³.

Machining of results.

Kitsch a share of phosphate in conversion on P₂O₅ (X₃) in percentage compute by formula:

$$X_3 = \frac{m1 * V2 * 100 * 100}{m * 25 * V4 * 10000} = \frac{m1 * V2 * 0,4}{m * V4}$$

Or

$$X_3 = \frac{m1 * V2 * 100}{m * 25 * V4 * 1000} = \frac{m1 * V2 * 0,1}{m * V4}$$

There are: m1 - weight of phosphate (P₂O₅) in the analyzed test, defined on гради ровочному to the schedule or by formula, mg;

$$m = 0,2 * V'1 + 0,2(V'2 - V'1) * \frac{D - D1}{D2 - D1}$$

m - weight exemplar analyzed test, g;

V2 - The volume of a measured flask applied at extraction, cm^3 ;

V4 - Volume of an analyzed solution, cm^3 ;

V'₄ - the volume of the analyzed solution which has been selected on tab., cm^3 ;

0,2 - mass fraction P₂O₅ in a working solution of potassium phosphate, mg/cm³;

V'₁ and V'₂ - volume of a working solution phosphate the potassium, taken for preparation of solutions of comparison, cm^3 ;

D - optical density of an analyzed solution;

D1 And D2 - optical density of solutions of comparison of volume V1 and V2 accordingly.

For result of measurement accept an average arithmetical results of two parallel definitions, admitted the divergence between which at confidence level P = 0,95 should not exceed:

At mass fraction P₂O₅ from 3 to 10 % - 0,2 %;

At mass fraction P₂O₅ from above from 10 to 50 % - 0,2 %;

At mass fraction P₂O₅ over 50 % - 0,5 %;

At working out of standards on a concrete product refinement of metrological characteristics is admitted.

The method of definition of the sum ammoniacal and nitrate nitrogen (a method of Devard) consists in restoration of nitrate nitrogen to ammoniacal an alloy of Devard, with the subsequent distillation of ammonia and its titrimetric definition.

Applied devices, chemical agents and solutions: the device for an ammonia distillation, an electric tile in accordance with GOST 14919 or heater of bulb, sodium hydrated oxide in accordance with GOST 4328, 40 %, 0,5 n. And 0,1 n. Solutions, chemical agent of Neccler - prepare in accordance with GOST 4517, an alloy of Devard pounded in a metal mortar to a size of corpuscles approximately 1 mm, methyl red and methyl blue (indicators), water distilled on GOST 6709-72,

acid sulfuric in accordance with GOST 4204-77, 0,5 n. And 0,1 n. Solutions, spirit ethyl technical in accordance with GOST 17299.

The indicator mixed, prepare as follows: 100 cm³ 0,03 % of a solution methyl red in 70 % ethyl spirit mix about 15 cm³ of 0,15 % of a solution methyl blue in water.

Analysis conducting: 1,2 - 2 fertilizers depending on the nitrogen maintenance weigh with a margin error no more than 0,0002 g, of 250 cm³ prevent in a measured flask capacity, refill to a label water and mix. The muddy solution is filtrated through the filter by «a dark blue tape». Then calibrated a pipette select 50 cm³ of a solution and transfer to a reactionary flask, if fertilizer completely solution. The test which is brought in to a reactionary flask, should contain no more 60 mg nitrate nitrogen. After that in a flask add 2-3g an alloy of Devarda and 100-300 cm³ of water. A flask connect through the drip pan to the refrigerator and the receiver.

From a burette pour 25-50 cm³ 0,5 n. Or 0,1 n. A sulphuric acid solution, add 3 drops of the mixed indicator and a water small amount that barbette has been closed by a liquid. In yearly a flask from the drop funnel carefully flow 40 cm³ of a solution of sodium hydroxide. After the termination of rough reaction a flask heat up on an electro tile and boil a solution until 2/3 liquids from a flask distils off. After that muster absence of ammonia and a condensate, for this purpose detach the receiver, wash the end barbette, type in a test tube about 1 cm³ of a condensate and add some drops of chemical agent of Nessler.

In the absence of ammonia there should not be a yellow painting. After the termination of a distillation the receiver with the refrigerator detach, wash out cold water, merging rinsing water in the receiver, and excess of acid or titrate 0,5 n. A solution of sodium hydroxide in the presence of the mixed indicator before change of painting from violet through grey to the green.

Simultaneously spend a check experiment in the same conditions and with that quantity of chemical agents, but without an analyzed product.

Nitrogen and a check experiment define daily and at application of new chemical agents. Weight exemplar analyzed fertilizer, a condition of dissolution and quantity of chemical agents specify in standards and the specifications installing technical requirements on fertilizer.

Machining of results: mass a nitrogen share in fertilizer (X_3) in percentage compute by formula:

$$X_3 = \frac{(V - V_1) * K * 250 * 100}{m * 50}$$

There are: V - volume precisely 0,5 n or the sodium hydroxide solution, spent for excess titration in a check experiment, sm^3 ;

V_1 - Volume precisely 0,5 n or the sodium hydroxide solution, spent for titration of excess of acid in analyzed to test, sm^3 ;

To - quantities of the nitrogen, the matching 1 sm^3 of a solution of sodium hydroxide (for 0,5 n a solution $T_o = 0,007$, for 0,1 n a solution $T_o = 0,0014$), g;

m - Weight exemplar, g;

For an assay value adopt an average of the arithmetical two parallel definitions admitted divergences between which should not exceed 0,3 % at confidence level $P = 0,95$.

2.3 Improvement of property of the grained ammonium nitrate

On existing production engineering for reception of an ammonium nitrate with magnesium or dolomite or a phosphate additive raw materials decompose at the big excess of nitric acid and the special reactor. Accordingly nitric acid the additive solution in the core consists from:

- Magnesium and calcium nitrates and insolubilize rests;
- Calcium and magnesium nitrates and insolubilize rests;
- Phosphoric acid, nitrates of calcium, a magnesium, equilateral oxides and insolubilize rests.

The gained solution separate from insoluble rest in the settler. Further nitric acid the solution the pump goes to the pressure tank or in storage. From the pressure tank it arrives in apparatus UNH (using of neutralization heat) where it will be neutralized by gaseous ammonia before sub acidic reaction. Gained lye until neutralization before alcoholize reaction in until neutralization also mix with a solution of a pure ammonium nitrate, упаренным in a prime steam. A mix of a solution of saltpeter with an additive solution finish above granulation towers where, taking place apparatuses of the second step steam and till steamer, turns to 99,5 % solution which splutter in granulation to a tower.

All additives consist from water-soluble (nitrates of calcium, a magnesium, monophosphate of ammonium, calcium, a magnesium, etc.) and water insoluble (di – tri phosphate calcium, a magnesium, equilateral oxides, gels, silicic acid, hydrofluoric acid salts etc.) joints.

At such introduction of an additive in ammonium nitrate production engineering it is observed, because of incomplete branch of insolubilize rest SiO_2 , indecomposable raw materials, collect in considerable quantities on a storage bottom, forming with a solution of nitrate of ammonium motionless weight which is chilled eventually and as a result ammonium nitrate crystallizations turns to a monolith. The same phenomenon is observed and in pressure tanks solution an ammonium nitrate.

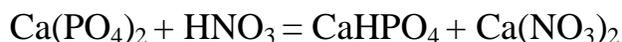
Sticking of insolubilize corpuscles to a wall of grain mills occurs under the influence of a centrifugal force therefore apertures of grain mills converge that in turn leads to a decline of a mechanical grading of a product.

Sticking of deposits to internal surfaces of kettles and communications. As a result insolubilize corpuscles of an additive start to settle on an internal surface tubular, forming firm layered crusts.

At evaporation of solutions of an ammonium nitrate in the 11-step apparatus steam and in until steamer the apparatus part P_2O_5 passes again in the citrate-soluble form. The maintenance last in a finished stock attains to 70 %. This results from the fact that at high-temperature evaporation of a solution of an ammonium nitrate there is a thermal decomposition of nitrate of ammonium on reaction:



Ammonia depositing thus passes in a steam phase, and nitric acid co-operates again with three calcium phosphate and translates it in di calcium phosphate :

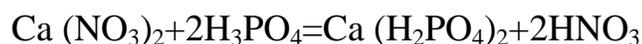


Thus, the considerable part of phosphorus is in a phosphate additive in comprehensibility plants to the form. by ammonium nitrate manufacture phosphate fertilizer is produced in addition.

From the above-stated follows that for the further improvement of quality of an ammonium nitrate by increase in quantity of a phosphate additive without development of existing production engineering it is not obviously possible.

For elimination pacification the equipment of department of an ammonium nitrate insolubilize components of an additive had been offered a way according to which the phosphorite after branch from insolubilize rest expelling upward грануляционных towers where it will be neutralized by gaseous ammonia in the separate apparatus and is added to solution an ammonium nitrate or directly ahead of its grain, or to solutiony, arriving in the film apparatus until boil down. Thus all equipment of department will work on pure lye of an ammonium nitrate therefore it is eliminated pacification equipment and communications by insolubilize components of an additive.

The laboratory trials spent by us showed that at neutralization the phosphorite NAS is formed by gaseous ammonia firm weight. Extraction of such weight from neutralizer and its introduction in solution an ammonium nitrate are under production conditions connected with great difficulties. Besides, the neutralized NAS contains in the composition to 30 % of a moisture that leads to a deleting solution. It in turn reduces quality of a finished stock.



These are condition under production conditions leads to a reinforcement of corrosion of equipment and the big losses of combined nitrogen.

Thus, выпарка neutralized or partially neutralized azotic acidity a solution of phosphorite and its introduction in solution an ammonium nitrate before a grain practically is not obviously possible.

Finding of the second part.

It is installed that in process of formation of granules of an ammonium nitrate an inducted flour carry out a role of crystallization centers and reduce warmth of Crystallization solutiona. They brake kinetics of modification transitions and promote metastable transformation IV=II at rather high humidity of an ammonium nitrate and to reception of granules with high extent of crystallinty and density.

3. AGENCY OF ADDITIVES OF AN AMMONIUM NITRATE

3.1 Agency of additives on physic mechanical properties of an ammonium nitrate.

One of the major parameters characterizing quality of an ammonium nitrate is strength of granules. With raise of strength of granules speeds of sorption of a moisture from an aerosphere are downgraded, salt dissolution in water and consolidation of a product is eliminated. Strength of granules and their stability in the course of long storage with other things being equal largely depend on quantity and an aspect of applied additives.

In the given section results of laboratory researches of quality of an ammonium nitrate with the phosphate additive gained on production engineering developed by us in comparison with quality of commercial machines, saltpeter with various additives are resulted.

On strengths of granules and consolidation of an ammonium nitrate and change of these parameters we spent researches of agency of additives depending on quantity of modification transformations by heating within 8,0 hours to 50⁰C with the subsequent cure to currents of 16 hours at room temperature and from long storage in polyethylene sacks at room temperature, without modification transformations.

Change of strength of granules of an ammonium nitrate depending on storage period shows that the ammonium nitrate with a phosphate additive of the LEACHES has the greatest initial strength of granules, gained on new production engineering - 1100-1250 are conceded a little to it by samples of an ammonium nitrate with a magnetite additive - 750-1000/granules possess the Least strength of granules an ammonium nitrate without an additive - 200-400 g/granules and with a dolomite additive-420-510/granules

At storage in equal conditions strength of granules of an ammonium nitrate with a phosphate additive raises a little, whereas strength of other samples considerably decreases. At storage it is possible to explain change of strength of granules, proceeding from following reasons: air temperature indoors fluctuated in limits from 16⁰C to 26⁰C and accordingly air relative humidity therefore saltpeter absorbed a moisture at night changed, and in the afternoon it parched. At repeated repetition of these processes occurs loosening structures of granules that leads to strength decrease. Raise of strength of granules with a phosphate additive at storage speaks that additive corpuscles have the developed specific surface and possess high adsorption ability. Thanks to it saltpeter with a phosphate additive at minor alterations of temperature and air relative humidity practically does not dry up also granules are not exposed to loosening. Therefore it does not lose initial strength of granules.

Research of change of strength of granules of an ammonium nitrate at modification transformations IV=III shows that they strongly reduce strength of granules of saltpeter without an additive and with a dolomite additive. After 10-15 aliquot modification transformations they are completely destroyed and lose spherical forms. The least decrease (10 %) strengths of granules is observed at an ammonium nitrate with the phosphate additive gained on offered production engineering.

Studying of consolidation of an ammonium nitrate showed that they at storage in room conditions within 30 days practically do not slump.

The phosphate additive is the most effective in raise of quality indicators of an ammonium nitrate and promotes raise of their stability at moistening. This results from the fact that the absorbed moisture adsorbs on surfaces of corpuscles of an additive and communicates in crystalline hydrates, i.e. the moisture does not participate in process of dissolution of saltpeter. As a result of it at moistening of

saltpeter to a phosphate additive there is no sharp decrease in strength of granules and consolidation raise.

Raise of disparity of corpuscles of an additive leads to increase in a surface of a coalescence of particles of an additive with crystals of an ammonium nitrate and quantity of crystallization centers in each granule. As a result of this considerable raises strength of granules. On the other hand, high dispersion phosphate has the developed specific surface and possesses high adsorption ability and they mainly extents prevent negative agency of a moisture on qualitative ammonium nitrate parameters. Therefore at the equal maintenance of a phosphate additive of LEACHES ammoniacal the saltpeter gained by introduction the ACRE of phosphate in solution, has higher strength of granules, smaller consolidation and is capable to keep high figures of merit at long storage, than the saltpeter gained on existing production engineering.

Value hygroscopic points of samples of an ammonium nitrate at 20 and 30⁰C are presented in table 1.

The analysis of results shows that values of hygroscopic points of pure nitrate of ammonium at 20 and 35⁰C will well be co-ordinated with the literary data/135/, the divergence between values is not exceeded by 2 % of the relative. With increase in quantity of an additive of LEACHES to 5 % of weights, in conversion on P₂O₅ values of a hygroscopic point of an ammonium nitrate decreases to 59,5 and 45,9 % of a relative humidity of air at 20 and 35⁰C accordingly. It is caused by increase in quantity of hygroscopic nitrate of calcium in samples.

Table 3.1

Change of a hygroscopic point of an ammonium nitrate depending on quantity of an additive

Ammonium nitrate with an additive of LEACHES Humidity, % on Fisher		Hygroscopic point, %	
		20 ⁰ C	35 ⁰ C
Without an additive	0,26	67,5	56,8
0,5% P ₂ O ₅	0,24	67,0	56,3
1,0% P ₂ O ₅	0,29	66,2	55,3
2,0% P ₂ O ₅	0,30	64,5	51,8
5,0% P ₂ O ₅	0,40	59,5	45,9

Pulverous samples of an ammonium nitrate under equal conditions absorb much more water steams, than grained. Powders of a pure ammonium nitrate and with an additive of 0,5 % P₂O₅ absorb equal quantities of steams of water. The further increase in concentration of a phosphate additive of LEACHES leads to raise sorption water vapors. Sorption water vapors the grained ammonium nitrate raises since 0,5 % of an additive in conversion on P₂O₅.

Raised sorption with additives LEACHES it is possible to explain ability of an ammonium nitrate to that additive corpuscles (phosphate of calcium, a magnesium, etc.) is in regular intervals distributed and almost completely cover a surface of granules and therefore sorption of steams of water occurs on surfaces of corpuscles of the additive, possessing high adsorption ability.

Usually, in warehouses where mineral fertilizers air relative humidity are stored fluctuates in limits from 10 to 100 %. Therefore, we also study speed of sorption of a moisture by samples of saltpeter with various additives at 100 % of a

relative humidity of air and 25⁰C. For this purpose samples of saltpeter with equal quantities and a mechanical grading placed in standard weighing bottles which stood in the exicator with water and hourly weighed. From the arithmetic-mean data on a gain in weight exemplar defined quantity of the absorbed moisture in % from saltpeter weight.

Speed of sorption of a moisture possesses the greatest saltpeter without an additive, and samples, saltpeter with a phosphate additive of the LEACHES, gained on offered production engineering have the lowest speed of sorption of a moisture. Reduction of speed of sorption of a moisture by saltpeter in the presence of additives speaks the raised strength of granules and decrease in solubility of saltpeter in absorbed a moisture.

Use of local phosphorites in the capacity of additives to an ammonium nitrate is more economic. Application of intermediate products of acid rehash - экстракционной phosphoric acid (EPhA) and azotic acid solutions of phosphate for ammonium nitrate air-conditioning represents the big practical and economic interest.

For the purpose of the solution of the questions set forth above we study agency of products of acid decomposition of phosphorites Central Kyzylkumov on ammonium nitrate quality indicators.

For preparation of additives azotic acid a solution phosphorites of Kyzylkum from a section of Tashkura and a thermo concentrate decomposed (ACRE) 56 %-nitric acid at norm of 120 % from a stoichiometry. Compositions of initial phosphorites and azotic acid solutions after branch from insolubilize rest are resulted in table 2.

ACRE of phosphate inducted in solution an ammonium nitrate at 180⁰C and simultaneously submitted necessary quantities of gaseous ammonia for neutralization. Ammonium nitrate granules gained by spraying solutiona with altitude 10 m.

For the purpose of definition stability of quality indicators of samples of saltpeter at long storage consolidation and strength of granules defined every other day their receptions under room conditions, repeated modification transition IV=III and saltpeter moistening on 2 %.

Table 3.2

Chemical compound of Kyzylkum phosphorites and Solution with azotic acid

Initial raw materials, %			Solution with azotic acid, %		
ingredients	Phosphorite	Thermo-head	ingredients	Phosphorite	Thermo-head
P ₂ O ₅	22,95	27,28	CaO	14,73	16,11
CaO	44,71	46,12	P ₂ O ₅	8,23	9,34
MgO	0,78	1,08	MgO	0,42	0,45
Fe ₂ O ₃	1,28	0,21	Fe ₂ O ₃	0,21	0,09
Al ₂ O ₃	1,36	1,25	Al ₂ O ₃	0,41	0,95
CO ₂	14,60	3,42	HNO ₃	11,36	12,88
F	2,09	2,65	H ₃ PO ₄	10,20	11,20
H ₂ O.	8,55	7,80			

The analysis of quality indicators (table 3) shows that strength of granules of an ammonium nitrate with an additive the ACRE from concentrate of phosphate a section of Tashkura raises with increase in quantity additive water (0,2-2,0 % in conversion on P₂O₅) in 2,5-5,0 times, in comparison with saltpeter without an

additive. At storage within 6 months strength of granules of pure saltpeter decreased almost in 2 times, and strength of granules of saltpeter with an additive - all on 9,0-10 %. High strength of granules remains and after 30-fold modification transition IV=III, also at product moistening on 2 %.

At the maintenance of an additive in number of 0,5 % P₂O₅ saltpeter does not slump. With increase in quantity of an additive decreases stickiness at 30-fold modification transition IV=III and moistening to 2 %.

Researches showed that the branch the ACRE from insolubilize rest of phosphorites is extremely difficult operation as its considerable part consists from высокодисперсных corpuscles of silicic acid and joints of equilateral oxides. For the purpose of simplification of production engineering of preparation the ACRE experiments on its introduction in solution an ammonium nitrate with an oozy part, after branch from large fraction of insolubilize rest an elutriation have been made.

Table 3.3

Agency azotic acid a solution of phosphorites of Kyzylkum on physical and chemical and commodity properties of an ammonium nitrate

amounts additive P ₂ O ₅ , %	Humidity, %	Strength, g/granules			Consolidation, kg/sm ²		
		The initial	In 6 months of storage	After 30 short modif. Transition	The initial	In 6 months of storage	After 30 short modif. Transition
-	0,29	385	200	90	0,70	6,71	4,45
0,50	0,29	950	870	830	no tracking	0,67	0,87
1,00	0,30	1385	1280	1140	no tracking	no tracking	0,31
2,00	0,32	1860	1785	1570	no tracking	no tracking	no tracking

The researches set forth above specify in possibility of use of phosphorites of a deposit Central Kyzylkum in the capacity of additives to an ammonium nitrate.

Developed technological circuit designs provide also mar tempering of quality of released products by a graining of fertilizer and consolidation decrease. These problems cause necessity of performance of a complex of provisions: optimization of a chemical compound of fertilizer, rational sampling of ways of a grain, application of the protective substances inducted into a blanket of granules or in their volume (inoculation) and other.

According to the data known from extensive published experimental researches on this question, conducting of the specified measures precede transpiring of features of process of structuration at mass crystallization of the given product (fertilizer), definition of character of defective structure of crystal blocks (crystals), composing granules of fertilizer and diffusion of water-salt components (WSC), carried in crystals. The diffusion stream has the sources and flows as which any cracks serve, pores, dints, including - points of targeting of granules. These factors "control" migration of equated ions and water to fertilizer blankets, causing its consolidation.

The thermodynamics of the superficial phenomena connects these factors with passage of process of diffusion to a direction of leveling of a landform of a surface of the firm phase, accompanying decrease of magnitude of free superficial energy.

Such model of process of consolidation served basic for working out of two directions in the solution of its elimination. According to first from them, it is recommended to conduct a product graining so that to suppress traffic of dislocations and their outcropping of crystals, to brake a volume diffusion of ions to a surface. It can achieve by introduction no coherent on crystal lattice parameters crystals some fertilizer non deep dispersion the impurity, dislocations braking traffic not only in the course of formation of structure of granules, but also at the

further mechanical affecting on a product. The second direction in elimination of consolidation of granules consists in application of one of known ways of a retardation of a surface diffusion. Application of following methods Is recommended: 1. Drawing on a surface of granules high hygroscopic substances (for example, a diatomite) that leads to destruction WSC; 2. Dusting of granules by hydrophobic substances which, sorption on the active centers in places of an exit of dislocations on a surface, block them and prevent formation WSC; 3. Drawing on granules of the surfactants giving to a surface water repellency and preventing process of surface diffusion WSC; 4. covering surfaces of granules the polymeric films also interfering diffusion WSC in a zone of contact of granules.

At implementation of the specified ways of decrease in consolidation of the grained product it is necessary to consider agency of changes of environment also. It is installed that as a result of such affecting origination of various physical structures and diffusion the processes resulting, in the final reckoning, to new, more resistant to equilibrium state of system with a minimum of a free surface is possible. The reason of such phenomena consists in ability of mineral fertilizers in certain conditions to be formed even at rather small loadings. These processes remain while a little studied in spite of the fact that on them the greatest is necessary numbers of unsatisfactory equipment claims of users.

The similar phenomena (formation of monoliths) can be called also recrystallization or reorganization of a surface of corpuscles in the field of local distortion where a bonding force responsible for stability of a crystal lattice, can relax, raising probability of a leakage of reactions.

Practical recommendations at sampling of a technological regime of a graining and the maximum decrease in consolidation are reduced to that it is necessary to set in advance certain type of structure and a size of granules, considering thus presence of inducted modifications which definitely influence formation of structure of a target product. Already many experimental data

specifying are saved up that many additives in paucities render a great influence on properties of mineral fertilizers.

From resulted obviously that processes of a grain, elimination of consolidation of mineral fertilizers represent extremely a challenge. She demands cumulative studying of many factors accompanied by deformations of a crystal lattice of granules, diffusion WSC, changes of contacts and communications between structural elements (ions) of fertilizers. The big role is played thus by the chemical nature of the components which are a part of fertilizer: presence of them some them promotes reaction with a generating of anions and their migrations from one phase in another or from a boundary of granules in a homogeneous phase. Are that, for example, четвертичные аммониевые joints-ammonium sulphate, sulphate-nitrate of ammonium, etc. The mechanism of such processes is studied while insufficiently and in each specific case sampling of a regime of a grain and selection of modifications should be spent empirically with the maximum account of all factors set forth above, and also mineral and a chemical compound of raw materials and a product of production engineering of its manufacturing.

3.2. Mar tempering of property of an ammonium nitrate carbonate inclusive an additive

The way of reception of the stabilized ammonium nitrate - lime -ammoniacal saltpeter - by mixture solutiona nitrate of ammonium with ground limestone or a dry carbonate of calcium or others with the subsequent grain and air-conditionings of a surface of granules by dusting by a calcareous flour (china-clay, a diatomite, etc.) the gained weight is known.

On a known way solution ammonium nitrate mix with ground (high dispersion) carbonate raw materials. In the capacity of raw materials apply limestone or the dry carbonate of calcium containing not less of 94 % of CaSO_3 and no more of 1,5 % SiO_2 , 0,5 % MgO , 1,5 % $\text{Al}_2\text{O}_3+\text{Fe}_2\text{O}_3$ and 3,0 % of a moisture. The

ground calcareous flour is gained by limestone crushing on jaw crushers, by drying by top internal gases at temperature 400-700 °C in a rotary drier and grinding in ball mills. The calcareous flour should meet certain demands - through a sieve with 2500 apertures should pass 15 % of a flour, and the rest on ситax thus should be 7 %, moisture content should be not above 0,05 %. Process of preparation of a calcareous flour is difficult and power-intensive. Calcareous flour and solution an ammonium nitrate mix, the gained weight depending on power of manufacture grain in a drum- granulate or on granulation and condition a surface of granules by dusting by a calcareous flour (or other dusting substances - china-clay, or a diatomite or others). The maintenance carbonate inclusive additives in a finished stock makes 22-41,5 %. The nitrogen maintenance in an ammonium nitrate fluctuates within 18,0-28 %.

At reception of lime -ammoniacal saltpeter there is an interacting carbonate inclusive additives - limestone with solution of ammonium nitrate to formation high hygroscopic nitrate of the calcium negatively influencing commodity properties of a finished stock. This results from the fact that well soluble salts of nitrates of ammonium and calcium under the influence of an atmospheric moisture on a surface of granules of an ammonium nitrate form pregnant solutions. At oscillation of temperature of air pass processes of dissolution and crystallization of salts of pregnant solution, therefore granules start to slump.

Air-conditioning of a surface of granules of saltpeter by dusting by a calcareous flour (or other dusting substances) does not give expected results as the dusting substance, in the absence of a binding reagent, is badly kept and does not cover a surface of granules, i.e. do not form a protective layer. The insignificant adhered quantity on a surface of granules at its transportation and storage is showered from granules and accumulates in the bottom layers of a finished stock. The gained product more гигроскопичен, than pure saltpeter and at long storage slumps.

Besides, regular application carbonate inclusive an ammonium nitrate in carbonate soils is inexpedient, as, a calcium carbonate usually apply to neutralization (lime pretreatment) of sour soils.

3.3. Mar tempering of property of an ammonium nitrate high carbonate a phosphoric flour

For reception of difficult azotic-phosphoric fertilizer of the improved property on the basis of an ammonium nitrate in the capacity of carbonate inclusive additives used высококарбонатную a phosphoric flour of a standard flour milling - the rest on a sieve 16 no more than 30 % at humidity not less than 1 %, special machining as in lime-ammoniacal saltpeter is not required.

In vitro 99,5 % solution ammonium nitrate mixed with high carbonate a phosphoric flour in number of 2 and 4 % from weight of nitrate of ammonium and the gained weight grained. At mixture solution a nitrate of ammonium with high carbonate a phosphoric flour there is a difficult interacting of nitrate of ammonium to phosphorite components. Insignificant quantities of nitrate of calcium and calcium phosphate are formed. Presence as a part of a finished product - an ammonium nitrate of phosphate salts improves physic mechanical properties of a product. Granules of the stabilized ammonium nitrate consist of nitrate of ammonium, nitrate of calcium, phosphate of calcium and the activated phosphorite.

The further mar tempering of commodity properties of the ammonium nitrate gained granules carried out dusting a surface high carbonate a phosphoric flour in number of 10 and 20 % from weight of nitrate of ammonium in the presence of pregnant solution of sulphate of ammonium. The solution of sulphate of ammonium is a connecting reagent.

High carbonate the phosphoric flour can be used for dusting of granules of the industrial ammonium nitrate released in accordance with GOST 2-85.

Table 3.4

Properties of the fertilizers gained on the basis of an ammonium nitrate

Ammonium nitrate	The maintenance, %		Strength of granules, kg/sm ²	Consolidation, Kg/sm ²
	N	P ₂ O ₅		
Without an additive	34,6	-	0,7	2,1
With a limestone additive, 10 %	30,0	-	1,1	1,2
The same of 20 %	27,6	-	1,2	1,3
With an additive of phosphorite of 10 %	30,2	1,8	1,5	no cloding
The same of 20 %	27,8	3,9	1,5	no cloding

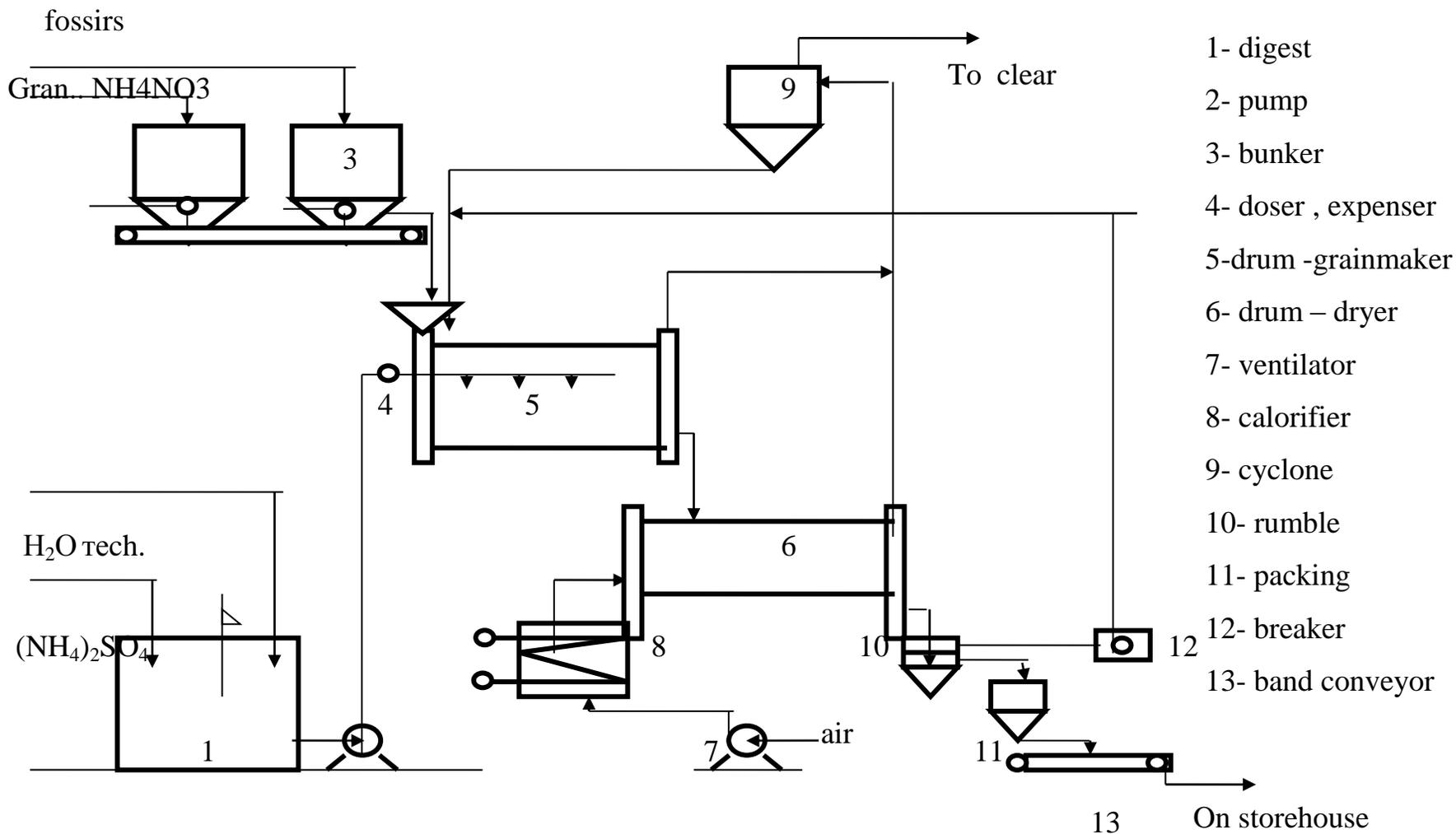
At a surfacing of granules of an ammonium nitrate the last fills with a solution of sulphate of ammonium micro pores (micro crack) of granules and strong links dusted substance on their surface, on a surface of granules of fertilizer the protective layer is formed. In the course of dusting on a surface of granules there is an interacting of a connecting reagent to components high carbonate a phosphoric flour. Sulphate of ammonium with an additive is formed by sulphate

of calcium and phosphate of calcium which cover with a thin layer a surface of granules, i.e. on a surface of granules the protective layer consisting of salts with smaller solubility is formed, it isolates hygroscopic salts of nitrates of ammonium and calcium of granules from an external circumambient. At the further surfacing of the gained granules the increase in a thickness of a protective layer occurs a connecting reagent and dusting substance. Because of absence on a blanket of the stabilized granule sweepingly soluble, hygroscopic salts - the ammonium and calcium nitrates, the gained product at long storage and humidity raise does not slump.

The gained product is effective on all types of soils. At application there is an activation of a phosphate mineral by an ammonium nitrate. All phosphorus and calcium high carbonate a phosphoritic flour pass in assailable forms. Thus gain difficult azotic-phosphor-calcium fertilizer. The granules of an ammonium nitrate processed in such a way keep stability at long storage.

Thus, at the organization of manufacture of the grained stabilized ammonium nitrate by use of easily accessible, low-cost raw materials – high carbonate a phosphoritic flour full use of energy of nitrate of ammonium for activation of phosphate raw materials is attained. Besides, on one technological thread it is possible to make difficult NPCa - the containing fertilizers meeting demands of agriculture.

Technological scheme of the reception nitric-phosphoric fertilizer



3.4 Mar tempering of physical and chemical and mechanical properties nitrophos

The way of reception of the difficult fertilizer containing nitrogen, phosphorus and calcium, for a way azotic acid decomposition of phosphate raw materials with quantitative transfer of all calcium of initial phosphate raw materials in calcium nitrate, removal from gained azotic acid down yards of phosphate of a part of nitrate of calcium a method of its cooling, neutralization of the sour azotic-phosphoric solution, containing residual calcium in the form of calcium nitrate, ammonia, evaporation of the neutralized solution before formation solution, drying and grains nitrophos is known. Deficiencies of a way are: the big charge of nitric acid for transfer of all calcium of phosphate raw materials in calcium nitrate; not possibility of use for reception of difficult fertilizer carbonate inclusive phosphorites because of ample foaming at a stage azotic acid their decomposition; Complexity of the circuit design and bulkiness of hardware registration of the process, connected with application for a stage of decomposition of the cascade of U-shaped reactors, presence of stages of crystallization of nitrate of calcium from azotic acid down yards a freezing, switching on forecastles of crystallizers and the assembly on manufacture of a cold, separation of crystals and their washings as a part of the filtrating equipment, separate rehash of firm and liquid phases accordingly on a limy ammonium nitrate and нитрофос.

The way of reception of difficult fertilizer by simultaneous di carbonization and decomposition high carbonate phosphorites of Central Kyzylkum in one apparatus is known also at norm of nitric acid of 25-100 % from a stoichiometry of reaction to full decomposition of CaO of raw materials. The residual acidity of a sour mash neutralize ammonia or high carbonate a phosphoric flour, and before drying and a graining in apparatus TGD, it is diluted with water within 5-30 % from its weight.

Deficiencies of a known way are: low efficiency and small productivity of knot of decomposition, not stability of a stage of drying and a grain of a product with the moistened weight formed as a result of the phenomenon congruence of fusion tetra hydrate of nitrate of calcium - of $\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$, crystallized from a mash in the course of its dehydration at a drying stage if for decomposition of phosphate raw materials nitric acid in stoichiometry norms of 60-100 % on CaO and concentration above 50 % is used, or a mash, before supply in siccative – granulation a drum, is diluted by addition in it to 30 % of water; not possibility of practical realization of process in the absence of use circuit design absorption a liquid formed at a stage of wet clearing, departing from a zone of drying and a product graining the gas with dust a stream.

Considering a deficiency of known ways on Open Society "SAMARQANDKIMYO" Independently functioning industrial line of difficult fertilizer (NCPHF – nitro calcium phosphate fertilizer) in the form of trial installation (EII) is put into operation.

At the heart of the production technology of difficult fertilizer - nitrophos the principle of decomposition of the Kyzylkum not enriched phosphoric flour incomplete norm of nitric acid lies. Interacting of components carry out in solution to medium with creation of the optimum concentration, temperature and time conditions promoting a total disappearance of free nitric acid in reaction particles.

Total reaction is presented by the equation:



According to this reaction the end-product consists of salts mono- and di calcium phosphates in a mix with nitrate of calcium with various extent hydrate. Last depends on conditions of crystallization of nitrate of calcium from a mash at выпарке and drying. The Azotic-phosphoric fertilizer gained from such mash, theoretically contains 33 % of water-soluble and 67 % solution of citrate forms P_2O_5 . From the general CaO containing in a product, 42 % are in the water-soluble form.

Fertilizer of such composition undoubtedly represents the big agrochemical interest.

57 %-nitric acid is applied to reception nitrophos.

Water in process is considered in two aspects:

1. Crystallization - enters into a crystal lattice of chemical substance ($\text{CaHPO}_4 \cdot 2\text{H}_2\text{O}$, $\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$).
2. Free - it is not connected in chemical compounds and does not enter into a crystal lattice.

The chemistry of process of decomposition of Kyzylkum phosphorites nitric acid at full decomposition of phosphorite by nitric acid proceeds reaction:



The quantity of nitric acid in the gramme-molecules, CaO of phosphate raw materials counted for a gramm-molecule on this reaction makes 2 gramme-molecules, i.e. a molar relationship of CaO: HNO_3 in a reactionary product should make 1: 2.

All sense of use of nitric acid in CaO specified under the relation is reduced to transfer неусвояемой forms P_2O_5 in an aspect fluor apatite in assimilable, mainly in water soluble P_2O_5 . At the further rehash of such mash in difficult fertilizers, with neutralization by ammonia, depending on extent of allocation from it calcium nitrate, water-soluble forms can pass in solution of citrate forms.

Features of phosphorites of the Kyzylkum deposit consists that the phosphate substance is largely presented flour hydroxi decarbonate apatite, with francolite and curcsite where phosphorus parts are isomorphic mixed in crystal lattices by carbon and to one atoms of oxygen hydroxide groups or atom of fluorine. Hence, at activation of such phosphorite at which there is a deformation of a crystal lattice of

a phosphate complex, the considerable part of phosphorus of phosphate substance should pass in assailable forms. So by researches it is installed that at decomposition ordinary high carbonated the phosphoric flour Kyzylkum containing 16-19 % P_2O_5 by nitric acid, at its norm of 50-60 % from a stoichiometry reaction of full decomposition of phosphate to phosphoric acid more than 50-60 % of phosphorus pass in assailable forms (mainly 90-95 % citrate-soluble and insignificant quantity in water solution forms).

Besides, the end-product though contains in the composition to 50 % of nitrate of calcium, besides the fact of quantitative transition of phosphorus in assailable the form, has satisfactory commodity properties i.e. not so slumps, keeps friability of granules.

For full decomposition of all CaO of phosphorite according to reaction (1) relationship of nitric acid in gramme-molecules on CaO makes 1:2. Considering that norm of the charge of nitric acid for phosphorite decomposition recommend within 50-60 % from a stoichiometry of the reaction (1), the given relationship in the reactionary should be in limits 1:1-1:1,2.

In these conditions process of decomposition of phosphorite by nitric acid is mainly presented by reaction:

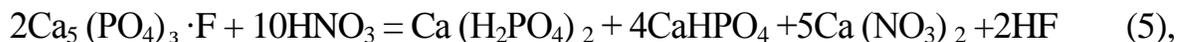


Where the molar relation CaO:HNO₃ makes 1:0,8. At a molar relationship of CaO:HNO₃ = 1: 1,4 reaction proceeds



Most likely, because of thermodynamic factors, in the beginning process there is a reaction (3), and then at excess of CaO in a reaction mixture, the part mono calcium phosphate passes in di calcium phosphate on reaction:

The general reaction of process can be presented as the sum of reaction (3) and (4);



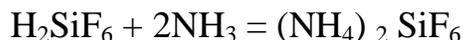
Where a molar relationship of CaO: HNO_3 in a mix makes as 1:1. Phosphoric acid formation is thus expelled.

Carbonates as a part of phosphorite are mainly presented in the form of calcite - $CaCO_3$. Because of a high reactive capacity of calcite in nitric acid all calcium connected with $CaCO_3$ passes in nitrate of calcium at the expense of reaction de carbonizatoin.



However in practice the nitric acid part though also its insignificant part, remains to the unreacted and additives at the expense of formation with silicon-fluorine-hydrogen acids on reaction (10), pH fluctuates medium within 1,5 - 2,0. For neutralization of the residual acidity of a mash it is exposed to an ammonation.

In these cases neutralization reaction is presented by the equations:



Almost gained end-product with aforementioned molar relationships of $CaO:HNO_3$ in initial raw streams as it follows from reaction (5), represents in the core, a mix of salts of $Ca(H_2PO_4)_2$, $CaHPO_4$ and $Ca(NO_3)_2$ and in matching гидратных

forms. Then proceeding from a stoichiometry (1 gramme-molecule of $\text{Ca}(\text{H}_2\text{PO}_4)_2$ and 4 gramme-molecules of $\text{Ca}(\text{NO}_3)_2$) under conditions when all necessary temperature, concentration and time factors for a full leakage of the given reaction are observed, theoretically from total P_2O_5 are in a finished stock of 33,3 % P_2O_5 in water-soluble and 66,6 % P_2O_5 in citrate of solution forms.

In such a manner that from general CaO in a finished stock of 60 % of CaO is in усвояемой and 40 % of CaO water-soluble forms.

On the basis of physical and chemical properties of four-water nitrate of calcium existing ways of its reception in the grained aspect in industrial conditions provide a stage steaming a solution of $\text{Ca}(\text{NO}_3)_2$ to concentration of 70-80 % passes in three - and calcium di hydrate.

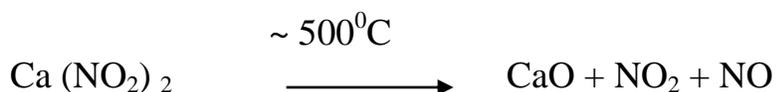
In composition nitrophos the maintenance of nitrate of calcium fluctuates within 35- 40 %. In a mash calcium nitrate is in a four-hydrate aspect. The grain and product drying is carried out in apparatuses TGD (a drum-type grain mill-dryer) in one stage by a method of spraying of a mash in the free-fall curtain in a stream of hot top internal gases. Therefore in apparatus TGD at its contact with top internal gases under the influence of high temperature of $\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$ instantly is exposed to a dehydration with transformation last in rub at calcium di hydrate. All process proceeds in solution. The temperature regime in TGD does not allow to remove a moisture from system in due time. Apparatus TGD 16 x 4,5M - tested, working in many manufactures dryer-granulation the equipment in diameter 4500, length 16000 mm, volume $25,4\text{M}^3$, is usually installed at an angle an inclination 3° , set in by redactor OGSCH - 900 M with gear ratio $Z = 41$ through electric motor AK-12-33 To power of 320 kw and a corrected speed $n = 1000$ rpm. Speed of twirl of a drum 0,47 rad./sec ($\sim 2,5$ rpm).

The firm phase of a product in the core consists from di calcium phosphatea (20-25 %), mono calcium phosphate (1-5 %), calcium nitrate (50-55 %) counting on anhydrous forms of salts. Anhydrous nitrate calcium is thermally resistant to

within temperatures more low 420⁰C. With an increase of temperature above 420⁰C, even at short contacts of small corpuscles to top internal gases, because of their thermo stability, they are exposed to thermal decomposition with formation of nitrite of calcium and allocation of free oxygen:



The further warming up of weight leads to decomposition of nitrite to nitrogen oxides:



The leakage of these reactions is involved in the beginning of process so-called «cigar-shaped burning». All weight in the apparatus starts to burn without a flame, reserving a white powder (CaO). There is a product loss. Besides, the gas phase becomes very aggressive in relation to materials TGD, flue gas paths and ventilating fans. Their intensive corrosion begins.

Proceeding from the above-stated, in a drying and graining regime nitrophos the temperature of top internal gases on an entry in apparatus TGD is recommended to be supported in limits 350-400⁰C. The second feature of a mix of salts nitrophos at their drying as is caused by presence in it of crystalline hydrates of nitrate of calcium. In cases of spraying of a mash nitrophos in a drum when in it the water maintenance is exceeded by 25 % and temperature more low 70⁰C, during transpiration of water from the stuck layer of a mash on surfaces of corpuscles the four-water form of nitrate of calcium - Ca (NO₃)₂ · 4H₂O crystalizes. In process of an increase of temperature (> 70⁰C) tetra hydrate, gating out one molecule of water, begins solution in it (congruence fusion) and in due course all weight in the apparatus turns to a fluid mash.

Process of drying of a product stops. The same picture repeats and in concentration and temperature conditions of Crystallization tri hydrate calcium nitrate. Hence, one of necessary conditions of drying and a product graining is hit exclusion in a zone распыла diluted ($> 25\%$ H_2O) and chilled ($<70^{\circ}C$) nitrophos mashes. At dehydration of a layer of a mash on surfaces of corpuscles di hydrate of nitrate of calcium or on extreme to a measure its mix with tri hydrate should crystallize. In the conditions of drying specified above process of a graining of a material occurs well. On an exit from apparatus TGD a product mechanical grading following (mas. %): granules a size more than 6 mm - 10-12 %; from 1 to 4 mm - 70-80 %; less than 1 mm - 5-15 %. Strength of granules of commodity fraction (1-4 mm) makes not less than 3,0 kgs/sm².

Physical and chemical and commodity properties nitrophos have great value at its storage, conveyance and entering into soil. In this connection we had been studied the basic physical and chemical properties of some samples nitrophos depending on their shelf life

The table 3.5

Physic - chemical and mechanical properties nitrophos depending on shelf life

Numbers of samples	Shelf life, days	Humidity, %	hygroscopic point, %	Extent clod – peafowl, %	Strength of granules, MPa
1	10	0,80	28,60	0,50	3,60
	20	1,10	31,30	0,95	3,35
	30	1,60	35,30	1,80	3,10
2	10	0,75	30,8	0,65	3,45
	20	1,55	35,35	2,15	3,06
	30	3,65	40,10	3,80	2,91

In composition nitrophos the maintenance of nitrate of calcium on the average makes 30-40 %. Therefore a hygroscopic point of fertilizer underrated (30-40 %). But at storage in warehouse conditions its hygroscopic point, strength of granules and consolidation almost changes not considerably.

On value of a hygroscopic point nitrophos to refer to strong enough to hygroscopic substances. In this connection it is recommended in summertime its storage and a transport in bulk, and in autumnal- clod a time to pack in bitumen or polyethylene sacks.

Physical and chemical and mechanical properties (hygroscopic the moisture capacity, consolidation, strength) granules are defined for the following fertilizers gained on the basis of decomposition of not enriched Kyzylkum phosphoritic flour accordingly at norms of nitric acid 37, 50 and 60 %. The Chemical compound of samples nitrophos is resulted in tab. 6

The table 3.6

Chemical compound nitrophos depending on norm of nitric acid

Norm HNO ₃	P ₂ O ₅ gen.	P ₂ O ₅ assim.	P ₂ O ₅ waterway	CaO gen.	CaO assim.	CaO waterway	N
40	15,12	6,87	0,5	40,67	31,63	14,55	7,74
50	14,03	7,71	0,74	37,72	29,83	13,78	9,87
60	13,08	8,13	0,76	35,19	29,17	13,59	11,57

Hygroscopic point of fertilizers defined desiccator in the way N.E.Pestov at temperature 25⁰C, for granules a size of 2-3 mm. It is installed that hygroscopic points make for examinees of fertilizers № 1 - 35 %, № 2 - 30 % and № 3 - 24 %, at initial humidity of 1 %. It is known that hygroscopic of fertilizers in certain extent depends on their humidity, presence of water-soluble salts, etc. By us are defined humidity agency nitrophos on its hygroscopic point

Hygroscopic point nitrophos depending on humidity

Numbers of samples nitrophos	Hygroscopic points nitrophos at humidity, %					
	1	2,5	4	5	6,5	8,0
1	35	43,5	49,0	52,0	60,0	66,0
2	30	42,0	45,5	48,0	56,0	60,0
3	24	38,0	40,0	42,0	51,0	56,0

Data shows that with moisture content increase in samples nitrophos their hygroscopic points considerably increase. For example, for nitrophos (№ 1, 2 and 3) the increase in humidity from 1 to 8 % promotes increment of a hygroscopic point from 35, 30 and 24 to 66, 60 and 56,0 % of a relative humidity of air accordingly.

On values of hygroscopic points gained nitrophos at norms HNO_3 37, 50 and 60 % from a stoichiometry refer to hygroscopic substances. However, as these values match to low natural humidity of a free air fertilizers at storage will not be exposed to sharp transitions from moistening to dry out. It in turn interferes with fall from a film of pregnant solution of the salts cementing crystals and reduces ability of granulated fertilizers to caking. Proceeding from value of a hygroscopic point, fertilizer practically will be always moistened, in communication, with what it is recommended to store in 4x layer polyethylene sacks.

It is necessary to note that values of a hygroscopic point of products define necessary conditions of their storage, conveyance and application. However hygroscopic of fertilizers is not the only stipulation for the solution of a question on possibility of their application. The moisture capacity and other factors have great value also.

Definition sorption moisture capacities nitrophos spent on M.E.Pestova's method at 25°C and in the interval moistures of air from 10 to 100 %. The size of

granules was 2-3 mm. Thanks to a thin layer of a product and affinity of its distance from a surface of sulphuric acid of certain concentration sorption water steams occurred rather sweepingly. Results shows that practically depending on an aspect of fertilizers and a hygroscopic point at initial humidity sorption begins at 15 - 20 % of a relative humidity of air.

At 100 %-s' humidity of air balance is not installed within a month.

Kinetics sorption water vapors nitrophos at the minimum mid-annual and maximum relative moistures 41; 61 and 80 % (in the conditions of average Asia) shows that at a relative humidity of 41 %, balance practically stepped in 7 days, at 61 % in 9 days, at humidity of air of 80 % in 13 days. The big moisture capacity of fertilizers probably speaks presence in them hygroscopic water-soluble salts: nitrate of calcium, a magnesium of monophosphate of calcium, ammonium, a magnesium, nitrate of ammonium and water-soluble salts of phosphate of calcium, a magnesium, etc.

Consolidation defined on N.E.Pestova's method according to which moistened at the maximum monthly average relative humidity of air of 80 % fertilizer of a certain mechanical grading $3 > 0,1 > 1$ mm fell asleep in gauze sacks and molded in the special device at certain loading within one days. In a day fertilizer, without pulling out from sacks, exsiccated at temperature 1050C to constant weight, then dumped from altitude of 1,5 m and sieved through a sieve with a diameter 3 mm. Conditional consolidation defined depending on initial humidity and from loading. At initial humidity to 1,5 and at moistening to 3 % of the sample nitrophos practically do not slump. It is possible to explain it to that the hygroscopic point of the recommended fertilizers lies in the field of lower relative humidity, than an aerosphere relative humidity. Therefore at storage in the conditions of oscillation of natural humidity of an aerosphere of air, fertilizer are not exposed to the periods from moistening to dry out that interferes with fall from

a film of pregnant solution of salts of the crystals cementing a product and causing its consolidation.

Finding of the third part.

For the purpose of property mar tempering nitrophos the production engineering, different from known is offered, phosphorite decomposition spend at incomplete norm of nitric acid without allocation of nitrate of calcium in the presence of water (absorption liquids) with the subsequent steaming mashes to density 1,7-1,8/sm³. The technological circuit design for dusting a surface of granules of the nitrogenous and difficult fertilizers, containing nitrate of calcium, high carbonate by a phosphoritic flour in the presence of a solution of sulphate of ammonium is offered.

FINDINGS

1. For the purpose of the further improvement of quality and development of production engineering of reception of an ammonium nitrate with a phosphoric flour the new way of introduction of an additive is offered.

2. Research of physic mechanical and commodity properties of samples with various additives showed that the ammonium nitrate with high carbonate a phosphoric flour does not slump and possesses high strength of granules and stability at repeated modification transformations and long storage.

3. It is installed that in process of formation of granules of an ammonium nitrate an inducted flour carry out a role of crystallization centers and reduce warmth of Crystallization solutiona. They brake kinetics of modification transitions and promote metastable transformation IV=II at rather high humidity of an ammonium nitrate and to reception of granules with high extent of crystallinity and density.

4. For the purpose of property mar tempering nitrophos the production engineering, different from known is offered, phosphorite decomposition spend at incomplete norm of nitric acid without allocation of nitrate of calcium in the presence of water (absorption liquids) with the subsequent steaming mashes to density 1,7-1,8/sm³

5. The technological circuit design for dusting a surface of granules of the nitrogenous and difficult fertilizers, containing nitrate of calcium, high carbonate by a phosphoric flour in the presence of a solution of sulphate of ammonium is offered.

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