Application of Modern Spectrophotometers for Determining the Amount of Phosphorus (V) Anhydride in Phosphoric Compounds

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ABSTRACT

In this article traditional and modern methods for determining phosphoric anhydride are compared and highlighted the advantages of modern UV spectrophotometer equipment for determining the optical density of substances using ultraviolet radiation.

The quantitative method to measure the concentration of a sample with unknown concentration from the absorption of a sample with known concentration is provided in two methods: The calibration curve method and the standard additive method. The UV-1280 spectrophotometer is designed for measurements in the wavelength range from 190 to 1100 nm and allows you to work in various modes: photometric, spectral, kinetic, quantitative, and biomedical. In particular, to determine the amount of phosphoric anhydride, its photometric model was chosen, and the wavelength was adjusted to 440 nm.

During the experiments, it turned out that almost all types of spectrophotometers can determine the optical density. The amount of P2O5 mg is determined using the graph and related formula. From the results of the following analysis, it can be seen that the value obtained when performing the indicators of both measuring instruments based on the same methodological manual does not differ significantly. This conclusion is also valid for FEK photoelectric colourimeters, which are considered a traditional method.

KEYWORDS: *UV-spectrophotometer, optical density, calibration graph, ammonium molybdate, ammonium metavanadate, citrate solution.*

Introduction

It is known that phosphorus fertilizers are evaluated by the amount of P_2O_5 in them, only the total amount of phosphorus in phosphorite meal and bone meal is determined, and in all other types of fertilizers, the absorbed base P_2O_5 is determined. In particular, although the amount of water-soluble phosphorus is important for the superphosphate, the amount of phosphorus-citrate soluble is suitable for the precipitate. The amount of P_2O_5 soluble in lemon was found for slag, martensitic slag, liquid magnesium phosphates and fluorinated phosphates. The soluble form in citrate refers to phosphorus diluted in an alkaline solution of ammonium citrate. [1]

The above solution is prepared according to Peterman's recipe, it should contain 42 g of ammonia nitrogen concerning 158.2 g of dehydrated or 173 g of crystalline citric acid. This liquid dissolves well in mono- and dicalcium phosphate, but does not dissolve in tricalcium phosphate. Due to the complete solubility of iron and aluminium phosphate in ammonium citrate, this method is not

suitable for fertilizers made from phosphorite with a low percentage and containing a large amount of these oxides. Soluble phosphorus in lemon is contained in 2% citric acid, in which mono dicalcium phosphate and tetra calcium phosphate, as well as silicophosphate, are readily soluble. The standard methods used were the Bechter-Wagner citrate method, Nissen's molybdenum method, Popp's iron-citrate method, and Lawrence's molybdenum method. [2]

Currently, the colourimetric method is widely used, according to which the amount of fertilizer obtained for the experiment is decomposed by boiling in horny vodka or another acid, and then experiments are carried out with a diluted filtrate.

Methods.

The absorbance wavelength to a longer wavelength is called “bathochromic movement”, and its movement to a shorter wavelength is called “hypsochromic movement”. Also, an increase in absorbance is called the “hypochromic effect”.[3]

Analysis to perform quantitative analysis by comparing the colour darkness of a substance is called colourimetric analysis. When the substance is transparent, if absorbance exists in the invisible ultraviolet or near-infrared area, it is measured. The latter is broadly included in colourimetric analysis.

The quantitative method to measure the concentration of a sample with unknown concentration from the absorption of a sample with known concentration is provided in two methods: The calibration curve method and the standard additive method.

In the calibration curve method, standard samples are operated according to an established method and then measured for absorbance. A calibration curve is prepared by using the absorbance obtained here in the vertical axis and the standard sample in the horizontal axis. There are times when the calibration curve does not make a straight line such as when the solution to be measured is a suspension. Although the calibration curve is sure to pass the origin when the solution is used, if it is not used, the curve may not pass the origin. Next, the concentration of the object components in the unknown samples is obtained using this calibration curve.

In the standard additive method, a standard sample is added by stages to four or more samples of measurement sample solution of the same concentration. Similarly to the calibration curve method, a relation curve between added value and absorbance is prepared. The concentration of the object component in the unknown sample is obtained from the point where the related curve crosses the vertical axis. This method is applied only when the related curve is straight as far as the low concentration range.

Generally, extra-large absorbance wavelength is used as a measurement wavelength for quantitative analysis.

Results.

Since the assimilated P_2O_5 for thermophosphates is in a form that is difficult to dissolve, its amount is determined by a 2% citric acid solution, as in drops.

Dissolve 500 g of chemically pure crystalline citric acid in water and make up to 5 litres. In some cases, 2.5 g of salicylic acid is added to it. Take 1 litre of prepared 10% solution, which is also dissolved in 5 litres of water. The final solution is considered to be 2%.

The experimental work is carried out as follows: phosphoric anhydride is dissolved in 2% citric acid, the ratio of the mass of the resulting fertilizer to the volume of acid should be 1: 100. The fertilizer weighing 1-2 g is weighed and transferred to a 250 ml volumetric flask. Pour 100-200 ml of 2%

citric acid into it, close the neck of the test tube with a stopper and mix for 30 minutes in a Wagner apparatus or a magnetic torch. Then a citric acid solution is added to the tube and filtered. The first portion of the filtrate should be discarded. Using a pipette, draw 2 ml aliquots from the clear filtrate into a 100 ml volumetric flask, add 40 ml (at least 25 ml) of the previously prepared ammonium-vanadium solution and top up with distilled water to the line. minutes the optical density is measured.[4]

The UV-1280 spectrophotometer is designed for measurements in the wavelength range from 190 to 1100 nm and allows you to work in various modes: photometric, spectral, kinetic, quantitative, and biomedical. In particular, to determine the amount of phosphoric anhydride, its photometric mode was chosen, and the wavelength was adjusted to 440 nm.

A mixture of metal, sodium sulfite and bisulfite was used as a reducing agent based on previous photocolorimetric methods. In this case, an intense blue-yellow phosphorus-molybdenum complex with the formula (MoO 2, 4MoO 3) $2H_3PO_4$ is formed.[5]

Reverse solution: 2.5 g of anhydrous or 5 g of crystalline sodium sulfide solution are dissolved in 350-400 ml of distilled water in a 500 ml beaker. Dissolve 1 g of metol (methyl para-aminophenol sulfate) in it, and then mix 150 g of sodium pyrosulfite. When the reagents are completely dissolved, the solution is transferred into a 600 ml measuring tube, water is added to the tube, mixed and filtered.[6]

A solution of ammonium molybdenum in sulfuric acid. The acidic salt of 50 g of ammonium molybdenum is dissolved in 10 N. 500 ml H2SO4 in a 600-800 ml beaker and then transferred to a 1 l volumetric flask. Water is poured up to the line and filtered. To prepare a sodium acetate solution, 680 g of sodium acetate are dissolved in 2 litres of water and filtered.

It is known that a standard solution must be prepared to determine the calibration curve. During the analysis, the same graduated graph was built for all types of spectrophotometers. At the same time, 100 ml of 5 volumetric flasks with concentrated solutions of potassium dihydrogen phosphate were prepared according to the following table.

Table 1.

The serial number of the compared solution	Volume of working solution KH_2PO_4, cm^3	Mass of P_2O_5 in a volumetric flask with a volume of 100 cm^3, mg
1	5.0	1.0
2	10.0	2.0
3	15.0	3.0
4	20.0	4.0
5	25.0	5.0

To prepare a standard solution, 7.6696 g of dried potassium dihydrogen phosphate salt was dissolved in a 5 cm^3 solution of nitric acid with a density of 1.4 g/cm^3 and placed in a 1 dm^3 volumetric flask and filled with distilled water at a temperature of 20 ^0C.

1 cm^3 0.2 mg of P_2O_5 stored in the working solution was prepared as follows: 4 mg of P_2O_5 of the stored solution of potassium phosphate acid salt with a volume of 50 cm^3 into a volumetric flask with a volume of 1 dm^3, and then 'water was poured to until the mark of the flask.

In the photocolorimeter, the optical density is filtered through a green light filter against the water in the cuvettes. The amount of P_2O_5 in mg is determined using the graph. The amount of P_2O_5 is

determined by the following formula:

P_2O_5 = (a * 250 * 100) / g * V * 1000

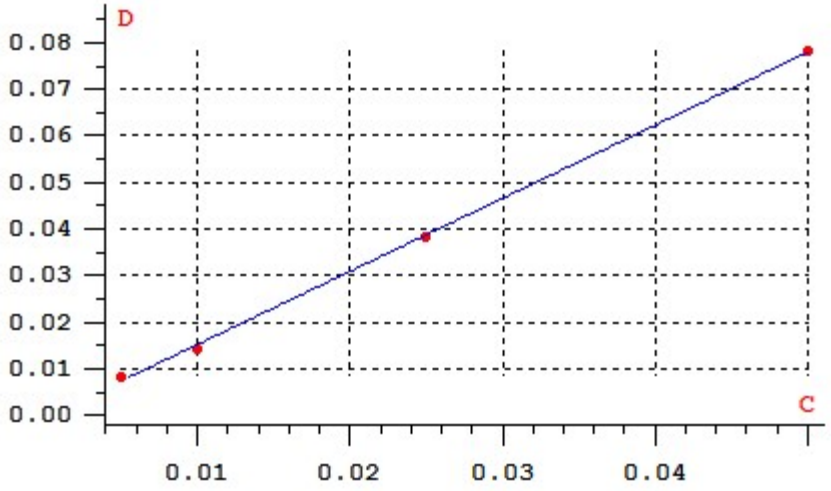
where a- is the amount of P_2O_5 in mg, determined graphically; g- is the mass of the analyzed sample, g; V- is the volume of the solution obtained for analysis, ml.

Discussion.

The determination of total phosphorus by the spectrophotometric method is also performed as a photocolorimetric method. In this case, the main measured value is the optical density, which is used to determine the concentration of the substance by constructing a calibrated graph. [9]

Some modern spectrophotometers also can create graphs, and the number of cuvettes will be a multiple. Optical density is a dimensionless quantity that is usually equal to a number between 0 and 2.

Picture 1.Calibrated graphical sample showing the relationship between the optical density of the analyte and the amount of the substance.



During the experiments, it turned out that almost all types of spectrophotometers have the ability to determine optical density. [9]

Table 2: The results of the analysis were obtained on an SF-56 spectrophotometer.

T/ p	Sample name	Optical density, d	Concentration,%
1	Mineralized mass	0.175	15.0
2	Sludge	0.106	9.1
3	1st layer	0.189	16.2
4	2nd layer	0.231	19.8
5	Dusty fraction	0.205	17.6

The same samples were taken when measuring the model UV-1280 on an ultraviolet spectrophotometer.Pic 2.

Picture 2

Table 3: Analysis of the results obtained with the UV-1280 model ultraviolet spectrophotometer.

T / p	Sample name	Optical density, d	Concentration,%
1	Mineralized mass	0.367	15.7
2	Sludge	0.231	9.9
3	1st layer	0.392	16.8
4	2nd layer	0.476	20.4
5	Dusty fraction	0.425	18.2

Conclusion.

In particular, from the results of the following analysis, it can be seen that the value obtained when performing the indicators of both measuring instruments based on the same methodological manual does not differ significantly. This conclusion is also valid for FEK photoelectric colourimeters, which are considered a traditional method. [11]

The presence of other interfering elements (arsenic, vanadium, silicon) together with phosphorus in the analyzed samples also causes their precipitation with the acidic ammonium molybdate salt, which leads to a high result. [13]

Conversely, other elements (zirconium, niobium, tantalum, titanium) precipitate some of the phosphoric acid during the dissolution of the substance obtained for analysis, thereby reducing the formation of acid salts with phosphorus by molybdenum and causing a drop in the amount. In addition, some elements (Ti and V) slow down the precipitation of ammonium phosphorus molybdate or form soluble compounds such as heteropolyacids. Considering the above reasons, the determination of the number of satellite elements (W, Zr, Ti, Nb, Ta, V, As, Si) in the sample, in which the amount of phosphoric anhydride should be determined, is more complete. Ensures that the colourimetric method can be the solution to quickly and efficiently perform this process.

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