

Study of Interaction in Systems Consisting of N Oxy 2.6 Dimethylpyridine, Carbamide, Ammophos and Ammonium Derivatives

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Abstract

The UN World Population Conference noted the high rates of population growth in the world. UN experts noted that now the population of the Earth is 7.7 billion people, by 2050 it will exceed 9.7 billion people, and by the end of the century it will grow to 11 billion. This is stated in the UN report prepared for the 52nd meeting of the Commission on Population and Development of the Economic and Social Council [1].

Introduction

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Below is a list of countries in the world with projected populations as estimated by the UN Department of Economic and Social Affairs. The average version of the forecast for July 1, 2015, 2020, 2030, 2050 and 2100 is presented (Table 1).

Table 1. Countries of the world with projected populations

Countries	2015	2020	2030	2050	2100
World	7 383 008 820	7 731 626 626	8 322 708 853	9 300 358 925	11072713524
India	1 309 053 980	1 383 197 754	1 512 985 205	1 658 978 163	1 516 597 388
China	1 397 028 553	1 424 548 267	1 441 181 819	1 364 456 729	1 020 665 209
Nigeria	181 181 744	206 152 598	264 067 525	410 637 684	793 942 313
USA	319 929 162	331 431 527	354 711 684	389 591 669	447 483 154
Brazil	205 962 108	213 863 046	225 472 214	232 688 044	190 423 052
Russia[2][3]	143 888 004	143 786 842	140 543 418	132 730 511	124 012 605
Turkey	78 271 472	83 835 750	88 416 609	95 626 879	85 776 302
Japan	127 974 958	126 495 647	121 580 505	108 794 446	84 532 388
Britain	65 397 080	67 334 208	70 578 614	75 381 137	80 975 491
France	64 457 201	65 721 165	67 894 271	70 608 998	74 241 659

Germany	81 707 789	82 540 450	82 186 736	79 238 486	71 033 283
Afghanistan	33 736 494	38 054 941	46 699 545	61 928 118	70 409 629
Yemen	26 916 207	30 245 305	36 815 286	48 304 040	53 535 658
Canada	35 949 709	37 603 205	40 417 535	44 948 602	51 621 993
Italy	59 504 212	59 132 073	58 109 918	55 093 194	47 819 324
Saudi Arabia	31 557 144	34 709 640	39 480 349	45 056 349	44 028 699
Australia	23 799 556	25 398 177	28 234 742	33 186 818	41 834 586
Malaysia	30 723 155	32 869 323	36 814 968	41 729 217	41 799 363
The Republic of Korea	50 593 662	51 506 975	52 701 817	50 456 832	38 707 134
Spain	46 397 664	46 459 219	46 115 125	44 394 558	38 378 242
Uzbekistan	30 976 021	33 235 825	36 712 267	40 950 366	38 142 231
Ukraine ^[4]	44 657 704	43 579 234	41 200 374	36 415 702	28 185 563
Tajikistan	8 548 651	9 475 246	11 194 411	14 521 291	18 928 234
UAE	9 154 302	9 813 170	11 054 579	13 163 548	14 776 335
Sweden	9 763 565	10 121 686	10 712 041	11 626 301	13 415 547
Switzerland	8 319 769	8 670 535	9 203 908	9 879 901	10 482 416
Azerbaijan	9 617 484	10 099 743	10 680 202	11 039 198	9 558 619
Czech	10 603 762	10 633 424	10 528 196	10 054 302	8 892 165
Kyrgyzstan	5 865 401	6 301 718	6 997 284	8 112 653	8 851 934
Turkmenistan	5 565 284	6 031 195	6 767 418	7 887 617	8 323 978
Belarus	9 485 772	9 415 431	9 163 188	8 570 898	7 605 140
Georgia	3 999 812	3 977 028	3 868 370	3 483 128	2 438 210
Lithuania	2 878 405	2 794 898	2 655 066	2 375 417	2 012 601
Moldavia	4 068 897	4 020 988	3 838 660	3 243 158	1 855 779
Armenia	3 017 712	3 038 097	2 992 693	2 728 735	1 793 392
Latvia	1 970 503	1 918 949	1 806 289	1 593 463	1 278 130
Estonia	1 312 558	1 295 159	1 243 331	1 129 047	903 682

Achieving sustainable growth in agricultural production, reliable provision of food and agricultural raw materials is inextricably linked with an increase in soil fertility. Currently, the bulk of crop production is produced thanks to mineral fertilizers: they account for 50-55% of the yield increase [2].

The demand for mineral fertilizers is constantly growing. World consumption of fertilizers in 2015/16 amounted to 181 million tons (dry matter), i.e. due to the general economic recession and drought in some

parts of the world (in South and Southeast Asia, Latin America and Africa) decreased by 1%. Nevertheless, the assessment of the market by experts of the international organization IFA in 2016/17 looks quite optimistic: an increase in demand of 2.9% (Table 2). The basis for optimism is some improvement in the economic situation and more favorable weather conditions.

Table 2. Fertilizer consumption in the world, thousand tons

Years	N	P ₂ O ₅	K ₂ O	Total
2012/13	108,1	41,6	29,1	178,8
2013/14	110,4	40,3	30,2	180,9
2014/15	111,8	41,3	31,5	184,6
Growth rate	+1,3%	+2,5%	+4,2%	+2,0%
2015/16	108,0	41,0	32,0	181,0
Growth rate	-1,0%	-1,0%	-0,8%	-1,0%
2016/17	111,0	42,0	33,0	186
Growth rate	+3,0%	+3,0%	+2,3%	+2,9%

In the medium term, until 2025, the mineral fertilizers market will show a moderate increase and, with capacity utilization by 80%, will reach 199 million tons (p.v.) (Table. 3), or 270 million tons in physical volume. For the period 2016-2020 . investments in the industry amounted to \$ 130 billion, more than 150 new capacities were introduced, i.e. the global capacity increased by more than 150 million tons.

Table 3. Medium-term forecast of the development of mineral fertilizers production in the world, thousand tons (p.v.)

Years	N	P ₂ O ₅	K ₂ O	Total
2013/14	110,4	40,3	30,2	180,9
2020/21	117,0	45,0	37,0	199
Growth rate	+1,2%	+1,7%	+2,3%	+1,6%

Ammonia production capacity by 2020 increased by 10% compared to 2010 - up to 230 million tons of NH₃. The main capacities have been introduced in China, Indonesia, USA, Algeria, Egypt and Nigeria. The increase in ammonia production capacity is determined by the expansion of the production base for the production of urea, which accounts for 55% of the nitrogen fertilizer market. The global urea intake capacity for the period 2015-2020 increased by 10% to 229 million tons. Approximately 35% of new projects were implemented in East Asia, 18% in Africa and 15% in North America. In total, 60 new projects for the production of urea were introduced, of which 20 were introduced in China.

The potash fertilizer market, which showed the greatest dynamism in previous years, continued to develop actively in the period 2015-2020: 25 projects were implemented, four of them large greenfield - in Canada,

Russia and Belarus. The global potash fertilizer production capacity in 2020 showed the level of 64.5 million tons (p.v.), i.e. increased by 22% compared to 2015.

The demand for potash fertilizers in 2020 was at the level of 51.6 million tons, i.e. increased by 2.1% per year, and capacity utilization at the level of 80%.

Global sulfur production in 2020 amounted to 72 million tons (p.v.), i.e. increased by 4% annually. Major projects will be implemented in Qatar, Russia, Saudi Arabia and Turkmenistan. In the USA, an increase in sulfur production is also expected, which will lead to a decrease in its imports.

The supply/demand of sulfur in 2020 will amount to 69 million tons (p.v.), i.e. capacities will be loaded by 96%, which is determined by the growth of demand from sulfuric acid producers.

In the phosphate raw materials market, the increase in supply was 11% - up to 250 million tons, while 80% of the increase in the volume of 35 million tons was due to the expansion of the production base in Morocco, Saudi Arabia, Jordan and China.

The global phosphoric acid production capacity for the period 2015-2020 increased by 13% to 65.3 million tons due to the commissioning of 30 new production facilities, with a third of them in China. In addition, new projects have been implemented in Morocco, Saudi Arabia and Brazil. The growth in demand for phosphoric acid by 2020 amounted to 2.5% per year.

In the period 2015-2020, 30 new capacities for the production of phosphorus fertilizers were introduced, as a result of which the global capacity increased by 7 million tons up to 52 million tons.

More than 60% of the consumption of mineral fertilizers in the world is accounted for by nitrogen fertilizers, which have a fairly wide range of products. In fig. 1–5 show the structure of consumption of the most demanded types of nitrogen fertilizers in the most capacious markets of the countries of the world.

The active growth of the world's population determines the aggravation of the problem of food supply. This means that the world market of mineral fertilizers, without which it is impossible to solve this problem, will continue to expand. This paradigm stimulates the growth of capacities for the production of almost all types of fertilizers, which increases competition among their suppliers, including Russian producers of mineral fertilizers.

Chemists who produce mineral fertilizers make a significant contribution to solving the global problem of providing the world's population with food.

With the production of this amount of nutrients, per capita consumption of fertilizers in developing countries will increase from 7 to 23 kg of nutrients, and in industrialized countries from 55 to 145 kg.

The main trends in the development of global production and consumption of mineral fertilizers will consist in increasing the concentration of nutrients in fertilizers as a result of the use of more advanced forms and compositions of fertilizers. Along with the use of fertilizers and various agricultural techniques to obtain high and stable yields of crops, it is necessary to use physiologically active substances (PAS) - growth regulators, herbicides, fungicides, etc.

Today, low yields are largely due to the disproportion in the development of the production of mineral fertilizers and chemical plant protection products. So, if in countries with developed agriculture for 1 ton of mineral fertilizers, 30-50 kg of preparations of chemical plant protection agents (CPPP) are used, then in post-Soviet countries - only 13 kg. As a result, a significant part of the nutrients of mineral fertilizers is absorbed by weeds, 20-30% of the possible harvest is lost due to pests and diseases [3-6].

The transition to an intensive path of development presupposes savings in total costs per unit of production. The creation and implementation of industrial technologies will save not only living, but also total labor [7-11].

There is a huge number of biologically active compounds, the use of which is impeded by insufficient efficiency, toxic effects on warm-blooded animals, the ability to accumulate in the external environment, inaccessibility of raw materials and intermediates for their synthesis, and high cost. Derivatives of pyridine are promising in this respect [12-15]. In particular, IVIN, benzimidazole derivatives, structural analogs of purine and pyrimidine bases [16, 17], among which were found drugs with a wide spectrum of growth-regulating and pesticidal activity (rosaline, Uzgen, olgin, BION, 5-HBION, etc.) and compounds furan, in particular, TPN [18-23], which are increasingly used in various areas of the national economy.

At present, growth substances are used for treating sowing seeds or spraying vegetative plants with aqueous solutions of a certain concentration. Combined application of fertilizers with PAS is both agrochemically and economically viable.

The use of fertilizers with PAS allows to improve the growth, development, productivity of plants, to increase resistance to diseases, to fully use the basic elements of mineral nutrition, and frees them from unnecessary costs for their individual application for crops [24-28]. As PAS, we chose N-oxide-2,6-dimethylpyridine, β - (2-tetrahydrofuryl) propionitrile benzimidazolone, 5-chlorobenzimidazolone, since the results of agrochemical tests indicate their high efficiency [29-35].

Obtaining complex fertilizers with PAS additives is based up to the present time by mechanical mixing of solid components. Thus, it is practically impossible to obtain a homogeneous mixture with a constant ratio between the components throughout the mass, since the content of PAS in the corresponding mixture is relatively small. The absorption of PAS by plants, in excess of a certain dose, can have a negative and sometimes destructive effect. Uniform distribution can only be achieved in the liquid phase. Consequently, for the physicochemical substantiation and development of the technology for obtaining fertilizers with PAS, first of all, knowledge of the solubility and interaction in systems that include the studied components is required.

Mineral fertilizers provide an increase in crop yields and an expanded reproduction of soil fertility; chemical plant protection products - preventing crop losses from pests and diseases; herbicides and growth regulators contribute to the introduction of industrial technologies for growing crops. The efficiency of production as a whole depends on the rational use of chemicals.

Obtaining the maximum positive effect from complex chemicalization with minimum cost and minimum danger of undesirable effects is an important task of science in agricultural practice [36-39].

In recent years, there has been a tendency for the combined use of fertilizers with microelements, growth substances and pesticides, which is the most rational and promising way of using these drugs [40-44].

There are a number of studies on interaction studies and the development of methods for obtaining mineral fertilizers containing growth substances [45-48].

The Institute of General and Inorganic Chemistry of the Academy of Sciences of the Republic of Uzbekistan conducts complex studies of the combination of liquid and solid mineral fertilizers with microelements and growth substances [49-51].

Research has been carried out on the processes of obtaining combined fertilizers based on urea, ammonium nitrate, ammophos and succinic acid [52]. The technology of urea-formaldehyde fertilizers (UFF) containing succinic acid and monoethanolamine has been developed.

Physicochemical and technological studies of the processes of obtaining fertilizers based on carbamide nitrate and PAS - nicotinic, succinic acids, nicotinamide were carried out by the authors [58].

A method has been developed for producing non-adhering urea containing oxalic acid [59]. Agrochemical tests have established that the introduction of urea nitrate and urea containing oxalic acid into the soil against the background of low doses of phosphorus fertilizers increases the content of mobile phosphorus in the soil and increases yields by 10-12 and 22-26%, respectively [60-66].

In addition to succinic and oxalic acids, effective PAS are also dicarboxylic acids: malonic, glutaric, adipic and others. In order to increase the efficiency of mineral fertilizers, pilot batches of fertilizers (ammophos, urea) containing malonic, glutaric and adipic acids were obtained. Agrochemical tests of fertilizers for cotton have shown their high efficiency [67-70].

A positive effect was obtained from the use of such PAS as thiocarbamide, acetamide, formamide in the composition of mineral fertilizers [71-73].

On the basis of physicochemical studies of the interaction of PAS with the components of mineral fertilizers, a number of compositions of liquid fertilizers with the highest content of nutrients and a low crystallization temperature have been proposed. Solid complex fertilizers have also been trained. Agrochemical tests of the latter have shown high efficiency. Formamide promotes the mobilization of soil phosphates [74-76].

Of great importance is the scientifically-based choice of components and complex application with mineral fertilizers, their optimal combination, providing not only maximum growth and metabolic rate, but also the best manifestation of biological efficiency.

Methods

To study the solubility of phases in water-salt systems, a visual-polythermal analysis method developed by A.G.Bergman was used [77].

The essence of the visual-polythermal method is to determine the crystallization temperature by visual observation of the temperature of the appearance of the first crystals, which are released during slow cooling and vigorous mixing of the solution, and the temperature of the disappearance of the last crystals when heated, after which a composition-crystallization temperature diagram is constructed.

The main advantage of the visual-polythermal method is the simplicity, speed, wide temperature range of the study, the versatility of the method, which allows to study aqueous, anhydrous, salt, oxide, organic metal, etc., the possibility of visual fixation of the phenomenon of stratification in the system and assessing the nature of the crystallizing phase, sufficient accuracy. This method makes it easier to detect unstable and metastable equilibrium, which are excluded in the isothermal method. To study technological processes in industry, it is necessary to know the solubility of system components in wide and concentration ranges.

Along with the listed positive aspects, this method has its drawbacks. The essential of them is the impossibility of studying this phase, the difficulty in working with opaque solutions, the possibility of obtaining inaccurate data in the study of systems prone to hypothermia and in which equilibrium is established for a long time, the subjectivity of obtaining data by a researcher who does not know enough about the methodology of this method.

Despite these disadvantages, the visual-polythermal method has been widely used in applied and scientific research.

The solubility diagram of the system was studied using internal polythermal sections. Based on the data obtained for sections and for double systems, a complete polytherm of the solubility of triple systems was constructed in the form of a right triangle using the Rosebohm method. Solution concentrations were expressed in mass percentages.

In order to clarify the nodal points and the steepness of the crystallization surface, projections of the polytherm were constructed on the lateral sides of the system [78].

When performing the research, recrystallized salts of analytical grade were used. and "chemically pure" and laboratory synthesized BION, 5-HBION, IVIN, T P N [79].

Analyzes were carried out according to known methods for the content of phosphorus, nitrogen according to the Kjeldahl method [80], and water according to the Fischer method [81].

The biuret content in the urea melt with PAS was analyzed according to a well-known method [82].

There are no data of physicochemical studies of C_7H_9NO , $C_7H_{11}NO$, $C_7H_6N_2O$, $C_7H_5N_2OCl$ in the literature. The identification of the latter was carried out using modern methods of physicochemical analysis.

Research is necessary in order to further evaluate the possibility of using these characteristics when creating complex fertilizers based on urea and ammophos.

The IR spectra were recorded on a VR-20 spectrophotometer in the frequency range 400-4000 cm^{-1} . The samples were prepared in Vaseline oil and also in the form of KBr tablets. Mass spectra were recorded on an MX-1303 spectrometer, UV spectra - on a Hitachi-EPS-3T spectrometer (solvent - ethanol) and on an SF-4A spectrometer (solvent - methanol).

Thermogravimetric analysis was performed on a MOM-Budapest derivatograph (Hungary) [83] at a heating rate of 12 K / min at a maximum temperature of 600 ° C. Used thermocouples platinum-platinum-rhodium, isolated from substances. The analysis was carried out in platinum crucibles with a lid. The sensitivity of the galvanometer was DTA - 1/10, DTG - 1/15, weighed portion - 0.15 g, the standard was calcined aluminum oxide.

X-ray images of the studied compounds were taken on a DRON-2.0 diffractometer with a Si - anti-cathode.

In order to quantitatively determine IVIN, BION, 5-HBION in the composition of urea and ammophos, methods for monitoring PAS in the composition of the latter by the spectrophotometric method of additives have been developed [84]. The essence of the method consists in determining the optical densities of the standard solution of FAS (A_{ST}) and the investigated solution of the composition (A_x). Taking into account the dilution of the test solutions and the weighed portion of the test samples of the compositions according to the well-known formula:

$$\tilde{N}_0 = C_u \frac{A_x}{A_{CT}}$$

we find the percentage of PAS in the compositions, where:

C_x - the required concentration of PAS, mg / ml,

A_x - optical density of the investigated solution of the composition,

C_{CT} - concentration of a standard solution of PAS, mg / ml,

A_{CT} - optical density of a standard solution of PAS.

The mathematical analysis of the research results was carried out according to [129]. A method has been developed for the determination of TPN in the composition of TPN-containing carbamide and ammophos, the method is based on the gas chromatographic separation of the mixture components in a column filled with sorbent, followed by their registration with a flame ionization detector (FID) or a thermal conductivity detector (katharometer).

The quantitative calculation of the mass fractions of the components is carried out by the method of internal standardization. Benzonitrile (BN) is used as an internal standard.

The density was determined by the pycnometric method [85]. A thoroughly washed and dried pycnometer was weighed on an analytical scale and filled with a test solution or float using a funnel. Then the pycnometer was placed in a thermostat and kept in it for 15-20 minutes. After this time, the pycnometer was removed from the thermostat and weighed. A 10 ml pycnometer was used. The viscosity of the solutions and the melt were studied by the capillary method using a viscometer with an internal capillary diameter of 0.99 mm and 1.12 mm [86].

The main commodity and physico-mechanical properties of complex fertilizers are determined: hygroscopicity, moisture capacity, granule strength, initial moisture capacity according to known methods. The hygroscopicity of fertilizers was determined by the desiccator method at 25 ° C. The hygroscopic point was calculated according to the equations:

$$Q = \frac{a * 180 * 100}{B S}$$

where: Q is the amount of moisture absorbed by a surface unit per unit of time,

a is the moisture gain,

B is the absorption time, in min,

S is the area of the upper base of the box, cm².

$$h = \frac{Q_2 h_1 - Q_1 h_2}{Q_2 - Q_1}$$

where: h is the hygroscopic point of the product,

h₁ and h₂ are the relative humidity (in %) in each desiccator,

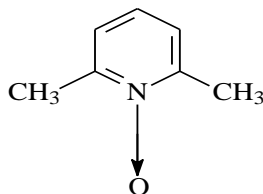
Q₁ and Q₂ are the moisture gain corresponding to the relative humidity of the air in the desiccators in g per 100 cm₂ for 3 hours.

The sorption moisture capacity of fertilizers was determined by us at the average and maximum relative humidity of the air in Central Asia, i.e. at 60 and 80% by daily fixing the moisture gain. Traceability was established using a device for compressing samples [86].

RESULTS AND DISCUSSION. The results of physicochemical studies of the solubility and fusibility of systems, data on the accumulation of biuret in urea, the study of the rheological properties of solutions of extraction phosphoric acid (EPA), urea melt, made it possible to develop and propose a basic technological scheme for the production of ammophos and urea containing PAS, which makes it possible to completely mechanize the process of simultaneous application of fertilizers and PAS in order to reduce the cost of their application. Agrochemical tests have shown their effectiveness [87].

At the Institute of Organic Chemistry of the Academy of Sciences of Ukraine, the preparation -N-oxide of - 2,6-dimethylpyridine (N-oxide of -2,6-lutidine) - IVIN was synthesized [88].

Empirical formula - C₇H₉NO, structural formula:



IVIN is a colorless liquid, transparent, hygroscopic with a specific odor, readily soluble in water, alcohol, acetone, dioxane, benzene and other solvents; boiling point 118-120 ° C (8-10 mm), 129-131 ° C (14-16 mm); molecular weight 123.16 c.u. ; specific gravity $d_4^{20} = 1.1151$ refractive index $n_D^{20} = 1.4975$.

N-oxide-2,6-lutidine has basic properties and forms hydrochloric salt and picrate [89]. The drug and its aqueous solutions are stable for 2-3 years. The control of purity is checked using GLC [90].

The drug is produced at the pilot plant of the Institute of Organic Chemistry of the Academy of Sciences of Ukraine. The synthesis of IVIN is described in the literature [91].

IVIN is on the list of plant growth regulators approved for use in agriculture [92]. IVIN belongs to the group of low-toxic compounds: oral administration LD50 of 1390 mg / kg (for white mice) and 1645 mg / ha (for white rats). It does not have a local irritant and skin resorptive effect [93]. Refers to weakly cumulative compounds, does not possess sensitizing and allergenic properties.

When studying the effect of IVIN, it was found that on specific test objects it exhibits weak auxin activity: it lengthens the segments of hypocotyls, tomatoes, and inhibits the growth of lettuce roots. In experiments on oat seedlings, on the third to fifth day after the onset of germination, the coleoptile stretches out, which becomes longer than the leaf in length, which is typical of substances with auxin activity [94].

Auxins are widely used for rooting cuttings [95]. IVIN has a positive effect on the formation of easily and medium-rooted plants, but in terms of its effect on cuttings of hard-to-root plant species, it is significantly inferior to auxins. In biotests, IVIN does not exhibit gibberellin activity. At the same time, in whole plants, it accelerates the onset of flowering, stimulates the processes of fruit formation, i.e. acts like gibberellin. On specific tests, IVIN exhibits cytokinin activity at the adenine level [96].

On isolated pea chloroplasts, IVIN accelerates the incorporation of inorganic phosphate into photophosphorylation processes, reduces the inhibitory effect of acid phosphatase on the synthesis of adenosine triphosphate acid (ATP), and stimulates light-dependent proton transport in chloroplasts [97].

During germination of spring wheat seeds, IVIN has a positive effect on protein synthesis. In grain (milk ripe), the amount of protein increased by 3.1–8.2%, and the content of albumin, globulins, and gliadins increased [98].

When pre-sowing treatment of seeds of cucumbers and tomatoes with IVIN solution, positive changes in the biochemical parameters of the fruits are noted. In cucumbers, the dry matter content increases by 0.4-0.7%, vitamin C - by 2-10 mg, the amount of sugars - by 0.9% [99].

When cucumber plants were sprayed with IVIN solution, the stem grew faster, the formation of the assimilation surface accelerated [80], and flowering began three to four days earlier. IVIN influenced the processes of sexualization: with an overall increase in the number of flowers by 29%, the number of female

flowers increased by 71, and male flowers - by 23%. The use of the drug has a positive effect in different types of greenhouses both in autumn-winter and in winter-spring crop rotation.

During the pre-sowing treatment of tomato seeds, the effect of IVIN on plants grown in open ground (seedling and non-seedling methods) and in greenhouses is similar to its effect on cucumbers. It helps to increase the energy of germination and germination of seeds, improves growth processes in the initial phases of plant development. At the same time, a higher quality of seedlings is achieved, a more powerful assimilation apparatus is formed.

Thus, IVIN exhibits weak auxin and cytokinin activity. IVIN has an effect on whole plants that is characteristic of auxins, gibberellins and cytokinins. It activates growth processes in the initial phases of development, somewhat accelerates the onset of flowering and fruiting, and increases the early and total crop yield. Consequently, the use of IVIN is economically and agrochemically feasible.

The solubility polytherm of the $C_7H_9NO-NH_4H_2PO_4-H_2O$ system was studied using nine internal sections: the first six of them are directed from the $C_7H_9NO-H_2O$ side to the $NH_4H_2PO_4$ vertex, and the remaining three - from the $NH_4H_2PO_4-H_2O$ side to the C_7H_9NO vertex.

The double system $NH_4H_2PO_4-H_2O$ is well studied and our results are consistent with the literature data. The cryohydrate mixture corresponds to the composition of 17.9% $NH_4H_2PO_4$ 82.1% H_2O at a temperature of $-4.3^\circ C$. The $C_7H_9NO-H_2O$ system was studied by us for the first time from -3.4 to $30.0^\circ C$. The eutectic corresponds to $-3.4^\circ C$ and the composition is 19.3% C_7H_9NO and 80.7% H_2O (Fig. 1, table 4).

Figure 1. Polytherm of solubility of the systems N-oxide-2,6-dimethylpyridine - water

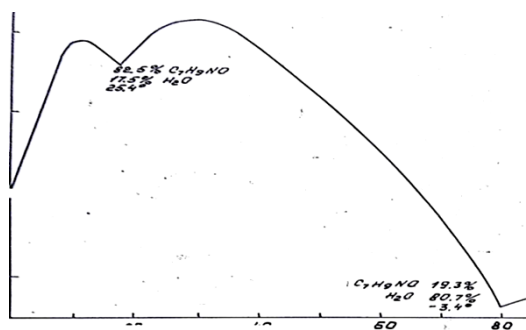


Table 4. Solubility data in the system N-oxide-2,6-dimethylpyridine – water

The composition of the solution, wt%.		Crystallization temperature, °C	Solid phases
C_7H_9NO	H_2O		
100	0	11,0	C_7H_9NO
90	10	28,7	The same
82,5	17,5	25,4	$C_7H_9NO + C_7H_9NO * H_2O$
80	20	27,6	$C_7H_9NO * H_2O$
70	30	31,5	The same
60	40	29,1	The same

50	50	23,5	The same
40	60	16,8	The same
30	70	8,9	The same
19,3	80,7	-3,4	Ice + $C_7H_9NO \cdot H_2O$
10	90	-1,2	The same
0	100	0	Ice
76,6	23,4	30	$C_7H_9NO \cdot H_2O$
61,8	38,2	30	The same

A polythermal solubility diagram of the system from -5.4 to 70.0 °C was constructed based on the totality of the polytherms of internal sections and binary systems, on which the zeros of ice crystallization, C_7H_9NO , $NH_4H_2PO_4$ and the new compound $C_7H_9NO \cdot H_2O$ were isolated, two triple points were established in the system.

The eutectic is equal to 9.2%, C_7H_9NO , 12.3% $NH_4H_2PO_4$, 78.5% H_2O at a temperature of -5.4 °C. The characteristics of the nodal points are given in Table 5. On the basis of polyterminal sections, isotherms are constructed every 10 °C using interpolation.

It can be seen from the polytherm that the main suit of the diagram is occupied by the crystallization field of C_7H_9NO , which indicates its poor solubility in aqueous solutions of $NH_4H_2PO_4$. C_7H_9NO and $NH_4H_2PO_4$ mutually saline each other; at the same time, the solubility of $NH_4H_2PO_4$ decreases by 34.7% and C_7H_9NO - 70.1%.

Figure 2. Polytherm of solubility of the N-oxide-2,6-dimethylpyridine - ammonium dihydroorthophosphate – water

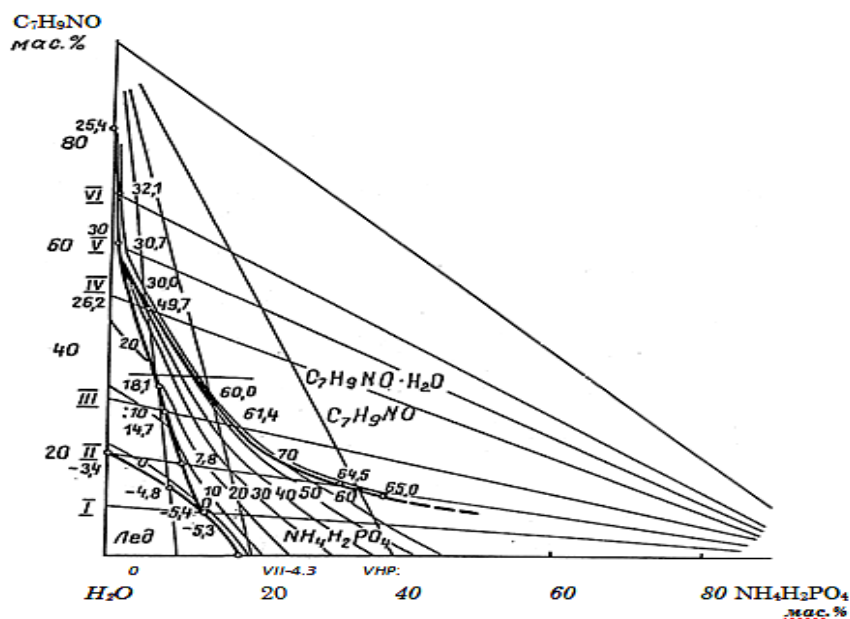


Table 5. Double and triple points of the system N oxide-2,6-dimethylpyridine-ammonium dihydroorthophosphate - water

The composition of the solution, wt%.			Crystallization temperature, ° C	Solid phases
<i>C₇H₉NO</i>	<i>NH₄H₂PO₄</i>	<i>H₂O</i>		
82,2	0	17,8	25,4	<i>C₇H₉NO*H₂O</i> + <i>C₇H₉NO</i>
69,8	0,7	29,5	32,1	The same
59,4	1,2	39,4	30,7	The same
57,0	1,9	41,1	30,0	<i>C₇H₉NO*H₂O</i> + <i>C₇H₉NO</i> + <i>NH₄H₂PO₄</i>
47,5	3,7	48,8	26,2	<i>C₇H₉NO*H₂O</i> + <i>NH₄H₂PO₄</i>
32,7	6,8	60,5	18,1	The same
17,1	8,0	74,0	14,7	The same
18,1	10,0	71,0	7,8	The same
19,5	0	80,5	-3,4	Ice + <i>C₇H₉NO*H₂O</i>
13,2	9,0	77,8	-4,8	The same
9,2	12,3	78,5	-5,4	Ice + <i>C₇H₉NO*H₂O</i> + <i>NH₄H₂PO₄</i>
8,6	12,8	78,6	-5,3	Ice + <i>NH₄H₂PO₄</i>
0	17,9	82,1	-4,3	The same
47,0	6,0	46,1	40,7	<i>C₇H₉NO</i> + <i>NH₄H₂PO₄</i>
30,0	14,2	55,8	60,0	The same
25,0	17,1	57,9	61,4	The same
14,0	30,8	55,2	64,5	The same
12,1	35,4	52,5	65,0	The same

The solubility in the *C₇H₉NO*– (*NH₄*)₂ *HPO₄*–*H₂O* system was studied using eleven internal sections, of which 1-VII were drawn from the side of *C₇H₉NO*–*H₂O* to the top of (*NH₄*)₂ *HPO₄*, and Y3-X1- from the side (*NH₄*)₂ *HPO₄*–*H₂O* to the top *C₇H₉NO* (Fig. 2).

The binary system (*NH₄*)₂ *HPO₄*–*H₂O* was studied earlier. We have experimentally reproduced the eutectic point of the system; it corresponds to a temperature of -6.5 ° C and a solution composition of 29.5% (*NH₄*)₂ *HPO₄*, 70.5% *H₂O*.

On the basis of cuts and binary systems, a solubility polytherm was constructed in the temperature range from -5.8 to 70.0 ° C, the crystallization fields of the initial components and the new compound *C₇H₉NO*H₂O* were delimited. The characteristics of double and triple points are given in table 6.

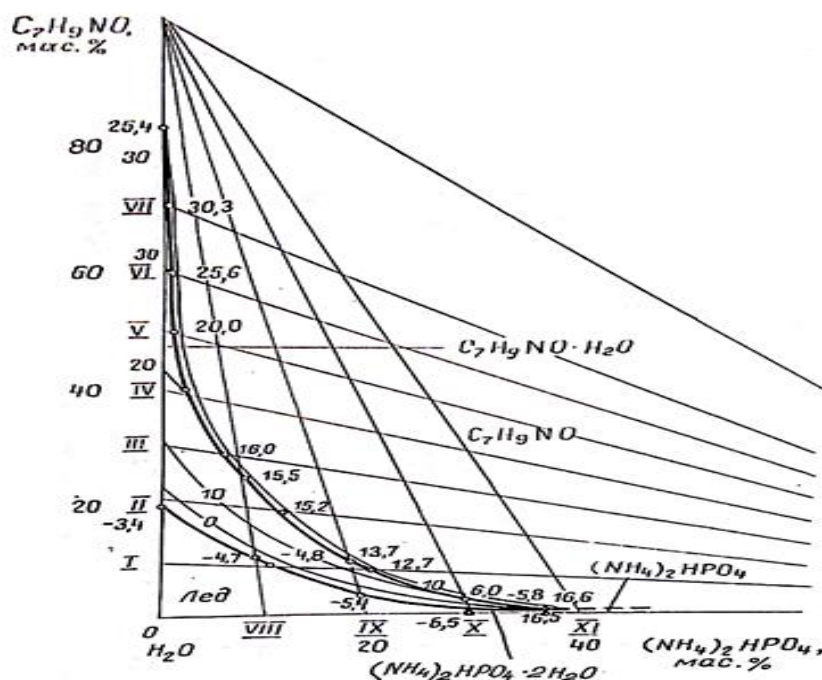
The eutectic point of the system corresponds to a temperature of -5.8 ° C and a solution composition of 1.2% *C₇H₉NO*, 34.0% (*NH₄*)₂ *HPO₄* and 64.8% *H₂O*. At the second triple point, the system at a temperature of 16.6 ° C and in the composition of 0.8% *C₇H₉NO*, 38.8% (*NH₄*)₂ *HPO₄*, 60.4% *H₂O* crystallizes the following

phases: ice, C₇H₉NO, (NH₄)₂ HPO₄, N-oxide-2-6-dimethylpyridine and ammonium hydrogen phosphate mutually salting out each other. The solubility of (NH₄)₂ HPO₄ decreases by 37.6%, and C₇H₉NO - by 81.4%.

Table 6. Double and triple points of the N-oxide-2,6-dimethylpyridine - ammonium hydrogen phosphate - water

The composition of the solution, wt%.			Crystallization temperature, °C	Solid phases
C ₇ H ₉ NO	(NH ₄) ₂ HPO ₄	H ₂ O		
82,2	0	17,8	25,4	C ₇ H ₉ NO*H ₂ O+ C ₇ H ₉ NO
69,7	1,2	29,1	30,3	The same
59,2	1,9	38,9	29,9	The same
49,6	2,0	48,4	25,6	The same
39,1	2,7	53,2	20,0	The same
28,1	6,0	65,9	16,0	The same
23,9	7,8	68,3	15,5	The same
18,3	11,8	69,9	15,2	The same
9,8	18,0	76,1	13,7	The same
7,8	20,0	72,2	12,7	The same
1,9	30,0	68,1	6,0	The same
1,2	34,0	64,8	-5,8	Ice+ C ₇ H ₉ NO C ₇ H ₉ NO*H ₂ O
10,0	9,0	81,0	-4,7	The same
9,2	9,9	80,0	-4,8	The same
3,2	19,6	77,2	-5,4	The same
0,8	38,8	60,4	16,6	Ice+(NH ₄) ₂ HPO ₄ + C ₇ H ₉ NO
0	38,2	61,8	16,5	Ice+ (NH ₄) ₂ HPO ₄
0	29,5	70,5	-6,5	Ice+ (NH ₄) ₂ HPO ₄ *H ₂ O

Figure 3. Solubility polyterm of the system N-oxide-2,6-dimethylpyridine - ammonium hydrogen phosphate - water



The binary system 90% $\text{NH}_4\text{H}_2\text{PO}_4$ + 10% $(\text{NH}_4)_2\text{HPO}_4$ - H_2O has been studied in the temperature range -3.7 to 70 ° C. The eutectic corresponds to a temperature of -3.7 ° C and a solution composition of 19.1% $\text{NH}_4\text{H}_2\text{PO}_4$ 80.9% H_2O

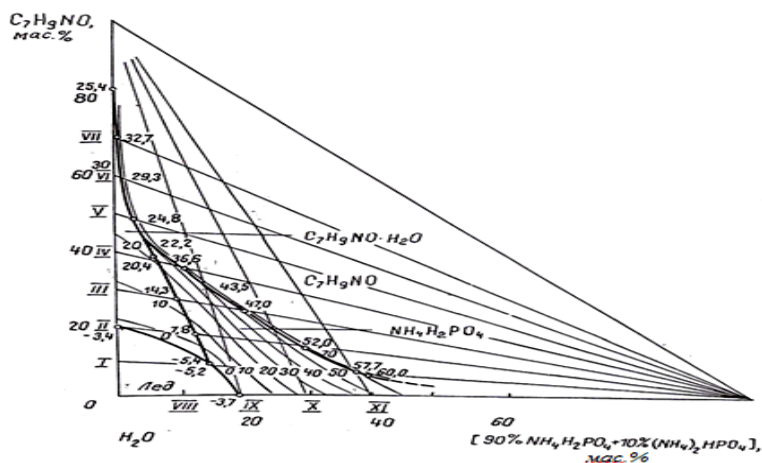
The $\text{C}_7\text{H}_9\text{NO}$ system 90% $\text{NH}_4\text{H}_2\text{PO}_4$ + 10% $(\text{NH}_4)_2\text{HPO}_4$ - H_2O was studied using eleven internal sections, I-VII were drawn from the lateral side of $\text{C}_7\text{H}_9\text{NO}$ to the apex 90% $\text{NH}_4\text{H}_2\text{PO}_4$ + 10% $(\text{NH}_4)_2\text{HPO}_4$, and VIII-XI - from the side 90% $\text{NH}_4\text{H}_2\text{PO}_4$ + 10% $(\text{NH}_4)_2\text{HPO}_4$ to the top $\text{C}_7\text{H}_9\text{NO}$ (Fig. 3.).

The polytherm of the system is characterized by the presence of ice crystallization fields, $\text{C}_7\text{H}_9\text{NO}$, $\text{NH}_4\text{H}_2\text{PO}_4$, and a new hydrate compound N-oxide-2,6-dimethylpyridine. The fields converge at two triple points. The triple point of co-crystallization $\text{C}_7\text{H}_9\text{NO}$, $\text{C}_7\text{H}_9\text{NO} \cdot \text{H}_2\text{O}$, 90% $\text{NH}_4\text{H}_2\text{PO}_4$ + 10% $(\text{NH}_4)_2\text{HPO}_4$ corresponds to the composition of the solution 41.6% $\text{C}_7\text{H}_9\text{NO}$, 4.8% (90% $\text{NH}_4\text{H}_2\text{PO}_4$ + 10% $(\text{NH}_4)_2\text{HPO}_4$), 46.4% H_2O at a temperature of 22.2 ° C. N-oxide-2,6-dimethylpyridine and (90% $\text{NH}_4\text{H}_2\text{PO}_4$ + 10% $(\text{NH}_4)_2\text{HPO}_4$) mutually saline each other out of solution.

The eutectic point of the system corresponds to a temperature of -5.4 ° C and a composition of 14.0% $\text{C}_7\text{H}_9\text{NO}$, 13.9% (90% $\text{NH}_4\text{H}_2\text{PO}_4$ + 10% $(\text{NH}_4)_2\text{HPO}_4$), 72.1% H_2O (Table 6). The solubility (90% $\text{NH}_4\text{H}_2\text{PO}_4$ + 10% $(\text{NH}_4)_2\text{HPO}_4$) decreases by 17.4% when $\text{C}_7\text{H}_9\text{NO}$ is added to the solution at 50 ° C.

It follows from the analysis of the systems that the components have a mutual salting-out effect on each other; salting out of $\text{C}_7\text{H}_9\text{NO}$ from solutions of the corresponding systems increases in the series $\text{NH}_4\text{H}_2\text{PO}_4 \rightarrow 90\% \text{NH}_4\text{H}_2\text{PO}_4 + 10\% (\text{NH}_4)_2\text{HPO}_4 \rightarrow (\text{NH}_4)_2\text{HPO}_4$, and salting out of ammonium phosphates under the influence of $\text{C}_7\text{H}_9\text{NO}$ increases in the series 90% $\text{NH}_4\text{H}_2\text{PO}_4$ - + 10% $(\text{NH}_4)_2\text{HPO}_4 \rightarrow \text{NH}_4\text{H}_2\text{PO}_4 \rightarrow (\text{NH}_4)_2\text{HPO}_4$.

Figure 4. Polytherm of solubility of the N-oxide-2,6-dimethylpyridine-ammophos-water system



The binary system CO (NH₂)₂-H₂O, which is the lateral side of the studied ternary system, is described in the literature in sufficient detail [133-140], and the results obtained agree with the literature. The cryohydrate mixture corresponds to the composition 32.2% CO (NH₂)₂ 67.8% H₂O at a temperature of -11.5 ° C

The solubility polytherm of the C₇H₉NO -CO (NH₂)₂-H₂O ternary system was studied using eleven internal sections. The first six of them are drawn from the side side of C₇H₉NO-H₂O to the top CO (NH₂)₂, and the rest - from the side of CO (NH₂)₂ -H₂O to the top CO (NH₂)₂ (Fig. 4).

On the polytherm in the studied temperature range, the fields of crystallization of ice, C₇H₉NO -CO (NH₂)₂, and two new compounds C₇H₉NO -H₂O, C₇H₉NO -CO (NH₂)₂ are distinguished. The characteristic of the nodal points is given in Table 8. The main part of the diagram is occupied by the crystallization field C₇H₉NO -CO (NH₂)₂. An insignificant part is occupied by the fields of C₇H₉NO - ice. The eutectic point of the system corresponds to a solution composition of 8.2% C₇H₉NO, 31.3% CO (NH₂)₂, 60.5% H₂O and a temperature of -12.4 ° C. At a solution composition corresponding to 22.5% C₇H₉NO, 22.5% CO (NH₂)₂, 55.0% H₂O and a temperature of -9.10C, ice crystallizes, C₇H₉NO * H₂O + C₇H₉NO * CO (NH₂)₂, and at 8 , 2% C₇H₉NO, 31.3% CO (NH₂)₂, 60.5% H₂O and a temperature of -12.40C-ice, CO (NH₂)₂, C₇H₉NO * CO (NH₂)₂.

Table 7. Double and triple points of the N-oxide-2,6-dimethylpyridine - [90% NH₄ H₂PO₄ + 10% (NH₄)₂HPO₄] -water system

The composition of the solution, wt%.			Crystallization temperature, ° C	Solid phases
C ₇ H ₉ NO	90% NH ₄ H ₂ PO ₄ +10% (NH ₄) ₂ HPO ₄	H ₂ O		
82.2	0	17.8	25.4	C ₇ H ₉ NO+ C ₇ H ₉ NO* H ₂ O
69.5	0.6	29.9	32.7	The same
59.7	1.2	39.1	29.3	The same
48.3	3.4	48.3	24.8	The same
41.6	4.8	46.4	22.2	C ₇ H ₉ NO* H ₂ O + NH ₄ H ₂ PO ₄ + C ₇ H ₉ NO

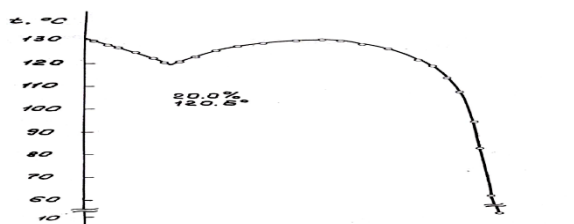
22,1	8,1	69,8	-5,7	The same
24,0	18,0	58,0	-8,4	The same
22,5	22,5	55,0	-9,1	ice+ C ₇ H ₉ NO* H ₂ O+ C ₇ H ₉ NO* CO(NH ₂) ₂
14,4	28,1	57,5	-11,4	ice+ C ₇ H ₉ NO* CO(NH ₂) ₂
8,2	31,3	60,5	-12,4	ice+ CO(NH ₂) ₂ + C ₇ H ₉ NO* CO(NH ₂) ₂
7,0	31,4	61,6	-12,3	ice+ CO(NH ₂) ₂
0	32,3	67,7	-11,5	ice+ CO(NH ₂) ₂
7,6	36,5	55,9	-3,0	CO(NH ₂) ₂ + C ₇ H ₉ NO* CO(NH ₂) ₂
11,7	43,2	45,1	9,3	CO(NH ₂) ₂ + C ₇ H ₉ NO* CO(NH ₂) ₂
14,5	51,1	34,4	32,0	The same
14,9	52,1	33,0	35,6	The same
17,5	66,5	16,0	81,2	The same
19,2	73,6	7,2	99,8	The same
20,0	80,0	0	120,6	The same
81,6	1,7	16,7	22,0	C ₇ H ₉ NO+ C ₇ H ₉ NO* H ₂ O+ C ₇ H ₉ NO* CO(NH ₂) ₂

The sixth cut in the system is the melting diagram of the C₇H₉NO - CO (NH₂)₂ binary system. The diagram shows the branches of crystallization C₇H₉NO, CO (NH₂)₂ * C₇H₉NO and CO (NH₂)₂ (Fig. 6). Two nodal points of the system were determined: 20.0% C₇H₉NO, 80.0% CO (NH₂)₂, at 120.60C and 99.08% C₇H₉NO, 0.02% CO (NH₂)₂ at 11.0 ° C (Table . eight). The C₇H₉NO * CO (NH₂)₂ phase occupies the most significant part in the diagram, the C₇H₉NO branch is weakly expressed.

It follows from the analysis of the diagram that C₇H₉NO has a salting-out effect on CO (NH₂)₂. Analyzing the solubility of the components within the crystallization field C₇H₉NO * H₂O, one can notice that with an increase in the concentration of C₇H₉NO, the solubility of CO (NH₂)₂ increases, i.e. a mutual increase in solubility is observed with an increase in the concentration of CO (NH₂)₂ in the system. For example, at 20 °C the solubility of C₇H₉NO increases by 8.0%, and CO (NH₂)₂ - by 13.7%.

The field C₇H₉NO * CO (NH₂)₂ occupies a significant part on the diagram, which indicates its good solubility in water. The system is of a complex type with the formation of two compounds.

Figure 6. Melting diagram of the system - N-oxide - 2,6-dimethylpyridine - urea



Thus, the studies carried out show that the organization of large-scale production of urea and ammophos containing IVIN, BION, 5-HBION, TPN does not present any particular difficulties. At the same time, it should be emphasized that the latter are quite accessible PASEs.

Conclusions

The fusibility and solubility in three binary and eight ternary aqueous systems, the components of which are physiologically active substances N-oxide-2,6-dimethylpyridine (IVIN) dihydroortho-, ammonium, carbamide hydroorthophosphates, ammonia and orthophosphoric acid, were investigated by the visual polythermal method.

determined that:

That in systems consisting of N-oxide-2,6-dimethylpyridine and orthophosphates of ammonium, carbamide, the formation of two new compounds of the following composition $C_7H_9NO \cdot H_2O$, $C_7H_9NO \cdot CO(NH_2)_2$ was first established.

Isolated from the systems $C_7H_6N_2O$, $C_7H_5N_2OCl$ and new compounds $C_7H_9NO \cdot H_2O$, $C_7H_9NO \cdot CO(NH_2)_2$ were identified by various thermogravimetric, X-ray phase, IR, UV, and Mass spectroscopic methods of physicochemical analysis.

On the basis of the carried out physicochemical studies of solubility, fusibility of systems and technological studies with the study of the rheological properties of solutions of EPA, carbamide fusion, data on the accumulation of biuret in carbamide containing physiologically active substances.

Experimental batches of ammophos and carbamide containing various physiologically active substances with good physicochemical and commercial properties were obtained at a laboratory pilot plant

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