

ATR-FTIR SPECTROSCOPY AS A RAPID COMPLEMENTARY TOOL FOR QUALITATIVE ASSESSMENT OF MINERAL FERTILIZERS

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ABSTRACT

Complementary analytical methods are crucial for the qualitative assessment of mineral fertilizers commonly applied in agriculture. In this study, both reference substances and fertilizer samples were analyzed using the ATR-FTIR technique. Pure compounds such as ammonium sulfate, potassium sulfate, and calcium sulfate heptahydrate were first recorded as standards to establish a reference spectral library. Fertilizer samples were ground to a fine powder (<1 mm) and placed on the diamond crystal surface for spectral measurement. The obtained absorption spectra enabled identification of the main components present in the fertilizers and provided a clear comparison with the standard spectra. Although the method allows only qualitative determination of compounds, comparison with the recorded standards provides valuable insights into the composition of the investigated samples. This approach is particularly useful when rapid screening of fertilizers is required without the need for time-consuming wet chemistry analyses. The results demonstrate that ATR-FTIR spectroscopy can serve as a reliable complementary method for routine fertilizer testing, ensuring both quality control and detection of possible adulteration or impurities. Furthermore, when combined with conventional laboratory techniques such as titrimetry, AAS, or ICP-OES, the method contributes to a more comprehensive and accurate characterization of fertilizer composition, ultimately supporting sustainable agricultural practices.

Key words: fertilizers, ATR-FTIR spectroscopy, spectral standards, qualitative analysis.

INTRODUCTION

The quality of fertilizers used in crop production is critical in ensuring that the right amount of nutrients is available to plants throughout the growth cycle. Excess or low levels of some nutrients can be harmful to plant health; therefore, it is critical to keep track of the nutrients in fertilizers. Fertilizers replenish the nutrients lost by crops in the soil, and agricultural productivity would suffer if fertilizers were not used. Since minerals are readily absorbed and utilized by plants, mineral fertilizers are commonly employed to supplement the soil's nutrition pool. In addition, the fertilizers provide critical elements that improve the soil's ability to retain water while also increasing its fertility (Morari et al. 2011). The macronutrients, phosphorus, nitrogen, and potassium in fertilizer determine its quality. At the same time, certain secondary elements such as magnesium, calcium, sulfur, and trace amounts of boron, copper,

cobalt, iron, molybdenum, manganese, and zinc are present in small amounts but are essential for plant growth. The elemental analysis of fertilizer ensures that the nutrients are present in sufficient quantity. Therefore, it protects the crops by preventing deficiency or excess nutrients in the fertilizer. Furthermore, the analysis also ensures that the quality and content of the fertilizer meet legal criteria (Chauhan et al. 2014). The need for efficient fertilizer comes from multiple directions. Except for nitrogen, all the other nutrient sources are mined and limited in the amounts available. The limitations for phosphate are well known and estimations of the global reserves range typically in the order of a few hundred years (Salazar, 2013; Dawson & Hilton, 2011). The second driver for efficient fertilizer use is that fertilizers can create pollution of soils, surface and ground water by overuse and/or adverse climatological conditions. Runoff to the surface water can cause eutrophication of rivers and coastal zones. Furthermore, high usage combined with high rainfalls can cause leaching to the groundwater. In some parts of the world, like in Europe, restrictions to fertilizer use are enforced. The fertilizer industry is endorsing best practices for the use of fertilizers and has defined the 4R principle: Use the right fertilizer, in the right dose, at the right place, and at the right time (IFA, 2009).

Using adequate fertilization will enhance the security of food in the world. Another important driver for nutrient efficiency is a –potential- economic benefit for both farmers and consumers. Improved nutrient efficiencies lead to lower (operational) costs. Efficient fertilization is thus beneficial for people, planet, and profit, which in a general sense are the key considerations for sustainable growth. Plants take up different nitrogen sources from the soil for their growth. Ammonia and nitrates can be taken up by their root system. In soil, ammonia can be converted to nitrate and if not taken up by the roots, it is immobilized by the soil micro flora or can eventually leach out of the root zone into the deeper soil layers. Therefore, stabilizing agents can be added to ammonia containing fertilizers to prevent or slowdown of this process. The authors do not consider urea to be a slow release fertilizer even though it needs to be hydrolyzed in the soil. Urea is quickly hydrolyzed to ammonia and unless the ammonia is moved in the deeper surface layers in the soil by placement or by water, a fraction can volatilize. Also stabilizing agents for urea have been developed. Stabilizing agents reduce the conversion of the urea to the ammonia. Trenkel (Trenkel, 2010) mentions in his paper the most important nitrification and urease inhibitors. In this paper, we do not discuss the analytical methods for identification and quantification of these types of nitrogen stabilizing agents. For these types of products, specific methods are available or are in development to quantify the amount of inhibitor. Methods for this category are described elsewhere. (European Norm EN16328; European Norm EN 15688, 2008; European Norm EN15905, 2010; European Norm EN 16024, 2011; European Norm EN 16075, 2011) . They are outside the scope of the current study. The first slow release products were the reaction products of urea and formaldehyde which were recognized in 1948 as slow release fertilizers by USDA. The urea-formaldehyde (UF) grades were sold in 1955 (Goertz, 1993). These products are also known as Methylene Urea (MU) products. The composition of the UF/MU fertilizer is basically dependent on the ratio of urea to formaldehyde but also on the condensation conditions, and different amounts of oligomers, i.e., MDU (methylene diurea), DMTU (dimethylene-triurea), TMTU (trimethylene tetraurea) etc. are formed as the ratio changes. As the length of the oligomers increases, the water solubility decreases. The amount and their distribution determine the slow release properties of the product in the field (Goertz, 1993). The slow release properties of UF/MU products are caused by the microbiological degradation and hydrolysis in the soil. For these types of products, methods have been developed to test the amount of product soluble in cold water and hot water. Furthermore, chromatographic methods to determine the different compounds are also available and described in this paper. In addition to these solid urea formaldehyde materials, liquid products are commercially available. The most important class of these liquid products are made by a condensation reaction of urea,

formaldehyde, and ammonia, yielding mainly a six membered ring known as triazone ring (Clapp 2001). There are small amounts of methylene urea compounds present in these products as well. The slow release nitrogen in Triazone type fertilizers is made available to the plants by hydrolysis and microbiological degradation similar to other UF/MU products.

MATERIAL AND METHODS

Sample Collection and Preparation

Samples of mineral fertilizers were obtained and analyzed using several standardized analytical methods. Prior to analysis, fertilizer samples were ground to a fine powder using a mechanical mill. Representative samples were selected for further instrumental and chemical analyses.

Infrared Spectroscopic Analysis (FTIR–ATR)

The infrared (IR) spectra were recorded on an Agilent Cary 630 FTIR spectrometer equipped with an ATR (Attenuated Total Reflectance) diamond crystal head, in the spectral range of 4000–650 cm^{-1} , with a resolution of 4 cm^{-1} . Initially, FTIR–ATR spectra of pure reference substances commonly found in solid mineral fertilizers were recorded. The spectra of fertilizer samples were then recorded under identical conditions and compared with those of reference substances to identify functional groups present in the samples. Multiple spectra were overlaid to evaluate spectral similarity.

Determination of Major Nutrients (N, P, K)

The empirical composition of the mineral fertilizers was determined following ISO standardized and validated analytical methods. Routine analyses included flame photometry, UV–Vis spectrophotometry, and atomic spectrometry. Flame photometry was used for the determination of potassium (K), UV–Vis spectrophotometry for phosphorus (P), and the Kjeldahl method for total nitrogen (N) content. Sample preparation involved grinding, dissolution in water or mineral acids, and subsequent instrumental measurement (ISO 6598).

Phosphorus Fractionation

Phosphorus determination was divided into four fractions: (1) water-soluble P, (2) acid-soluble P, (3) citrate-soluble P, and (4) citric acid-soluble P. Absorbance of filtrates was measured by UV–Vis spectrophotometry and phosphorus concentration was calculated using calibration curves (ISO 6598).

Nitrogen Determination (Kjeldahl Method)

Nitrogen in fertilizers occurs as ammonium ($\text{NH}_4\text{-N}$), nitrate ($\text{NO}_3\text{-N}$), urea (amide-N), and organic N. The Kjeldahl method was used for total nitrogen and its forms. For fertilizers containing nitrates, 2 g of salicylic acid and 5 g of sodium thiosulfate were added during digestion to convert nitrate nitrogen into nitrosalicylic acid, which was subsequently transformed into ammonium nitrogen in the presence of sulfuric acid (H_2SO_4). The analysis was performed using a Kjeldahl digestion and distillation unit (ISO 5315).

Sulfur Determination (Turbidimetric Method)

Total sulfur content, including elemental and combined forms, was determined by oxidation to sulfate using hydrogen peroxide (H_2O_2). The resulting sulfates were quantified turbidimetrically as barium sulfate (BaSO_4). Percent transmittance was measured with a spectrophotometer and compared with standard sulfate solutions using a calibration curve (ISO 18644).

Determination of Secondary and Trace Elements

Elements such as Mg, Fe, Ca, B, and Zn were determined by atomic spectrometry or ICP–OES. Samples were ground to fine powder, dissolved in suitable solvents, and analyzed for emission or absorbance intensity.

Instrumentation Summary

Instrument	Purpose
Agilent Cary 630 FTIR (ATR)	Infrared spectral analysis (functional group identification)
Flame Photometer	Determination of potassium (K)
UV–Vis Spectrophotometer	Determination of phosphorus (P) and sulfur (S)
Kjeldahl Digestion and Distillation Unit	Determination of total nitrogen (N)
ICP–OES / Atomic Spectrometer	Analysis of Mg, Fe, Ca, B, Zn, and other trace elements

Solvents, Reagents, and Samples

Solvents: In this method, solvents were primarily used for cleaning the diamond ATR crystal of the infrared spectrometer. Depending on the solubility characteristics of the analyzed substances, distilled water or ethanol was used as the cleaning solvent.

Reagents: The following analytical-grade substances were used for IR spectroscopic reference and calibration purposes: ammonium sulfate ($(\text{NH}_4)_2\text{SO}_4$), potassium sulfate (K_2SO_4), calcium sulfate (CaSO_4), and magnesium sulfate heptahydrate ($\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$).

Samples: The analyzed mineral fertilizer samples included:

- UREA (pure urea sample)
- Fertilizer 1: NPK(S) 15:15:15 + 2% SO_3
- Fertilizer 2: Ultra Complex NPK(S) 15:15:15 + 25% SO_3 + 0.5% CaO + 0.5% MgO + trace elements (TE)
- Fertilizer 3: NPK(S) 15:15:15 + 8% SO_3 + Zn
- Fertilizer 4: NPK–S 33:0:0:11

All samples were homogenized and finely ground before analysis.

Instrumentation and Experimental Conditions

The analytical instruments employed for the characterization of mineral fertilizer samples were as follows:

- Infrared spectrometer: Agilent Cary 630 FTIR equipped with ATR (Attenuated Total Reflectance) module with a diamond crystal head
- UV–Vis spectrophotometer: Agilent Cary UV–Vis
- Flame photometer: JENWAY model, used for potassium determination.

All measurements were performed under controlled laboratory conditions (temperature 20–25 °C, relative humidity below 60%), using analytical-grade reagents and freshly prepared calibration standards.

Methods

Infrared Absorption Spectroscopy (FTIR–ATR): Infrared absorption spectroscopy measures the amount of radiation absorbed by a compound in the infrared region of the electromagnetic spectrum. The infrared region spans wavelengths between 2.5 and 15 μm , corresponding to frequencies between 4000 and 650 cm^{-1} . The obtained IR spectrum represents the intensity of absorbed radiation as a function of frequency, providing an absorption spectrum characteristic of the molecular structure. When electromagnetic radiation in the infrared range is absorbed by molecules, transitions occur between vibrational energy levels, resulting in molecular excitation from lower to higher vibrational states. The energy changes involved are

typically 2–10 kcal mol⁻¹, producing characteristic molecular vibrations. Each absorption band or peak in the IR spectrum corresponds to a specific vibrational mode of the molecular bonds, and the position of these peaks provides valuable structural information. The intensity of absorption is proportional to the change in the dipole moment during vibration. For simple molecules, the number and position of peaks can be correlated with specific vibrational frequencies, allowing complete spectral interpretation. Currently, Fourier Transform Infrared (FTIR) Spectroscopy is the standard analytical technique. In FTIR, the electromagnetic radiation is split into two beams, one of which travels a longer optical path than the other. When recombined, the beams produce an interferogram, which is mathematically transformed using a Fourier transform to yield a conventional infrared spectrum. The advantages of FTIR over dispersive methods include rapid acquisition of the entire spectrum (within seconds), minimal sample quantity requirements, and high spectral resolution. These features make FTIR–ATR spectroscopy an efficient and reliable method for the qualitative identification of chemical functional groups in mineral fertilizer samples.

RESULTS AND DISCUSSION

Analysis of Standards by Infrared Spectroscopy

Molecular spectroscopy studies the interaction of electromagnetic radiation with matter, without considering possible accompanying chemical effects. When electromagnetic radiation (light) interacts with a sample, absorption, emission, or scattering may occur. Infrared (IR) spectroscopy measures the absorption (or transmission) of different infrared frequencies by a sample placed in the path of an infrared beam, whereas Raman spectroscopy, also a branch of molecular spectroscopy, is based on inelastic light scattering. From the obtained results and spectral assignments of the recorded IR spectra of the reference substances (Figures 1 and 2), characteristic absorption bands were identified at specific wavenumbers (cm⁻¹). The sulfate anion (SO₄²⁻) exhibits distinctive infrared absorption bands due to its internal vibrational modes. These bands are primarily associated with S–O stretching and O–S–O bending vibrations. Specifically, the most significant bands appear in the region of 1200–900 cm⁻¹ for stretching vibrations and 650–400 cm⁻¹ for bending vibrations: Asymmetric S–O stretching gives rise to the most intense peak, typically observed around 1100 cm⁻¹. This band may appear as a doublet or as a broad, multi-peaked band depending on symmetry changes or interactions with neighboring ions or molecules. Symmetric S–O stretching produces a weaker absorption band, usually located at approximately 980 cm⁻¹. Asymmetric O–S–O bending results in a band in the 650–600 cm⁻¹ region, while symmetric O–S–O bending gives a weak band near 450 cm⁻¹ (Figure 1). The last two bending vibrations were not detected in this study, as the operational range of the ATR instrument was limited to 4000–650 cm⁻¹. These observations are clearly evident in the infrared spectrum of potassium sulfate (K₂SO₄), where absorption bands were recorded at 1098 cm⁻¹ (asymmetric S–O stretching) and 982 cm⁻¹ (symmetric S–O stretching). In the infrared spectrum of ammonium sulfate ((NH₄)₂SO₄), the sulfate anion exhibits a characteristic band at 1057 cm⁻¹, while additional absorption bands correspond to the ammonium cation (NH₄⁺). These bands arise from N–H stretching and bending vibrations within the NH₄⁺ group. Specifically, N–H stretching vibrations are observed in the region around 3000–3400 cm⁻¹, whereas N–H bending vibrations appear near 1400 cm⁻¹ (Figure 2).

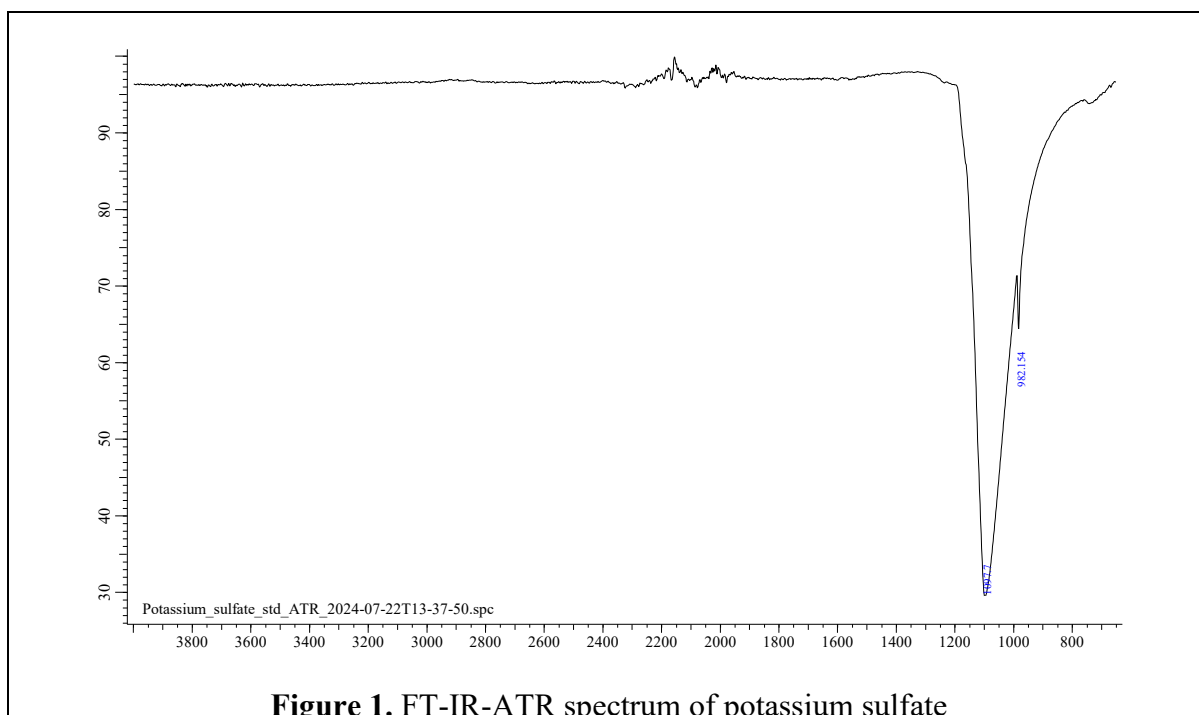


Figure 1. FT-IR-ATR spectrum of potassium sulfate

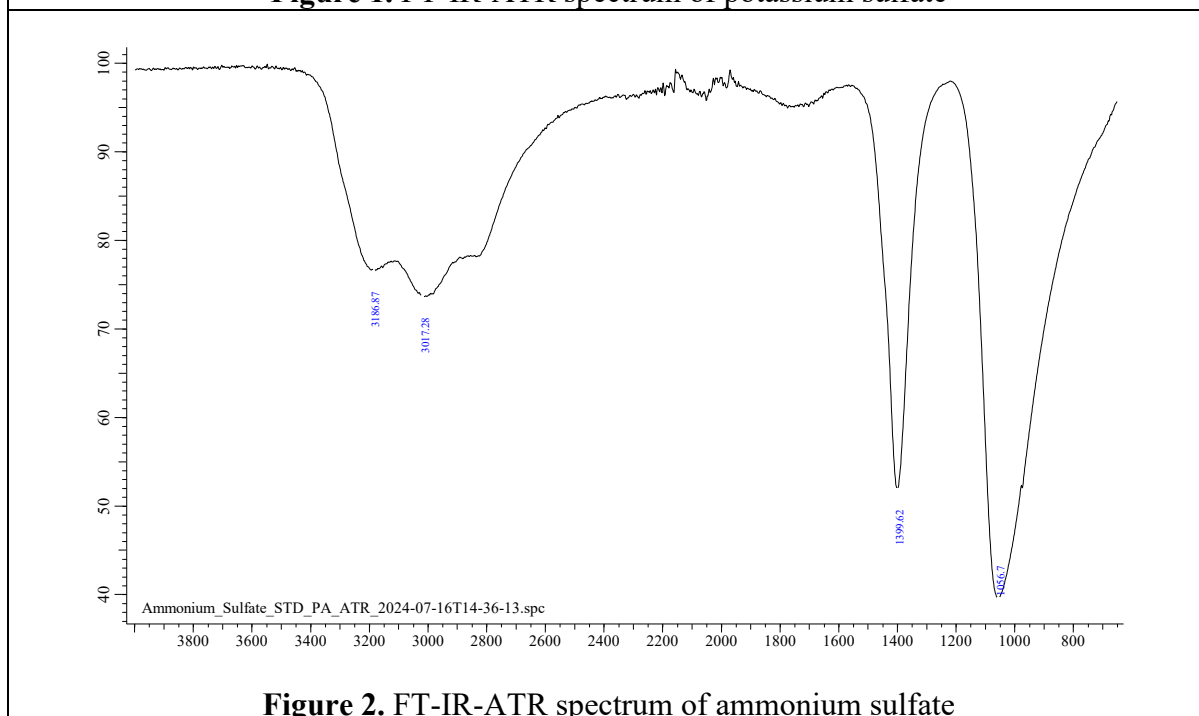


Figure 2. FT-IR-ATR spectrum of ammonium sulfate

The calcium sulfate dihydrate (gypsum) exhibits characteristic infrared (IR) absorption bands associated with both the sulfate anion (SO_4^{2-}) and the crystalline water molecules (H_2O). The key absorption bands include those corresponding to the sulfate stretching vibrations, typically observed in the 1200–1000 cm^{-1} region (at 1098 cm^{-1}), and the deformation and stretching vibrations of the O–H bonds of water molecules, which appear at approximately 1620 cm^{-1} and 3400–3600 cm^{-1} , respectively (Figure 3).

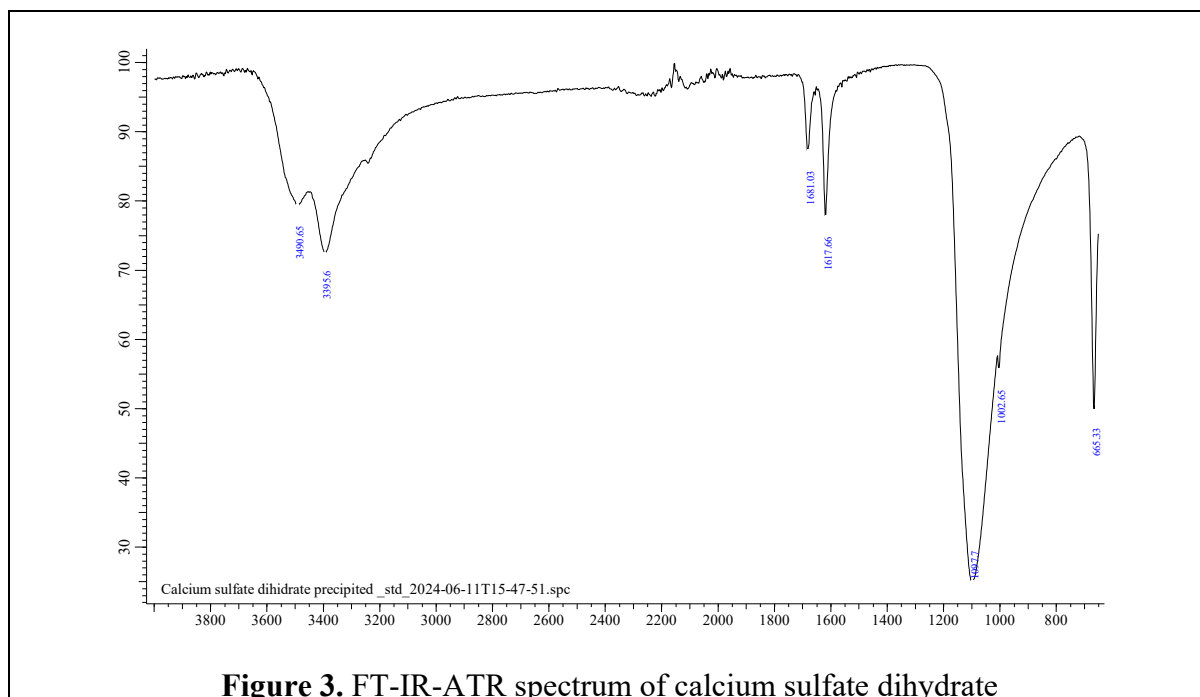


Figure 3. FT-IR-ATR spectrum of calcium sulfate dihydrate

To determine the presence of other commonly used pure components in solid mineral fertilizers, FT-IR-ATR spectra were recorded for the following standard compounds: urea, potassium nitrate, ammonium nitrate, monoammonium phosphate (MAP), and diammonium phosphate (DAP). The corresponding spectra are presented in Figures 4.5a–4.5d. It can be concluded that when sulfate compounds are present in fertilizers at concentrations above approximately 8%, the absorption band around 1100 cm⁻¹ can generally be detected.

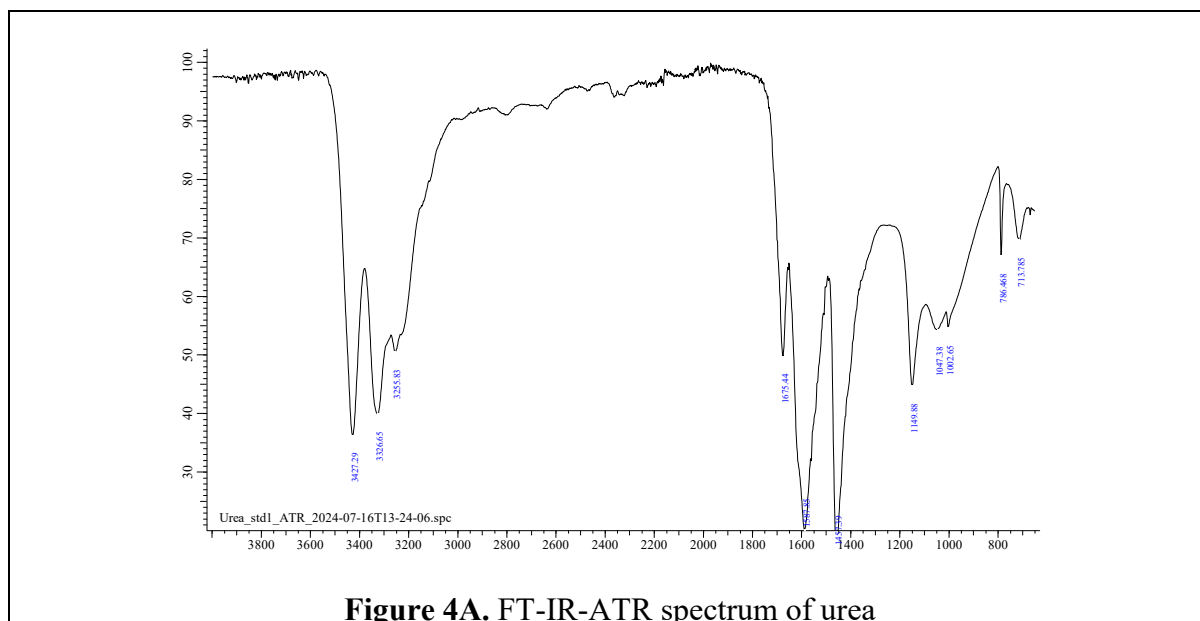


Figure 4A. FT-IR-ATR spectrum of urea

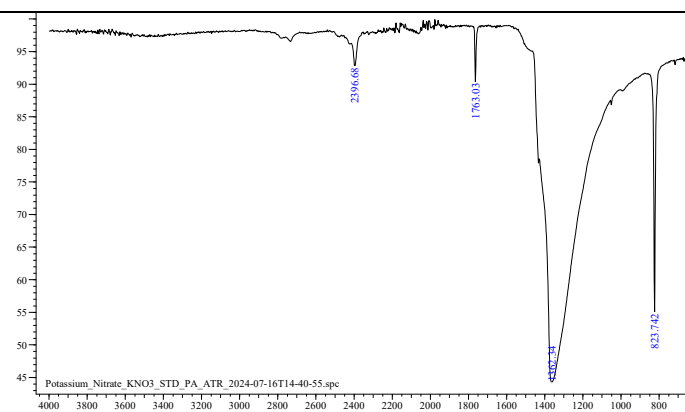


Figure 4B. FT-IR-ATR spectrum of potassium nitrate

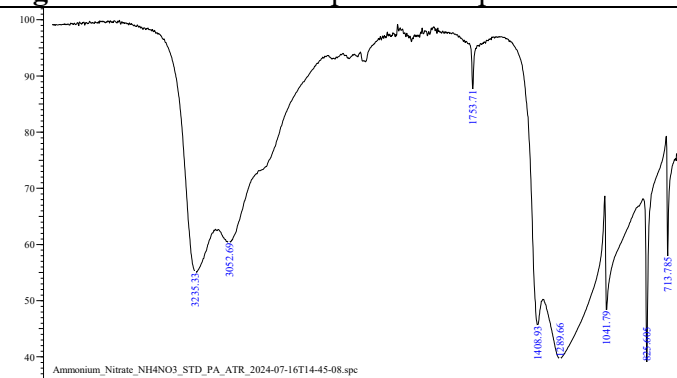


Figure 4C. FT-IR-ATR spectrum of ammonium nitrate

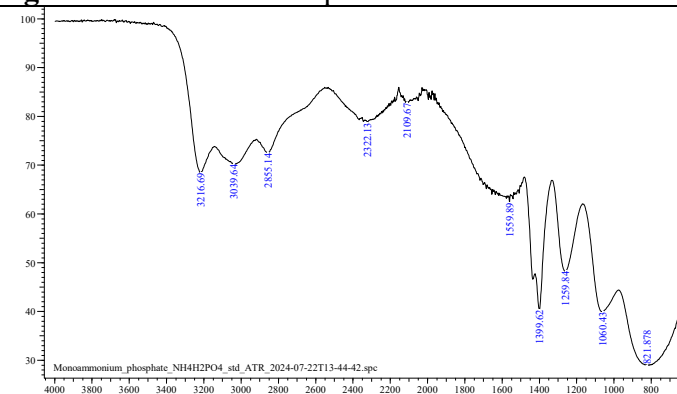


Figure 4D. FT-IR-ATR spectrum of monoammonium phosphate (MAP)

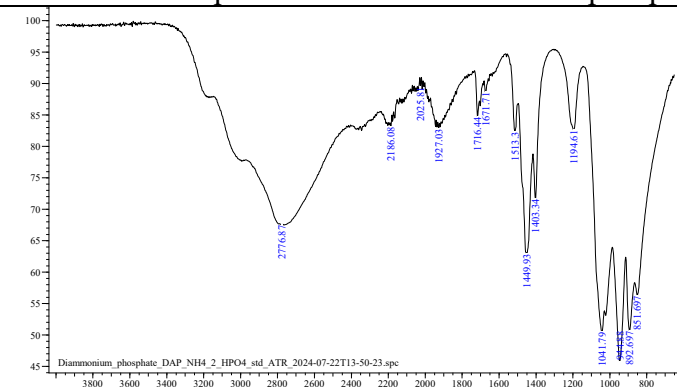


Figure 4E. FT-IR-ATR spectrum of diammonium phosphate (DAP)

Analysis of Solid Fertilizers Containing Sulfur by Infrared Spectroscopy

In the first solid fertilizer sample, based on the FT-IR-ATR spectrum, the presence of sulfur in the form of sulfate could not be confirmed from the characteristic absorption bands. The spectrum most likely indicates the presence of ammonium ions and possibly diammonium phosphate (DAP). This observation is not unexpected, considering that the fertilizer contains only 5% sulfur (Figure 5).

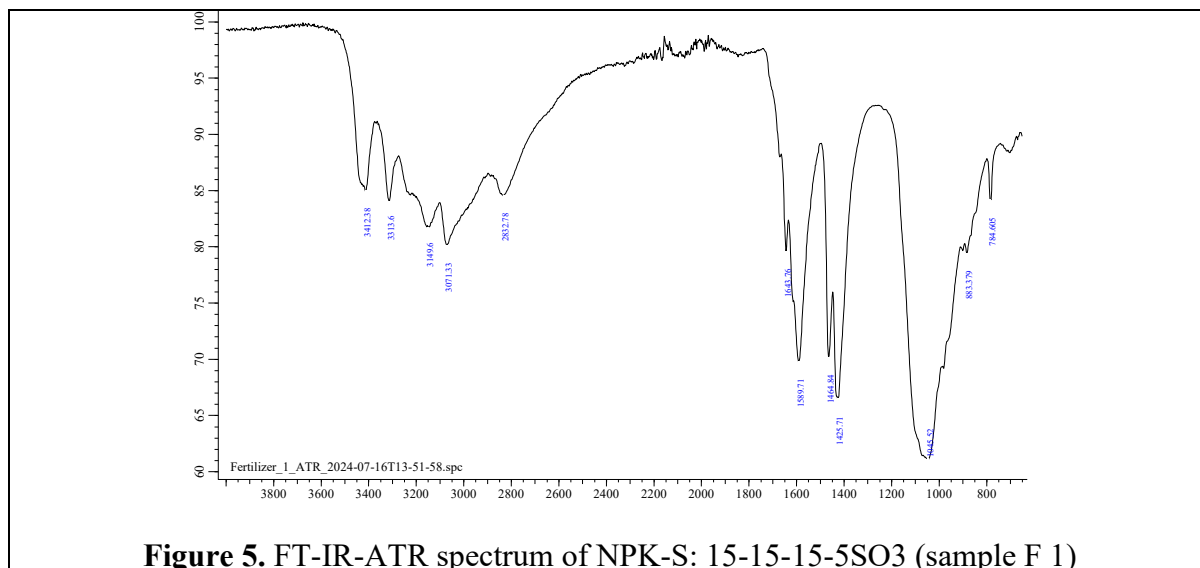


Figure 5. FT-IR-ATR spectrum of NPK-S: 15-15-15-5SO₃ (sample F 1)

In the second sample (F2) of solid mineral fertilizer, the spectrum clearly indicates the presence of sulfate, with characteristic absorption bands observed at 1041 cm⁻¹ and 1050 cm⁻¹, corresponding to ammonium sulfate. In this case, the fertilizer contains 10% sulfur, and the intense absorption bands of the sulfate anion (SO₄²⁻) confirm its presence. This finding is consistent with the fertilizer's declared sulfur content (25%) and is further confirmed by comparison of the FT-IR-ATR spectra of the fertilizer sample and the standard ammonium sulfate reference (Figure 6).

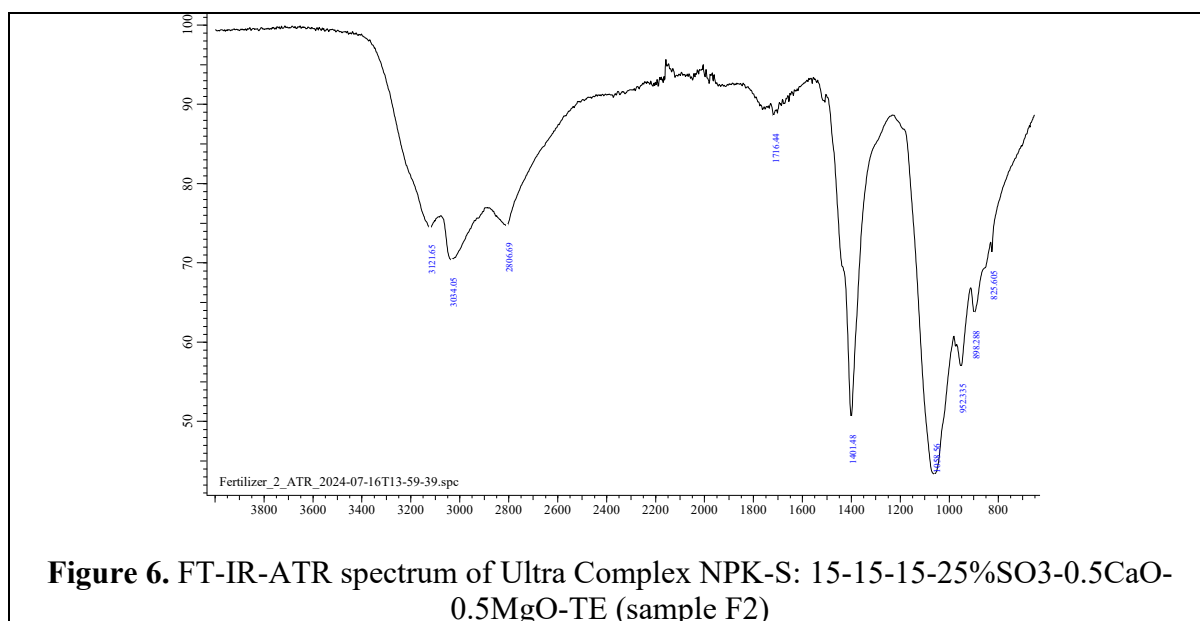


Figure 6. FT-IR-ATR spectrum of Ultra Complex NPK-S: 15-15-15-25%SO₃-0.5CaO-0.5MgO-TE (sample F2)

In the third sample (F3) of solid mineral fertilizer, based on the FT-IR-ATR spectrum, the presence of sulfur in the form of sulfate could not be confirmed from the characteristic absorption bands. The spectrum most likely indicates the presence of N–H bonds, which is not unexpected given that the fertilizer contains only 3.2% sulfur.

In the fourth sample (F4), labeled NPK-S 33-0-0-11, the fertilizer is composed exclusively of nitrogen and sulfur. The spectrum clearly reveals the presence of sulfate, as evidenced by the absorption bands at 1057 cm^{-1} and 1405 cm^{-1} , characteristic of ammonium sulfate. The other component identified in the fertilizer is urea (Figure 7). All these observations were further confirmed by superimposing the spectra of the three analyzed samples (Figure 8). Based on the NPK-S designation and the composite spectrum of sample F4, it can be concluded that the fertilizer represents a 50:50 mixture of ammonium sulfate and urea.

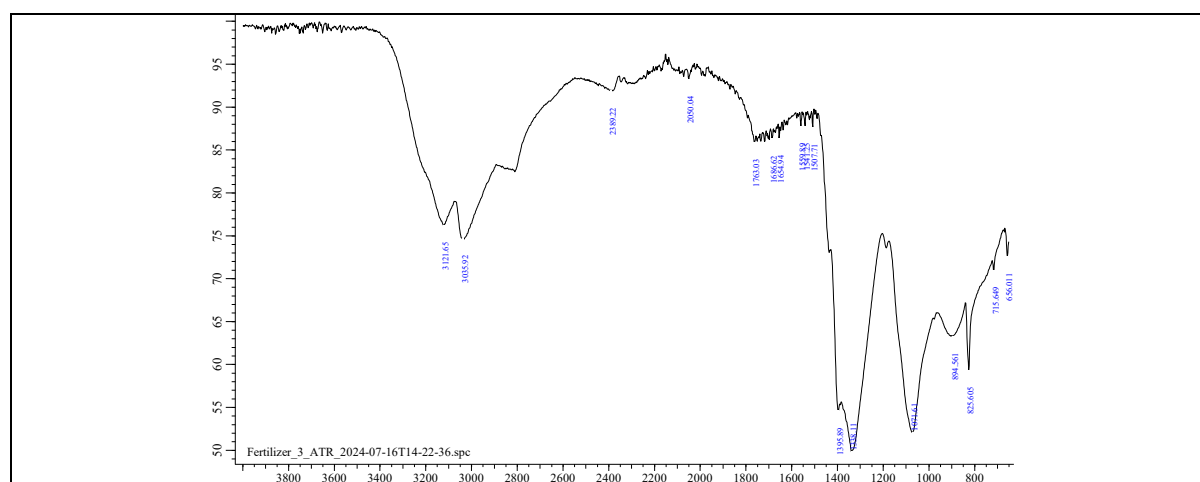


Figure 7. FT-IR-ATR spectrum of Complex NPK-S 15-15-15-8SO₃+Zn (sample F3)

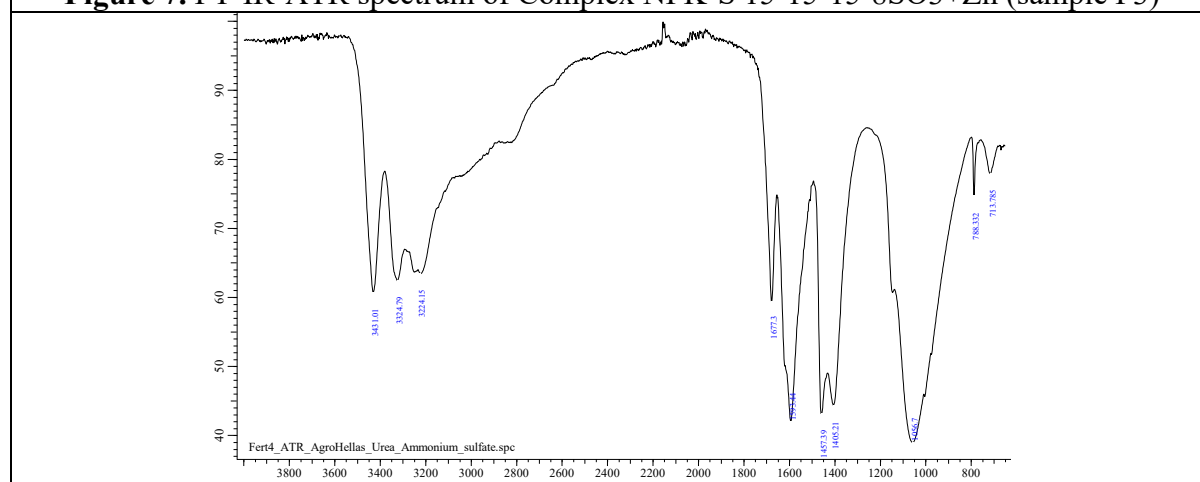
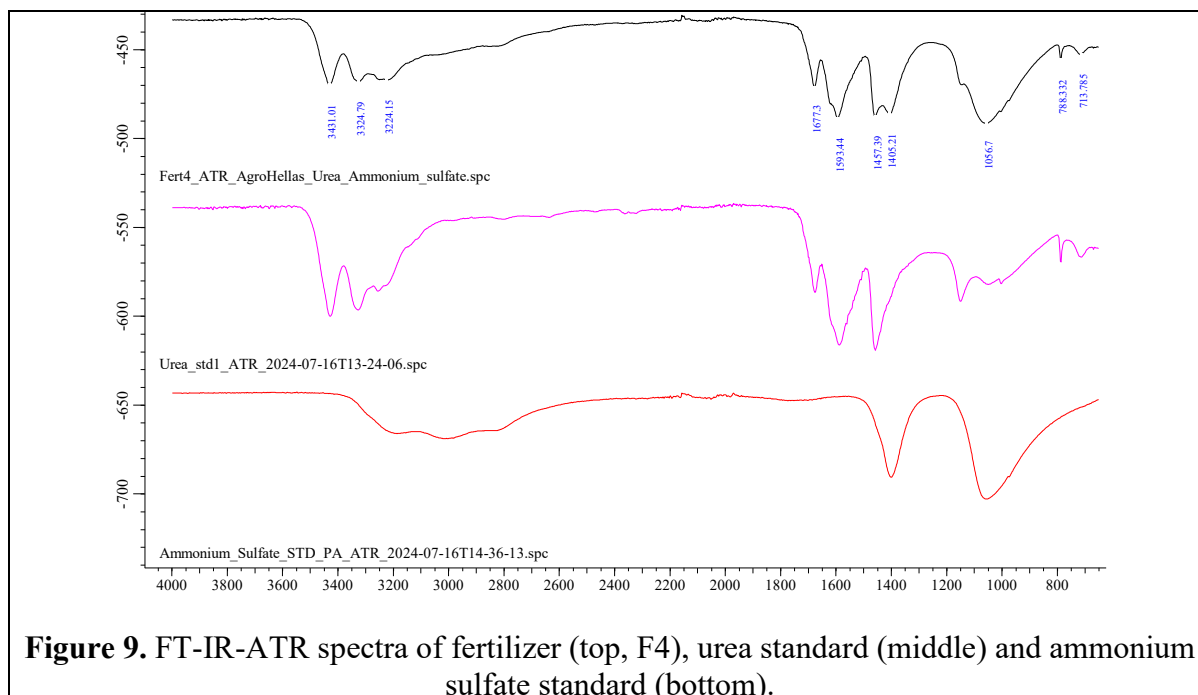
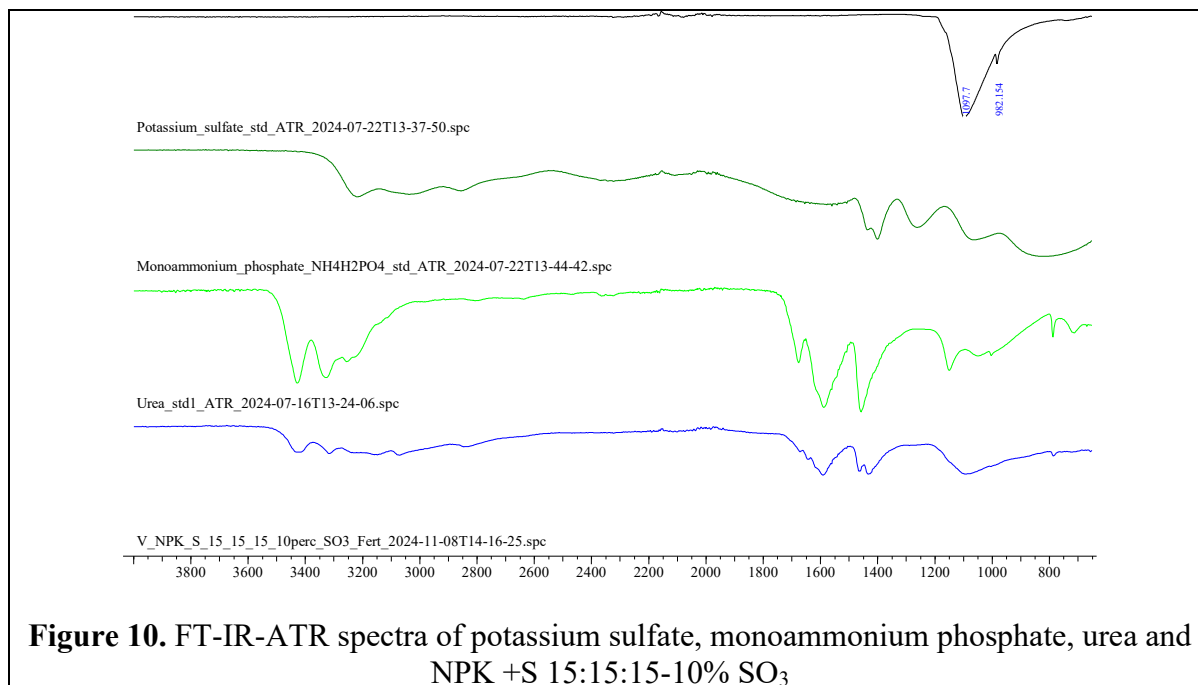


Figure 8. FT-IR-ATR spectrum of AgroHellas NPK-S 33-0-0-11 fertilizer (sample F4)

On this IR spectrum, three types of mineral fertilizers were analyzed — single-component, two-component, and multi-component fertilizers — originating from different manufacturers. Clear differences between their spectra can be observed, reflecting the distinct chemical composition and structure of each fertilizer type (Figure 9).



On this IR spectrum, four types of mineral fertilizers were analyzed — single-component, two-component, and multi- or multi-element fertilizers — originating from different manufacturers, where clear differences between their spectra can be distinctly observed (Figure 10).



For the two fertilizer samples (F1 and F9), in addition to the FT-IR-ATR spectroscopic analyses, standard chemical analyses were also performed. Table 1 shows the presence of amide nitrogen and ammonium nitrogen (NH_4^+). The amide nitrogen originates from urea, which can also be observed in the FT-IR-ATR spectrum. The potassium likely originates from

potassium chloride (KCl) or partially from potassium sulfate (K_2SO_4). It should be noted that potassium chloride cannot be detected by IR spectroscopy.

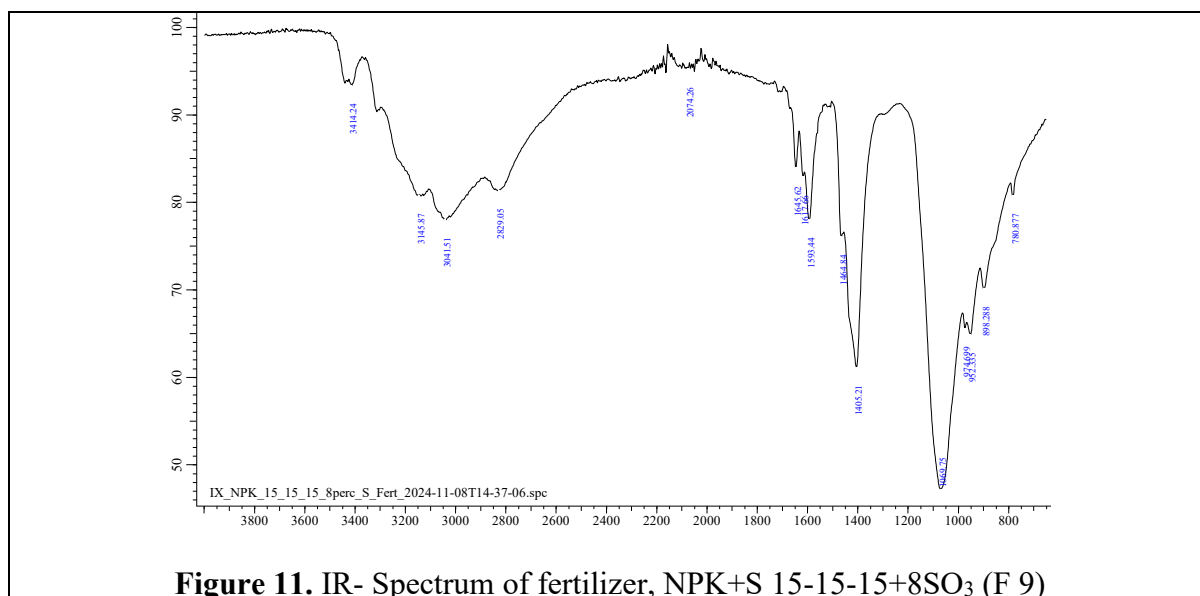


Figure 11. IR- Spectrum of fertilizer, NPK+S 15-15-15+8SO₃ (F 9)

Four types of mineral fertilizers are placed on this IR spectrum. Ammonium sulfate, Urea, Monoammonium phosphate, NPK 15:15:15+8SO₃.

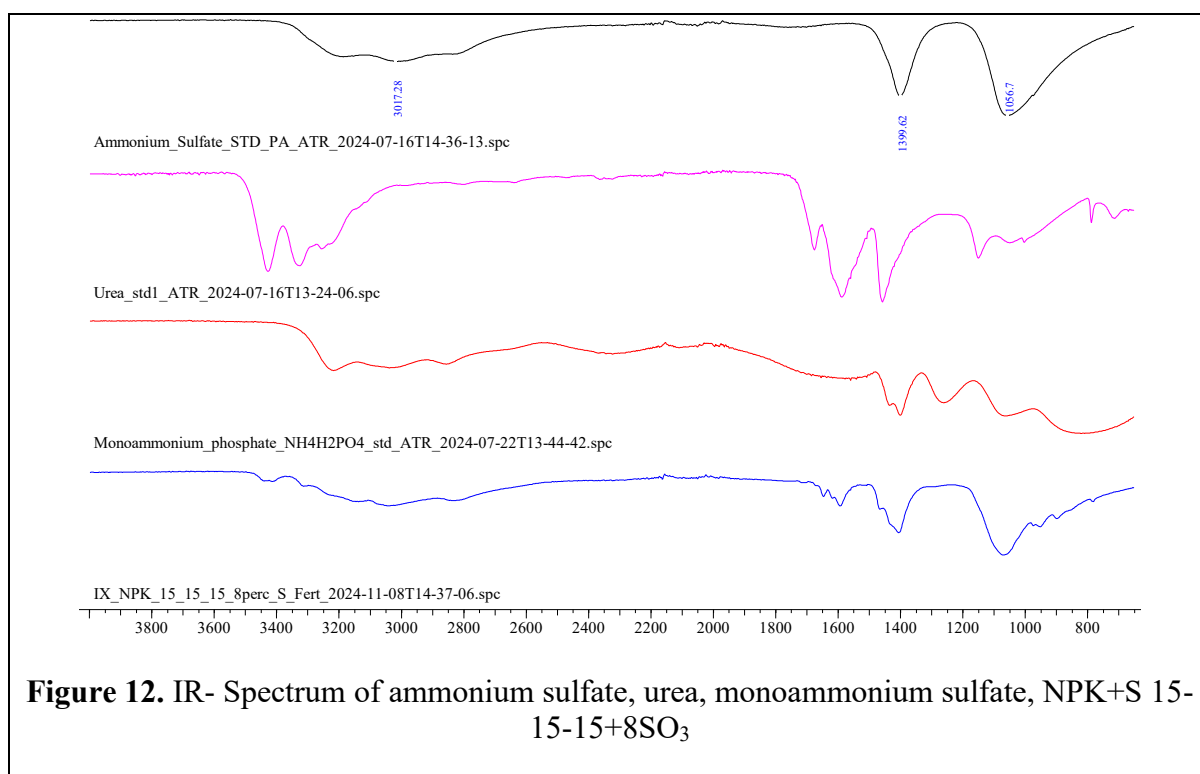


Figure 12. IR- Spectrum of ammonium sulfate, urea, monoammonium sulfate, NPK+S 15-15-15+8SO₃

Table 1. Results of chemical analyses of inorganic fertilizers containing sulfur NPK-S 15:15:15+5% SO₃ (F1)

Chemical parameter	%
Total nitrogen	15.01
Nitrogen in ammonia form	4.01
Amide nitrogen	11.01
Total phosphorus	15.02
Phosphorus soluble in neutral ammonium citrate	7.01
Phosphorus soluble in water	5.02
Potassium soluble in water	15.01
Sulfur in the form of SO ₃	5.01

Table 2 shows that standard chemical analyses indicate nitrate nitrogen, most likely from potassium or ammonium nitrate, and that there is no amide nitrogen present. The remainder is in the ammoniacal form (NH₄⁺).

Table 2. Results of chemical analyses of inorganic sulfur-containing fertilizer NPK 15:15:15+8% SO₃ (F9)

Chemical parameter	%
Total nitrogen	15.01
Nitrogen in ammoniacal form	9.00
Nitrate nitrogen	6.01
Total phosphorus	15.00
Water-soluble phosphorus	13.50
Water-soluble potassium	15.02
Sulfur in the form of SO ₃	8.00
Zn	0.010

CONCLUSION

The characteristic absorption bands of sulfate anions (SO₄²⁻), particularly in the 1200–900 cm⁻¹ region, were successfully used as diagnostic indicators for fertilizers containing sulfur. Samples with sulfur content above 8% exhibited clear and well-defined sulfate bands, whereas those with lower sulfur content (<5%) showed weak or undetectable signals. Through FT-IR-ATR spectra, fertilizers of different composition—single-component, double, and complex multi-nutrient types—could be distinguished based on their specific vibrational patterns. The comparison of spectra from different manufacturers also revealed distinct spectral features attributable to formulation and raw-material differences. The combined use of spectroscopic (ATR-FTIR) and classical chemical methods (titrimetry, UV-Vis spectrophotometry, flame photometry, Kjeldahl, and ICP-OES) provided consistent results and confirmed the presence and forms of nitrogen (amide and ammonium), phosphorus, potassium, and sulfur compounds. Overall, ATR-FTIR spectroscopy proves to be an efficient complementary tool for fertilizer quality control, allowing rapid screening, identification of adulteration or impurities, and verification of label composition. When integrated with standard ISO analytical procedures, it contributes to more comprehensive fertilizer characterization and supports the implementation of sustainable agricultural practices through accurate nutrient management.

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