



# Alex Stewart International

## CERTIFICATE OF ANALYSIS

Ref. No. UJ26241

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ORIGINAL

This is to certify that, at the request of Messrs. **ALMAZ FERTILIZERS LLC**, we have performed analysis of the under mentioned commodity:

<b>DESCRIPTION OF GOODS</b>	: <b>DIAMMONIUM HYDROGENORTHOPHOSPHATE</b>
<b>CAS</b>	: <b>7783-28-0</b>
<b>PLACE OF SAMPLING</b>	: <b>ALMAZ FERTILIZERS LLC, RUSSIA</b>
<b>DATE OF SAMPLING</b>	: <b>OCTOBER 16, 2020</b>
<b>DATE OF ANALYSIS</b>	: <b>OCTOBER 26 - 29, 2020</b>

## CHEMICAL COMPOSITION ANALYSIS REPORT

Laboratory analyses were performed for a provided sample according to requirements and following results were obtained:

Parameter	Test method	Actual results
Mass share of base material ((NH <sub>4</sub> ) <sub>2</sub> HPO <sub>4</sub> ), %	Titration	99,93
Mass share of phosphates in terms of P <sub>2</sub> O <sub>5</sub> , %	Photocolorimetric	53,70
Mass share of ammoniacal nitrogen (NH <sub>4</sub> ), %	Titration	21,19
Mass share of potassium in terms of K <sub>2</sub> O, %	Gravimetric	0,0001
Mass share of water insoluble residue, %	Gravimetric	< 0,001
Mass share of moisture, %	Gravimetric	0,051
Mass share of fluorides (F), %	Photometric	< 0,001
Mass share of chlorides (Cl), %	Photometric	0,0048
Mass share of Arsenic (As), mg/kg	ICP-MS/ ICP-AES	1,0
Mass share of Lead (Pb), mg/kg	ICP-MS/ ICP-AES	< 0,1
Mass share of Cadmium (Cd), mg/kg	ICP-MS/ ICP-AES	< 0,05
Mass share of Mercury (Hg), mg/kg	AAS	<0,0025
Mass share of Chromium (Cr), mg/kg	ICP-MS/ ICP-AES	< 1,0
Mass share of Iron (Fe), mg/kg	ICP-MS/ ICP-AES	< 1,0
Mass share of Sulphur (S), mg/kg	ICP-MS/ ICP-AES	50
Exponent of hydrogen ion activity in 0,1% solution in terms of pH	Ionometric	7,67
Turbidity 20 %, NTU (FTU)	Photometric	4,9
Solubility at 20 °C, g/dm <sup>3</sup>	Gravimetric	665
Mass share of Copper (Cu), mg/kg	ICP-MS/ ICP-AES	< 0,25
Mass share of Manganese (Mn), mg/kg	ICP-MS/ ICP-AES	< 1,0
Mass share of Zinc (Zn), mg/kg	ICP-MS/ ICP-AES	< 1,0
Mass share of Molybdenum (Mo), mg/kg	ICP-MS/ ICP-AES	< 1,0

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Mass share of Cobalt (Co), mg/kg	ICP-MS/ ICP-AES	< 1,0
Mass share of Boron (B), mg/kg	ICP-MS/ ICP-AES	< 1,0
Mass share of Calcium (Ca), mg/kg	ICP-MS/ ICP-AES	4,2
Granulation: Mass share of granules below 4 mm, %	Gravimetric	100
Mass share of granules from 4 to 6 mm, %		0

### EQUIPMENT USED:

- Analytical scales Sartorius MSA 225S-100-DI, verified 11.2019, valid till 11.2020
- Atomic Absorption Spectrometer Hitachi Z-5300, verified 03.2020, valid 03.2021
- Mercury analyzer Lumex-Marketing RA-915, verified 06.2020, valid 03.2021
- Ionometer produced by Izmeritelnaya Technika LLC, I-160MI, verified 12.2019 valid till 12.2020
- Spectrophotometer LEKI SS 2107 UV, verified 03.2020 valid till 03.2021
- ICP spectrometer Shimadzu, ICPE 9000, verified 03.2020, valid till 03.2021.

### BRIEF DESCRIPTION OF METHODS USED:

#### Mass Share of base material – titration method

About 2 g of a finely ground preparation is placed in a conical flask and dissolved in 5 cm<sup>3</sup> of pre-boiled water. Then 5 g of sodium chloride is added to the solution. After dissolution the content of the flask is cooled in a cooling mixture (ice with sodium chloride). The cooled solution is titrated at 0 ° C with sodium hydroxide solution to a slightly pink color, using phenolphthalein as an indicator.

#### Mass share of phosphorus – photocolometric method

2 g of the substance is placed in 500 cm<sup>3</sup> volumetric flask and added 200 cm<sup>3</sup> of distilled water. The flask is closed and shaken for 15 minutes. Then it is filtered through a "blue tape" filter. Then 5 cm<sup>3</sup> filtered solution is put in 100 cm<sup>3</sup> volumetric flask. Then distilled water is added up to 20 cm<sup>3</sup> and 25 cm<sup>3</sup> of reagent A. The solution in the flask is stirred and brought to the mark with distilled water. After 15 minutes, the absorbance is measured relative to a reference solution containing 1 mg P<sub>2</sub>O<sub>5</sub>. The P<sub>2</sub>O<sub>5</sub> concentration is found by the calibration graph.

#### Mass Share of ammonical nitrogen – titration method

1 g fertilizers is placed in a glass, added 10 cm<sup>3</sup> of hydrochloric acid solution, boiled for 5 minutes, cooled and quantitatively transferred into a 250 cm<sup>3</sup> volumetric flask, the volume of the solution is lead up to the mark with distilled water and thoroughly mixed. 5 cm<sup>3</sup> of the resulting solution is placed into a conical flask, neutralize with a solution of sodium hydroxide with 1-2 drops of methyl orange until the color becomes yellow, add 10 cm<sup>3</sup> of a buffer solution, mix and add 5 cm<sup>3</sup> of chloramine solution. Closed flask is mixed and left for 15 minutes. Then 3 cm<sup>3</sup> potassium iodide solution and 10 cm<sup>3</sup> of sulfuric acid solution is added, Then, after 5 minutes is titrated the released iodine with sodium thiosulfate solution until the solution is light yellow, then 0.5 ml of starch solution and titrated until the blue color disappears. At the same time, a control experiment is carried out under the same conditions, with the same amount of reagents, but without the analyzed product.

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### **Mass Share of potassium – gravimetric method.**

25 cm<sup>3</sup> of analyzed solution is taken with adding water up to 30 cm<sup>3</sup> and 1-2 drops of methyl red solution and a solution of 10% acetic acid until the color turns red. If the solution immediately turns red when the indicator is added, it is neutralized with a sodium hydroxide solution of 0.1 mol / dm<sup>3</sup> concentration until the color of the solution turns yellow, and then a solution of acetic acid with a volume fraction of 10% is added until the red color is restored. The solution is heated in a water bath up to 50 ± 10 ° C. After heating 10 cm<sup>3</sup> of sodium tetraphenylborate solution is added immediately. After 5 minutes it is quickly cooled in a crystallizer with cold water or in running water to a temperature below 20 ° C and left for 10 minutes. Then solution is filtered. The remains is dried in an oven at 120 ° C for 60 minutes and then cooled for 45 minutes. Then it is weighed and calculated.

### **Mass Share of insoluble residue – gravimetric method.**

20 g of the substance dissolve in 100 cm of hot water. Then It is covered with a watch glass and kept in a water bath for 1 hour. Then the dried to constant weight and weighed solution is filtered through filter crucible. The filter residue is washed with 100 cm<sup>3</sup> of hot water and dried in desiccator at 105-110 ° C til constant weight.

### **Mass Share of moisture – gravimetric method.**

2-5 g of fertilizer is placed in a weighing bottle or cuvette, previously dried to constant weight, and weighed.

A bottle or cuvette with fertilizer is placed in a thermostat and dried with the lid open for 3 hours with temperature of 60-65 C. Then the bottle or cuvette is closed and cooled in a desiccator, keeping for at least 30 minutes before weighing.

### **Mass share of Fluorine – photometric method.**

25 cm<sup>3</sup> of the solution obtained by distillation is placed in 50 cm<sup>3</sup> volumetric flask up to the mark with a buffer solution and mixed. The solution is poured into 50 cm<sup>3</sup> glass placed on a magnetic stirrer, the electrodes are immersed, and after 1-5 minutes the potential value is measured.

### **Mass Share of Chlorine – photometric method.**

25.0 cm<sup>3</sup> neutral test solution is placed in a conical flask, added 1.0 cm<sup>3</sup> of 10% solution of hydrochloric acid and 3.0 cm<sup>3</sup> of starch solution, then it is mixed thoroughly for 1 min. Then it is added 3.0 cm<sup>3</sup> barium chloride solution. After 40 minutes, the optical density of the analyzed solution is measured in relation to the control solution, prepared simultaneously. According to the obtained value of optical density, using the graph, the mass of sulfate ions in the analyzed solution is found in milligrams.

### **Granulation – gravimetric method.**

Weigh 170-250 g of the fertilizer sample. The weighing result is recorded with an accuracy of the first decimal place, and sieving is carried out in accordance with the required size, mechanically or manually, within 2-10 minutes.

When determining the particle size distribution manually, the sieve or set of sieves is taken with both hands and reciprocated approximately 120 times per minute with an amplitude of approximately 70 mm.

If the mineral fertilizer is difficult to sift, especially with a particle size of 1 to 4 mm, the reciprocating motion should be interrupted three times a minute in a circular motion.

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After sieving, the residue is removed from each sieve and weighed. The weighing result is recorded to the nearest decimal place.

It is allowed to combine the remains on sieves within the size of one fraction.

The particles trapped in the sieve mesh are combined with the coarse fraction.

### Exponent of hydrogen ion activity in 0,1% solution in terms of pH – Ionometric method.

100 cm<sup>3</sup> volumetric flask is added 50 ml of distilled water and placed a sample of diammonium phosphate weighing 1.3 g with the weighing result recorded to the third decimal place.

The contents of the flasks are mixed manually using a glass rod and rotating the flasks or shaker for 1-2 minutes. The volume in the flasks is filled up to the mark with distilled water and mixed again.

When using a pH meter that is not provided with a thermal compensation system, the flasks are heated on a hotplate to  $20 \pm 2$  ° C.

In a clean dry glass, it is taken about 50 or 100 cm<sup>3</sup> of the resulting solution. The ionomer electrodes are rinsed with the same solution or suspension, and then the ends of the electrodes are immersed into the beaker by at least 15 mm and the pH value is measured after 1-2 minutes.

### Mass share of elements – ICP-MS method.

Inductively coupled plasma mass spectrometry (ICP-MS) is a type of mass spectrometry that uses an Inductively coupled plasma to ionize the sample. It atomizes the sample and creates atomic and small polyatomic ions, which are then detected. It is known and used for its ability to detect metals and several non-metals in liquid samples at very low concentrations. It can detect different isotopes of the same element, which makes it a versatile tool in Isotopic labeling.

An inductively coupled plasma is a plasma that is energized (ionized) by inductively heating the gas with an electromagnetic coil, and contains a sufficient concentration of ions and electrons to make the gas electrically conductive. Not all of the gas needs to be ionized for the gas to have the characteristics of a plasma; as little as 1% ionization creates a plasma. The plasmas used in spectrochemical analysis are essentially electrically neutral, with each positive charge on an ion balanced by a free electron. In these plasmas the positive ions are almost all singly charged and there are few negative ions, so there are nearly equal amounts of ions and electrons in each unit volume of plasma.

An inductively coupled plasma (ICP) for spectrometry is sustained in a torch that consists of three concentric tubes, usually made of quartz, although the inner tube (injector) can be sapphire if hydrofluoric acid is being used. The end of this torch is placed inside an induction coil supplied with a radio-frequency electric current. A flow of argon gas is introduced between the two outermost tubes of the torch and an electric spark is applied for a short time to introduce free electrons into the gas stream. These electrons interact with the radio-frequency magnetic field of the induction coil and are accelerated first in one direction, then the other, as the field changes at high frequency (usually 27.12 million cycles per second). The accelerated electrons collide with argon atoms, and sometimes a collision causes an argon atom to part with one of its electrons. The released electron is in turn accelerated by the rapidly changing magnetic field.

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The process continues until the rate of release of new electrons in collisions is balanced by the rate of recombination of electrons with argon ions (atoms that have lost an electron). This produces a 'fireball' that consists mostly of argon atoms with a rather small fraction of free electrons and argon ions. The temperature of the plasma is very high, of the order of 10,000 K. The plasma also produces ultraviolet light, so for safety should not be viewed directly. The ICP can be retained in the quartz torch because the flow of gas between the two outermost tubes keeps the plasma away from the walls of the torch. A second flow of argon (around 1 liter per minute) is usually introduced between the central tube and the intermediate tube to keep the plasma away from the end of the central tube. A third flow (again usually around 1 liter per minute) of gas is introduced into the central tube of the torch. This gas flow passes through the centre of the plasma, where it forms a channel that is cooler than the surrounding plasma but still much hotter than a chemical flame. Samples to be analyzed are introduced into this central channel, usually as a mist of liquid formed by passing the liquid sample into a nebulizer.

To maximise plasma temperature (and hence ionisation efficiency) and stability, the sample should be introduced through the central tube with as little liquid (solvent load) as possible, and with consistent droplet sizes. A nebuliser can be used for liquid samples, followed by a spray chamber to remove larger droplets, or a desolvating nebuliser can be used to evaporate most of the solvent before it reaches the torch. Solid samples can also be introduced using laser ablation. The sample enters the central channel of the ICP, evaporates, molecules break apart, and then the constituent atoms ionise. At the temperatures prevailing in the plasma a significant proportion of the atoms of many chemical elements are ionized, each atom losing its most loosely bound electron to form a singly charged ion. The plasma temperature is selected to maximise ionisation efficiency for elements with a high first ionisation energy, while minimising second ionisation (double charging) for elements that have a low second ionisation energy.

### **Determination of elements – ICP-AES method.**

Inductively coupled plasma atomic emission spectroscopy (ICP-AES), also referred to as inductively coupled plasma optical emission spectrometry (ICP-OES), is an analytical technique used for the detection of chemical elements. It is a type of emission spectroscopy that uses the inductively coupled plasma to produce excited atoms and ions that emit electromagnetic radiation at wavelengths characteristic of a particular element.

The ICP-AES is composed of two parts: the ICP and the optical spectrometer. The ICP torch consists of 3 concentric quartz glass tubes.[1] The output or "work" coil of the radio frequency (RF) generator surrounds part of this quartz torch. Argon gas is typically used to create the plasma.

The ICPs have two operation modes, called capacitive (E) mode with low plasma density and inductive (H) mode with high plasma density, and E to H heating mode transition occurs with external inputs.[2] The torch is operated in the H mode.

When the torch is turned on, an intense electromagnetic field is created within the coil by the high power radio frequency signal flowing in the coil. This RF signal is created by the RF generator which is, effectively, a high power radio transmitter driving the "work coil" the same way a typical radio transmitter drives a transmitting antenna. Typical instruments run at either 27 or 40 MHz.[3] The argon gas flowing through the torch is ignited with a Tesla unit that creates a brief discharge arc through the argon flow to initiate the ionization process. Once the plasma is "ignited", the Tesla unit is turned off.

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The argon gas is ionized in the intense electromagnetic field and flows in a particular rotationally symmetrical pattern towards the magnetic field of the RF coil. A stable, high temperature plasma of about 7000 K is then generated as the result of the inelastic collisions created between the neutral argon atoms and the charged particles.[4]

A peristaltic pump delivers an aqueous or organic sample into an analytical nebulizer where it is changed into mist and introduced directly inside the plasma flame. The sample immediately collides with the electrons and charged ions in the plasma and is itself broken down into charged ions. The various molecules break up into their respective atoms which then lose electrons and recombine repeatedly in the plasma, giving off radiation at the characteristic wavelengths of the elements involved.

In some designs, a shear gas, typically nitrogen or dry compressed air is used to 'cut' the plasma at a specific spot. One or two transfer lenses are then used to focus the emitted light on a diffraction grating where it is separated into its component wavelengths in the optical spectrometer. In other designs, the plasma impinges directly upon an optical interface which consists of an orifice from which a constant flow of argon emerges, deflecting the plasma and providing cooling while allowing the emitted light from the plasma to enter the optical chamber. Still other designs use optical fibers to convey some of the light to separate optical chambers.

Within the optical chamber(s), after the light is separated into its different wavelengths (colours), the light intensity is measured with a photomultiplier tube or tubes physically positioned to "view" the specific wavelength(s) for each element line involved, or, in more modern units, the separated colors fall upon an array of semiconductor photodetectors such as charge coupled devices (CCDs). In units using these detector arrays, the intensities of all wavelengths (within the system's range) can be measured simultaneously, allowing the instrument to analyze for every element to which the unit is sensitive all at once. Thus, samples can be analyzed very quickly.

The intensity of each line is then compared to previously measured intensities of known concentrations of the elements, and their concentrations are then computed by interpolation along the calibration lines.

### **Determination of elements – AAS method.**

The absorption spectrum of an element in its gaseous atomic state consists of a series of well defined and extremely narrow lines arising by the electronic transitions of outer most electrons. In case of metals, most of these transitions belongs to visible and UV regions. Most obvious wavelengths at which absorption or emission is observed are associated with the transitions where minimal energy change occurs e.g., 3s-3p transition in Na atom gives rise the emission of yellow light and this is referred as D-line transition. For the analysis, the sample(element) is converted into atomic vapour and then the absorption, either in visible and UV region (at a selected wavelength), which is specific for a given element is measured. For converting element into its gaseous atomic state initial step in the whole procedure of estimation involves spraying a solution of the sample into the flame. This process is accomplished by drawing the solution of the sample as a fine mist into a suitable flame. In terms of Functionality, the flame serves as analogous to cell or cuvette containing the solution in the conventional spectroscopy.

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This analytical method based on atomic absorption is highly sensitive because atomic absorption lines are extremely narrow (0.02-0.5Å) and transition energies are unique for each of the element. However, the limited line width creates a problem in AAS measurement which is usually not the case in the spectroscopy for a solution. In order to follow the Lambert Beer's law, it is necessary that the bandwidth of the source must be narrow to the band width of the absorption peak. Even good quality monochromators with effective band widths has significantly greater bandwidth than the bandwidth of the absorption peaks. So, when the atomic absorption measurements are made with ordinary monochromators equipped with continuous source of radiations then also non-linear calibration curves were obtained. The problem of limited width of the atomic absorption peak is solved by the use of line sources with band width narrower than the absorption peaks. It is therefore, necessary to use the exciting beam that contains high intensity of light of the required wavelengths. This is achieved by the use of hollow cathode lamp. For example, the absorbance peak at 213.9 nm is to serve the basis of determining zinc then the emission peak of the zinc at the same wavelength is isolated and used. In this instance, the line is produced by the zinc vapour lamp exciting the zinc atoms by an electric discharge. The other zinc lines emitted from the source are removed with the help of filters or with a relatively cost-effective monochromator.

**DATED: 30<sup>TH</sup> OCTOBER, 2020**

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