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Composition and Properties of NPK-Fertilizers Produced on the Basis of an Ammonia Saltpeter Solution, A Ground Phosphate Rock and Potassium Chloride

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Abstract

The present article contains the research results of NPK-fertilizers production. The ammoniated solution of the ammonia saltpeter manufacture formed at the first evaporation stage was applied as a basic substance, and a ground phosphate rock and potassium chloride as phosphorus and potassium-containing components. Scientific and applied importance of the research consists in the manufacture of fertilizers according to the traditional ammonia saltpeter production scheme in compliance with the technological regimes of ammonia saltpeter primary evaporation, drying and granulation stages. The method of controlling the nutrients' ratio in the end product was developed. The possibility of manufacture of the NPK-fertilizers with a regulated N:P2O5:K2O ratio was revealed. It was found, that the calculation data on the regulation of the nutrients ratio in the products well agree with the experimental results; the average deviation between them does not exceed 1,0 %. The composition and basic physicochemical properties of the target products were studied; discharge coefficients of the initial components per 1 t of the product were determined. The quality and physicochemical properties of the NPK fertilizers produced full enough meet qualifying standards of commercial fertilizers. In addition, in comparison with ammonia saltpeter they have improved consumer properties and higher agrochemical value.

Keywords: Ammonia saltpeter, ground phosphate rock, potassium chloride, nutrient, fertilizer.

I. INTRODUCTION

Wide application of ammonia saltpeter in agriculture is caused its high fertilizer value as it contains 34,4-34,5 % of nitrogen in ammonium and nitrate forms in equal portions[1,2]. It is an effective fertilizer for all climatic zones and practically all crops. But it has one very serious disadvantage – explosion hazard [3-5]. Under the influence of some external factors it can detonate. The explosion of ammonia saltpeter in warehouses of the chemical enterprise in Toulouse (France) in 2001, which reasons did not found out up to now, a number of terrorist acts in Southeastern Asia and Russia with use of ammonia saltpeter have put consumers and manufacturers of this product in a complicated position. For this reason a number of the countries – China, Philippines, Colombia, and Ireland – prohibited application of ammonia saltpeter as a fertilizer for agriculture [6-8].

In this connection, requirements to ammonia saltpeter quality and conditions of its storage [9-12] were toughened, and its manufacturers received a new task – to develop the ammonia saltpeter based fertilizers, which keep its fertilizer efficiency and have higher stability to external actions and, accordingly, lower explosion hazard [13,14]. At the same time, it was recommended to take into consideration the well-known fact that the uncontrolled application of nitric fertilizers leads to accumulation of nitrogen in soil that promotes mineralization of the organic matter of the soil and, as a consequence, causes ingress of nitrates in the composition of agricultural products from the soil [15]. Therefore, to achieve rich and high-quality harvests it is necessary to apply balanced mineral fertilizers, i.e. containing, besides nitrogen, phosphorus and other

nutritious components in reasonable ratios, i.e. so-called complex fertilizers which major characteristic is their high agrochemical value [16,17].

Thus, at present, manufacturers of the major fertilizer – ammonia saltpeter – should decide the urgent question which basic essence consists that this product according to its consumer and physicochemical properties and also agrochemical value not full enough meets modern requirements of its consumers [18,19].

Long-term researches of agrochemists, and soil scientists have substantiated a multipurpose role of phosphorus and potassium for life of plants. Phosphorus accelerates development of plants, stimulates processes of fertilisation, formation and maturing of fruits, raises crop capacity and quality of production. Potassium has the very important physiological value in the carbohydrate and protein metabolism of plants improving conditions of application of the ammonium form of nitrogen. Potassium is one of important nutrients; it promotes sugar accumulation in cellular fluid, deposition of starch in tubers raising thereby quality of fruits, cereals, vegetables and other crops. The presence of potassium in soil increases the winter hardiness of plants and accelerates the development and thickening of their cells. It is well-known, that one hectare of arable land on the average takes 80 kg of nitrogen, 40 kg of phosphorus and 70 kg of potassium at the crop capacity of 15 metric centners. For this reason, at present, there is a great demand for the fertilizers containing nitrogen, phosphorus and potassium. Scientists carry out numerous studies connected with addition of phosphorus and potassium-containing components in ammonia saltpeter melt [20-25]. These components are natural minerals, the products obtained at their industrial processing or phosphorus and potassium-containing technogenic wastes [26-30].

The only manufacturer of ammonia saltpeter in the Republic of Kazakhstan is "KazAzot" JSC (Aktau, Kazakhstan). To solve the above mentioned problem on the improvement of ammonia saltpeter agrochemical value and consumer properties the own phosphate ores of the Republic of Kazakhstan can be successfully applied. As is known, the Republic of Kazakhstan on reserves of phosphate raw materials is the largest raw-material base of Eurasia [31,32]. The major part of Kazakhstan phosphates is applied for manufacture of a ground phosphate rock. It is a dry, dust-forming powder of grey, yellowish or brown colour. It is slightly hygroscopic, does not cake. The average chemical composition of ground phosphate rock: CaO – 33 %, SiO2 – 35 %, P2O5 – 17 %, Fe2O3 – 2,1 %, K2O+Na2O – 1,6 %, MgO – 0,6 %, SO3 – 1,5 %, Al2O3 – 2,3 %. Fineness (the residue on a 0,18 mm mesh) is 10 %, humidity – 1,5 %, bulk weight – 1,5-1,8 t/m3. "Temir Service" LP in Aktyubinsk processing the Chilisay phosphorite produces 200 ths t of a ground phosphate rock per a year [33].

Kazakhstan has also sufficient resources of potassium minerals basically as natural potassium chloride, sylvinite and carnallite in Aktyubinsk Oblast (Zhilyanskoye, Chelkar and Satimola deposits), and also over 2500 high-mineralized brine lakes [34,35].

According to practical operation data of "KazAzot" JSC [36], the ammonia saltpeter solution, obtained in an ammonizator at neutralisation of nitric acid with ammonia under the vacuum, has pH of no more than 2 and concentration of 62-64 %. This solution is fed to the primary evaporation, realized under the vacuum of 0.2-0.3 atm and temperature of 110oC; in the process its concentration raises to 71 %. At the same time, as is known [37], ammonia saltpeter is partly decomposed forming the initial substances – ammonia and nitric acid. Consequently, owing to practically complete evaporation of ammonia under the specified temperature conditions, the evaporated solution keeps the acid medium that is undesirable since negatively affects consumer properties of commercial ammonia saltpeter. Therefore, the technology provides for its additional ammoniation with gaseous ammonia at the secondary evaporation stage that increases the end-product cost price.

According to a draft proposal of the "KazAzot" JSC concerning to determination of new possibilities of improvement of ammonia saltpeter agrochemical and consumer properties, the scientific personnel of the chair "Chemical technology of inorganic substances" of M. Auezov South Kazakhstan State University (Shymkent, Kazakhstan) has implemented the research on production of new NPK fertilizers on the basis of the ammonia saltpeter solution, Chilisay ground phosphate rock and the most available potassium salt – potassium chloride. Under the agreement with the customer, the ammonia saltpeter solution obtained in accordance with the traditional ammonia saltpeter technology at the first evaporation stage was applied as an initial component. A list of the additional mineral additives influencing on physical and mechanical properties of the end products was also co-ordinated with the customer. The agreed content of nitrogen, phosphorus pentoxide and potassium oxide in the end products were 10-18 %, 6-9 % and 6-9 %, respectively.

II. EXPERIMENT TECHNIQUE

The composition and properties of the target products were studied at the laboratory conditions using standard methods:

- total nitrogen content in the NPK fertilizer obtained – in accordance with State Standard 30181.6-94;
- total, assimilable and water-soluble P2O5 content in the NPK fertilizer obtained – in accordance with State Standard 20851.2-75;
- potassium mass fraction in the NPK fertilizer obtained – in accordance with State Standard 20851.3-93;
- moisture content in the NPK fertilizer obtained – in accordance with State Standard 20851.4;
- strength of the fertilizer granules – device IPG-1M;
- pH – device I-160 MI.

In addition, other modern devices for implementation of physical and chemical analysis were applied: a scanning electronic microscope JEOL of JSM6490LV type, a

spectrophotometer SPECORD-75, an infrared Fourier spectrometer Shimadzu IR Prestige-21, an X-ray phase analyzer DRON-3, a moisture meter Mettler Toledo, a measuring instrument of granules' strength IPG-1M.

III. RESULTS AND THEIR DISCUSSION

The research was carried out under the laboratory conditions of the department "Chemical technology of inorganic substances" of M. Auezov SKSU and at a trial plot of the operating ammonia saltpeter manufacture ("KazAzot" JSC) on the basis of the 71 % ammonia saltpeter solution, ground phosphate rock, potassium chloride and modifying additives (iron and ammonium sulphates) in compliance with regime parametres of the operating manufacture for the stages of primary evaporation of the ammoniated ammonia saltpeter solution and its subsequent drying and granulation. 4 samples of NPK-fertilizers were synthesized. The initial quantities of ammonia saltpeter, phosphorite, potassium chloride and mineral additives, necessary for production of the NPK fertilizers with the desired N:P2O5:K2O ratio, were calculated under the following analytical expressions:

$$M_{prod} = \frac{ax}{c};\ b = \frac{M_{prod} \cdot d}{y};\ e = \frac{M_{prod} \cdot f}{z};$$

$$m_d = M_{prod} - (a+b+e)$$

where: – weight of the NPK fertilizer with the regulated nutrients ratio;

a – weight of the ammonia saltpeter contained in the calculated volume of the initial 71 % ammonia saltpeter solution;

x – the nitrogen content in the ammonia saltpeter determined in accordance with State Standard 2-2013, %;

c – the expected nitrogen content in the NPK fertilizer produced, %;

b – the weight of phosphate additive necessary for mixing with the calculated volume of the ammonia saltpeter initial solution;

y – known content of P2O5 in the phosphate additive of FM-2 grade determined according to the "Temir-Service" LP standard 930640000252-01-2011, %;

d – expected P2O5 content in the NPK fertilizer produced, %;

e – the weight of potassium chloride necessary for mixing with the calculated volume of the ammonia saltpeter initial solution;

z – known K2O content in potassium chloride according to technical regulations 2184-048-00203944-2014, %;

f – expected K2O content in the NPK fertilizer produced, %;

md – the total mass of modifying mineral additives.

The research results, data about a chemical composition and basic physical and chemical properties of the target products obtained and calculated discharge coefficients of initial substances per 1 tonne of the end product are represented in tables 1-3.

Table 1: Calculated and experimental results

№	Nutrients content in the NPK product (calculation), mass %			Nutrients content in the NPK product (experiment), mass %			Divergence of calculation and experimental results, %		
	N	$P_2O_{5\ total}$	K_2O	N	$P_2O_{5\ total}$	K_2O	N	$P_2O_{5\ total}$	K_2O
1	10,0	9,0	9,0	9,57	9,74	8,65	-0,43	+0,74	-0,35
2	15,0	7,5	7,5	14,68	8,00	7,20	-0,32	+0,50	-0,30
3	16,0	7,0	7,0	15,96	6,75	6,65	-0,04	-0,25	-0,35
4	18,0	6,0	6,0	17,82	5,98	5,75	-0,18	-0,02	-0,25

Table 2: Discharge coefficients of the initial substances per 1 tonne of the end product and nutrients content in it

№	Masses of initial substances					Nutrients content in the end product	Nutrients mass ratio in the end product
	AS, t	H_2O, t	GPR, t	KCl, t	Mineral additive, t	$N/P_2O_5/K_2O$, %/%/%	$N/P_2O_5/K_2O$
1	0,291	0,119	0,529	0,145	0,291	10/9/9	1,1/1/1
2	0,436	0,178	0,441	0,121	0,436	15/7,5/7,5	2/1/1
3	0,465	0,190	0,412	0,113	0,465	16/7/7	2,28/1/1
4	0,523	0,213	0,353	0,096	0,523	18/6/6	3/1/1

Table 3: Composition and basic physicochemical properties of the NPK fertilizers produced

№	Nutrients mass ratio in the product, $N:P_2O_5:K_2O$	Nutrients content in the product, %				Humidity, %	Strength of granules,N/gran	pH of 10% solution	Granulometric composition, mass %	
		N	P_2O_{5total}	$P_2O_{5assimilable}$	K_2O				1-4 mm	2-4 mm
1	1,1:1:1	10	9,0	7,25	9,0	0,17	66,15	6,65	89-91	78-80
2	2:1:1	15	7,5	6,43	7,5	0,16	62,47	6,55	92-98	81-88
3	2,28:1:1	16	7,0	6,00	7,0	0,15	60,65	6,50	93-98	83-89
4	3:1:1	18	6,0	5,14	6,0	0,17	58,68	6,47	93-98	84-89

As follows from table 1, the calculated and experimental values of nutrients content in the end products agree with each other. The average deviation between them does not exceed 1,0 %. The data of tables 2 and 3 show, that the NPK fertilizers produced full enough meet the standard requirements to common fertilizers.

The NPK fertilizers produced were studied using physical and chemical analysis techniques. The results of scanning electronic microscopy, infrared microscopy and diffraction thermal analysis of a sample of the NPK fertilizer with the nutrients ratio of 16:7:7 are represented in fig. 1-3 and in tables 4-5. Other NPK-fertilizer varieties are characterized by identical results.

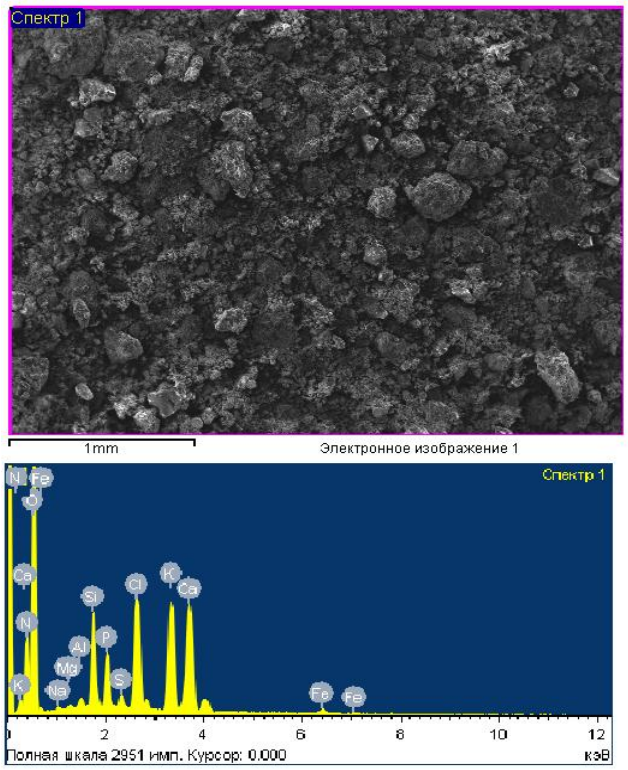

Fig. 1. The microstructure photograph of the NPK fertilizer (16/7/7) obtained at 40-fold magnification

Table 4: Elemental mass composition of the NPK fertilizer (16/7/7)

Element	Mass %	Atomic %	Mass composition in terms of oxides, %
N	25.94	32.85	
O	49.46	54.84	
Na	0.26	0.20	0.35
Mg	0.19	0.14	0.32
Al	0.34	0.23	0.64
Si	2.28	1.30	5.22
P	3.18	2.01	6.80
S	0.48	0.27	1.20
Cl	4.82	2.41	
K	5.85	2.65	7.05
Ca	6.56	2.90	9.18
Fe	0.64	0.20	0.92

For more careful studying and obtaining of detailed information on the composition and structure of individual components of the fertilizer sample in a solid phase, it was analyzed by a scanning electronic microscopy (SEM) method. The SEM photographs and their spectra are represented in fig. 1. It was found that the sample contains 12 chemical elements (N, O, Na, Mg, Al, Si, P, S, Cl, K, Ca, and Fe) in various quantities. The average elemental mass composition of the NPK fertilizer (16:7:7): CaO – 9,18 %, K_2O – 7,05 %, P_2O_5 – 6,80 %, SiO_2 – 5,22 %, SO_3 – 1,20 %, Fe_2O_3 – 0,92 %, Na_2O – 0,35 %, Al_2O_3 – 0,64 %, MgO – 0,32 %.

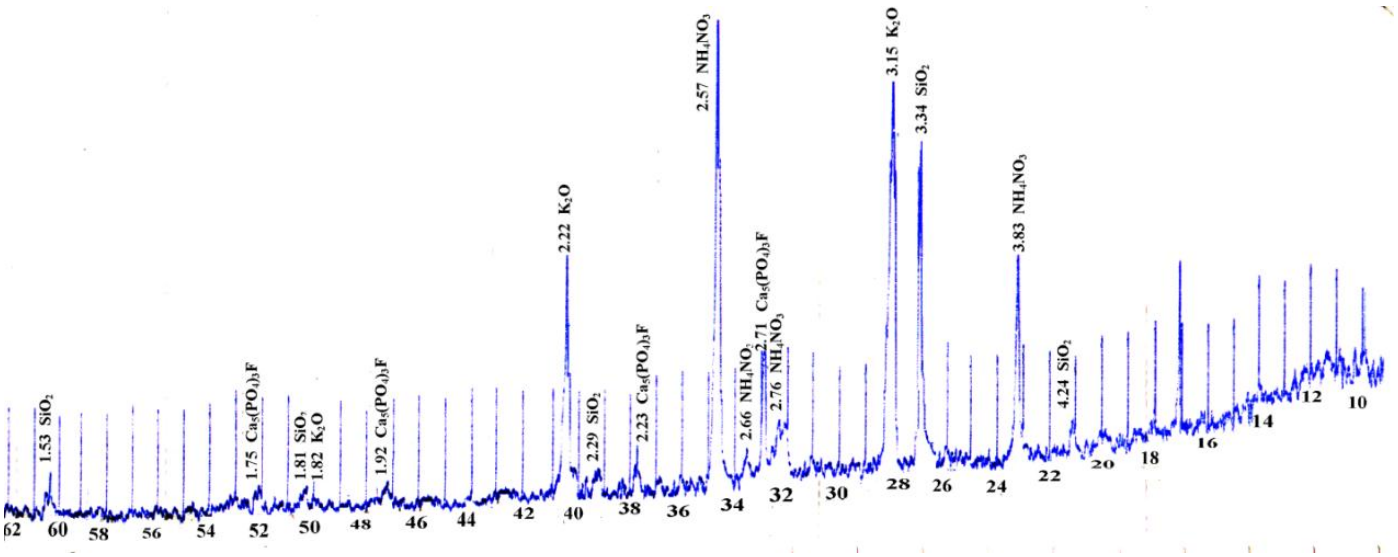

Fig.2. The roentgenogram of the NPK sample (16/7/7)

The X-ray phase analysis of the sample (fig. 2) was implemented on a diffractometer DRON-3(a general-purpose X-ray diffractometer) with an x-ray tube 1,5BSV29Cu with copper radiation and a nickel filter. The range of displacement angles of a detector (diffraction angles from 8° to 64°) was counted according to a scale of a goniometric device and marks on a diagram tape of a recording potentiometer. Rotational velocity of the counter is 8°/min. The roentgenograms were taken at the tube pressure of 30 kV and current strength of 20 mA. The samples were crushed to passage through a sieve of 10000 apertures per cm2. In the X-ray picture the diffraction maxima with values of interplanar spaces of d/n=3.83-2.76-2.66-2.57Â are typical of ammonium nitrate NH4NO3 – the basic phase. The diffraction maxima with d/n=2.71-2.23-1.92-1.75 Â belong to fluorapatite Ca5(PO4)3F, the interplanar spaces d/n=3.15-2.22-1.82 Â are typical of KCl, the presence of quartzite SiO2 is proved by d/n=4.24-3.34-2.29-1.81-1.53Â.

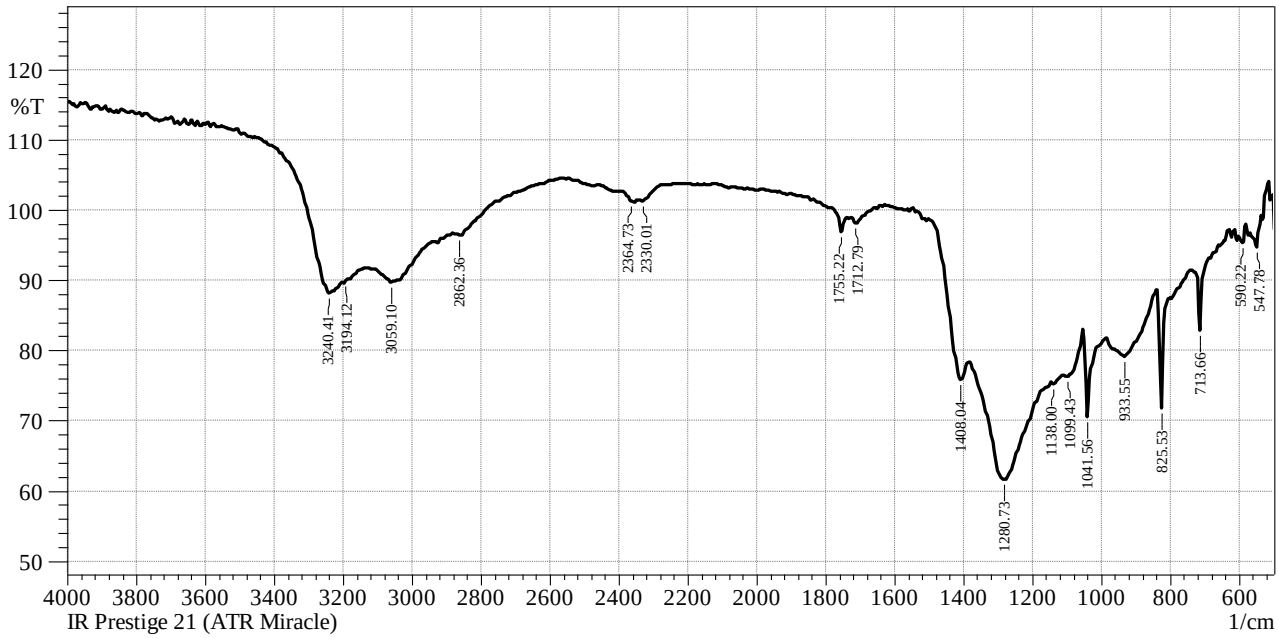

Fig.3. The infrared spectrum of the NPK fertilizer (16/7/7)

Table 5: Decoding of the infrared spectrum of the NPK fertilizer (16/7/7)

No.	Peak	Intensity	Corr. Intensity	Base (H)	Base (L)	Area	Corr. Area
1	547.78	94.763	3.587	567.07	536.21	0.505	0.238
2	590.22	95.322	1.804	601.79	578.64	0.390	0.097
3	713.66	82.805	9.049	725.23	659.66	2.537	0.523
4	825.53	71.792	17.153	837.11	740.67	5.919	1.530
5	933.55	79.148	2.985	960.55	840.96	10.135	1.378
6	1041.56	70.597	11.178	1049.28	987.55	6.494	1.097
7	1099.43	76.240	1.133	1107.14	1053.13	5.732	0.472
8	1138.00	75.209	0.252	1141.86	1111.00	3.703	0.020
9	1280.73	61.573	15.546	1381.03	1145.72	37.428	10.548
10	1408.04	75.828	6.755	1496.76	1384.89	7.617	1.439
11	1712.79	98.086	1.094	1724.36	1674.21	0.246	0.118
12	1755.22	96.905	2.614	1782.23	1735.93	0.246	0.171
13	2330.01	101.205	0.597	2341.58	2276.00	-0.648	0.055
14	2364.73	101.274	0.173	2391.73	2360.87	-0.266	-0.008
15	2862.36	96.368	0.499	2870.08	2758.21	0.676	0.114
16	3059.10	89.679	3.087	3109.25	2939.52	6.181	1.290
17	3194.12	89.616	0.134	3197.98	3136.25	2.567	0.002
18	3240.41	88.228	4.897	3425.58	3201.83	0.657	-0.128

The same sample was subjected to infrared investigation for determination of structural features and impurity components of the fertilizer. The infrared spectra were taken using a spectrometer Shimadzu IR Prestige-21 with an attachment of frustrated total internal reflection Miracle (PikeTechnologies) in the frequency range of 600-4000 cm-1 (fig. 3).

The analysis of the absorption spectra of infra-red radiation shows the presence of the following bonds: P=O, P-O-P, P-O-Al, absorption bands of 1280-933 cm-1; P-F, bands of 900-825 cm-1; P-Cl, P=O-S, bands of 520-460 cm-1; PO4-3, HPO42-, H2PO4-, bands of 1100-950 cm-1; Si-O-Si, bands of 1099-1041 cm-1; OH-, bands of 3000-3240 cm-1. On the basis of these data one can say that the fertilizer contains fluorapatite, quartz (bands of 1099-1041 cm-1), N-H (bands of 3059-3140 cm-1), NO3- (bands of 1050 cm-1, 830 cm-1, 1390 cm-1,713 cm-1).

Generalizing the data of fig. 1-3 and tables 4 and 5 it is possible to draw a conclusion that the compounds produced on the basis of the ammonia saltpeter solution, ground phosphate rock, potassium chloride and modifying mineral additives represent new complex NPK fertilizers in which the content of nutritious components – N, P2O5 and K2O – can be changed in the wide range: nitrogen from 10 % to 18 %, phosphorus pentoxide and potassium oxide both from 6 % to 9 %. In this case the total content of the main nutritious elements will make 28-30 % that proves their high agrochemical value.

IV. CONCLUSION

Thus, the results of laboratory studies and industrial tests convincingly testify to the real possibility of manufacture of new nitrogen, phosphorus and potassium containing fertilizers on the basis of the ammonia saltpeter, ground phosphate rock ("Temir-service" LP) and potassium chloride. In comparison with ammonia saltpeter these fertilizers are characterized by both improved consumer properties and higher agrochemical value. In addition, the content of nutritious components (N, P2O5 and K2O) can be changed in the wide interval: from 10 % to 18 % for nitrogen and from 6 % to 9 % for P2O5 and K2O, and the total content of the main nutritious elements makes 28-30 %.

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