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METHOD FOR THE DETERMINATION OF PHYSIOLOGICALLY ACTIVE SUBSTANCES CONTAINED IN THE AMMOPHOS

Ibrohimjon Abidov

Ph.D., Associate Professor, Namangan Institute of Engineering and Technology E-mail: ib56abidov@gmail.com

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Abstract: The article presents the results of the development of methods for the spectrophotometric and gas chromatographic determination of benzimidazolones introduced into the composition of ammophos. The developed methods make it possible to quantitatively and qualitatively determine the content of benzimidazolone compounds.

Keywords: physiologically active substances, UV spectra, maximum, absorption curves, bathochromic shift, ammonium phosphate, mixing, humidity, composition, temperature, solubility, concentration, chromatographic peak, correction factor, katharometer.

МЕТОД ОПРЕДЕЛЕНИЯ ФИЗИОЛОГИЧЕСКИ АКТИВНЫХ ВЕЩЕСТВ, СОДЕРЖАЩИХСЯ В АММОФОСЕ

Аннотация: В статье представлены результаты разработки методов спектрофотометрического и газохроматографического определения бензимидазолонов, вводимых в состав аммофоса. Разработанные методы позволяют количественно и качественно определять содержание бензимидазолоновых соединений.

Ключевые слова: физиологически активные вещества, УФ-спектры, максимум, кривые поглощения, батохромный сдвиг, фосфат аммония, смешивание, влажность, состав, температура, растворимость, концентрация, хроматографический пик, поправочный коэффициент, катарометр.

INTRODUCTION

Benzimidazolone and its derivatives are of great interest both from the point of view of practical application and in theoretical terms. Individual derivatives of benzimidazolone have fungicidal [1-3], herbicidal and growth properties [4–7].

The study of the herbicidal activity of 6-acylbenzimidazolin-2-ones [8-10] showed that isomers with a normal acyl chain are more active than isomers with a branched part of the acyl. The structural formulas of benzimidazolin-2-one (1) and 5-chlorobenzimidazolin-2-one (2) are as follows:

Molecular weight BION (1) is 134, and CBION (2) - 168.5, odorless substance. After recrystallization from alcohol with activated carbon, light brown brilliant crystals are obtained. Poorly soluble in benzene, chloroform, hot water, soluble in alcohol, acetone, and acids.

METHODS

The essence of the visual-polythermal method. To study the solubility of phases in water-salt systems, a visual-polythermal method of analysis was used, developed by A.G. Bergman [11-13].

Solution concentrations were expressed in mass percent. In order to refine the nodal points and the steepness of the crystallization surface, projections of the polytherm onto the lateral sides of the system were constructed [14]. All projections of the polytherm on the sides are given in Fig. 1.

METHOD OF CHEMICAL ANALYSIS

When performing research, recrystallized salts of the "analytical grade" grade were used. and "c.p." and laboratory-synthesized BION, 5-CBION, IVIN, TPN [15-17].

Analyzes were carried out according to known methods for the content of phosphorus [18], nitrogen according to the Kjeldahl method [19, 20], water according to the Fischer method [22]. The content of biuret in the melt of carbamide with FAS was analyzed according to the known method [21].

Thermogravimetric analysis was carried out on a MOM-Budapest (Hungary) [22] derivatograph at a heating rate of 12 K/min at a maximum temperature of 600°C. The essence of the method lies in the fact that the optical densities of the standard solution of FAS (Ast) and the studied solution of the composition (Ax) are determined. Taking into account the dilutions of the studied solutions and weighing of the studied samples of the compositions according to the well-known formula:

$$C_x = (C_{st} * A_x)/A_{st}$$

We find the percentage of FAS in the compositions, where: Cx - the desired concentration of PAS, mg/ml, A_X - optical density of the investigated solution of the composition, C_{CT} - concentration of standard solution of FAS, mg/ml, A_{st} -optical density of the standard solution of PAS.

Mathematical analysis of the research results was carried out according to [23]. A technique has been developed for determining TPN in the composition of TPN-containing carbamide and ammophos, the method is based on gas chromatographic separation of mixture components in a column filled with a sorbent, followed by their registration with a flame ionization detector (FID) or a thermal conductivity detector (katharometer).

The density was determined by the cyclometric method [24]. The thoroughly washed and dried pycnometer was weighed on an analytical balance and filled with the test solution or melt using a funnel. Then the pycnometer was placed in a thermostat and kept in it for 15-20 minutes. After this time, the pyknometer was removed from the thermostat and weighed. A 10 ml pycnometer was used. The viscosities of solutions and melts were studied by the capillary method using a viscometer with an internal capillary diameter of 0.99 mm and 1.12 mm [25].

The main commercial and physical-mechanical properties of complex fertilizers were determined: hygroscopicity, moisture capacity, granule strength, caking, initial moisture capacity according to known methods [26].

RESULTS AND DISCUSSION

The content of IVIN in the samples was on the same level with similar "dry compositions". When fusion of carbamide and IVIN for 10-15 minutes at a temperature 135°C and re-weighing followed by cooling in the analysis of it was found that IVIN did not undergo changes in the

selected range of parameter variation, and, therefore, retains all the properties of a physiologically active compound (Fig. 1).

The content of IVIN in IVIN-containing carbamide is determined with an accuracy of 0.003%.

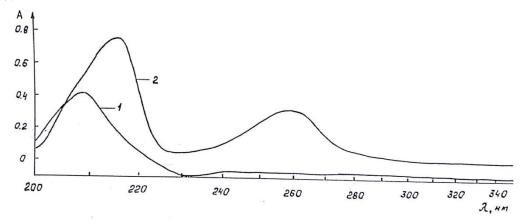


Fig. 1. UV spectra: 1-Ammophos, 2- C7H9NO

To develop methods for monitoring IVIN, BION, 5-CBION in the composition of PAS-containing ammophos, UV spectra of PAS and ammophos were also taken (Fig. 2). The characteristic absorption bands for the quantitative determination of IVIN were λ =260 nm, for BION - λ =280 nm, for 5-CBION - λ =227 nm. The developed methods for the determination of the above PAS are suitable in the case of analyzed compositions containing IVIN, BION, 5 CBION, of various concentrations.

To determine the correction factor, artificial mixtures are prepared, consisting of pure components (WBC and BN) in various ratios to each other. Weighing is carried out with an accuracy of up to 0.0002 g. Mixtures are prepared in flasks with ground stoppers with a capacity of 5 cm3 and diluted with a solvent (acetone, DMF) for better mixing. Store in the refrigerator for no more than 5 days. Chromatograph each artificial mixture at least 5 times.

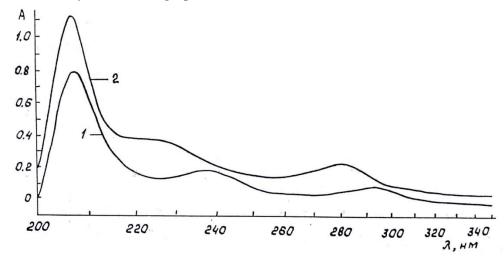


Fig. 2. UV-spectra: 1-Ammophos, 2-C7H6N2O

The correction factor is calculated by the formula (1)

 $K_{TPN} = (R_{TPN} * BN) / (R_{BN} * TPN) (1)$

Where: R_{TPN} , R_{BN} - hitches of TPN and BN (g), TPN, BN - areas of chromatographic peaks, respectively, TPN and BN, in mm2.

Calculate the average value of the correction factor for WBC in each artificial mixture. Determination of the content of TPN in the mixed composition (in ammophos), %. Weigh in a conical flask with a ground stopper with a capacity of 5 cm³ 0.15-1.5 g of the analyzed mixture with an accuracy of 0.0002 g. Add a sample of the internal standard - (0.01 - 0.1 g) of benzonitrile. The mixture is dissolved in the minimum amount of solvent (DMF) and injected into the chromatograph several times.

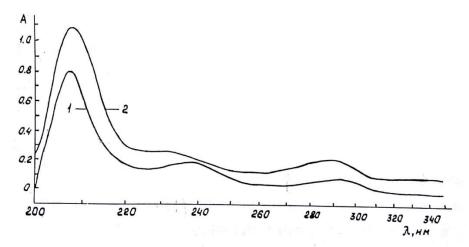


Fig. 3. UV-spectra: 1-Ammophos, 2- C₇H₅N₂OCl

The determination is carried out on at least two samples for each sample. In case of discrepancy in the result, another sample is taken.

The calculation of the content of TPN in the analyzed mixture is carried out according to the formula (2)

 $S_{TPN}\% = (K_{TPN} * S_{TPN} * P_{BN} * 100) / (S_{BN} * m)$

where: K_{TPN} - correction factor for WBC for benzonitrile (BN), S_{TPN} , S_{BN} - areas of chromatographic peaks of TPN and BN (mm²), PBN - sample of benzonitrile (g), m - weight of sample of the analyzed mixture (g).

To develop the technique, we obtained compositions of urea and ammophos with different contents of WBC from 0-13%. TPN was introduced into the hot melt of urea, having a temperature of 135°C. At certain intervals (0, 15, 30, 60 minutes), samples were taken and analyzed for the content of ESRD. To obtain TPN-containing ammophos, TPN was introduced into ammophos pulp neutralized with ammonia, since its decomposition is possible when mixing TPN with phosphoric acid.

CONCLUSIONS

Thus, the studies carried out show that the organization of large-scale production of urea and ammophos containing IVIN, BION, 5-CBION, TPN does not present any particular difficulties. It should be emphasized that the latter are available physiologically active substances. Methods have been developed for the spectrophotometric determination of BION, 5-CBION, IVIN and the gas chromatographic determination of TPN in the composition of the obtained fertilizers.

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