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# Analysis of Nutrient Elements in Soils using the Agilent 5110 VDV ICP-OES

## Fast, high-throughput analysis of AB-DTPA universal soil extracts

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## Introduction

Soil is a natural source of the nutrients that plants and crops need for growth and to produce good yields. Soils contain different levels of minerals, organic matter, water, and air. To improve the use of fertilizers and to better target their use, it is important to analyze the different components of soil, including elemental nutrients. The Government of India has launched a Soil Health Card (SHC) scheme through the Department of Agriculture and Co-operation under the Ministry of Agriculture and Farmers' Welfare (*1*). The SHC is being implemented through the Department of Agriculture of all the State and Union Territory Governments.

An SHC provides each farmer with information on the nutrient status of the farm's soil and offers advice on how to maintain soil health. Under the SHC scheme, farmers receive a printed report for each of their holdings every three years. The SHC includes status data on 12 parameters namely N, P, K (major/macro nutrients), S (secondary-nutrient), Zn, Fe, Cu, Mn, B (micronutrients), pH, electrical conductivity (EC), and organic content (OC). Depending on the nutrient status of the soil, the SHC will include recommendations on the required dosage of different nutrients. Also, it advises the farmer on the type and quantities of fertilizers they should apply, and other soil amendments to optimize crop yields.

India is an agriculture-based economy with more than 50% of the country's workforce depending on agriculture. Given the scale of the agricultural sector, the SHC scheme requires large numbers of soil samples to be processed. A robust, high throughput, and accurate method is needed for the routine determination of available nutrients in soil.

In this study, the Agilent 5110 Vertical Dual View Inductively Coupled Plasma-Optical Emission Spectrometer (ICP-OES) was used for the analysis of eight nutrients in soil extracts prepared using the AB-DTPA universal soil extraction procedure (2, 3). While other soil extraction methods can be used after appropriate optimization and validation, the AB-DTPA method simplifies the analytical workflow and is suited to high throughput applications. The elements included macronutrients, P and K, secondary-nutrient, S, and micronutrients, Zn, Fe, Cu, Mn, and B.

## Experimental

### Instrumentation

A true-simultaneous Agilent 5110 Vertical Dual View (VDV) ICP-OES was used. The sample introduction system consisted of a double-pass glass cyclonic spray chamber, a OneNeb Series II nebulizer, and a standard 1.8 mm ID injector standard one piece easy-fit torch. An Agilent SPS 4 autosampler was used for the fast and automated delivery of the samples to the ICP-OES.

The 5110 uses a solid-state radio frequency (SSRF) system operating at 27 MHz to produce a robust plasma that is capable of excellent long-term analytical stability during the analysis of soil extracts. The high speed (1 MHz) VistaChip II CCD detector enables fast warm up, fast analysis times, and high sensitivity, as required by soil testing laboratories. The VistaChip II measures the full wavelength range simultaneously from 167 to 785 nm from a single entrance slit without the need for multiple detectors or slits. The detector also has a large dynamic range of up to eight orders of magnitude allowing for both macro- and micro- nutrients to be analyzed from a single sample preparation method. The instrument operating conditions used are listed in Table 1.

**Table 1.** 5110 VDV ICP-OES operating parameters.

| Parameter | Setting |
|---|---|
| Read time (s) | 3 |
| Replicates | 3 |
| Sample uptake delay (s) | 12 |
| Stabilization time (s) | 5 |
| Rinse time (s) | 5 |
| Pump speed (rpm) | 12 |
| Fast pump (rpm) | 80 |
| RF power (kW) | 1.40 |
| Auxiliary flow (L/min) | 1.0 |
| Plasma flow (L/min) | 12.0 |
| Nebulizer flow (L/min) | 0.7 |
| Viewing mode | Radial |
| Viewing height (mm) | 8 |
| Sample pump tubing | White/White |
| Waste pump tubing | Blue/Blue |
| Background correction | Fitted |

## Standard and sample preparation

### AB-DTPA extraction solution

The AB-DTPA universal method extraction solution was composed of 0.005 M of di-ethylenetriaminepentaacetic acid (DTPA) and 1 M ammonium bicarbonate ($NH_4HCO_3$). To make the solution, 1.97 g DTPA and 79.06 g of $NH_4HCO_3$ were dissolved in distilled water. The pH was maintained at 7.6 while making up the volume to 1000 mL using distilled water.

### Extraction procedure and analysis

Soil was collected from the northern region of India. The soil was air-dried and sieved (2 mm sieve). 10 g samples of soil were weighed into 250 mL conical flasks and 20 mL of AB-DTPA solution was added. The flasks were shaken for 15 min at 180 RPM, then filtered through 2.5 µm filter paper (Whatman grade 42, Sigma-Aldrich) and diluted 1:1 using distilled water. The samples were spiked with known concentrations of analytes to test the recoveries of the required elements. The samples and spiked samples were then loaded on the SPS 4 autosampler, ready for analysis, as shown in Figure 1.

**Figure 1.** Sample extraction and analytical workflow.

## Standard preparation

Calibration standards for all the required elements were prepared using Agilent single element standards (1000 mg/L), as detailed in Table 2. The standards were diluted using the AB-DTPA extraction solution to ensure matrix matching. Linear calibrations were obtained for all analytes and wavelengths, with calibration coefficients greater than 0.999, as shown in Table 3. The concentration of the calibration solutions for B, Fe, K, and S are shown in the calibration curves (Figure 2).

**Table 2.** Working calibration concentration range.

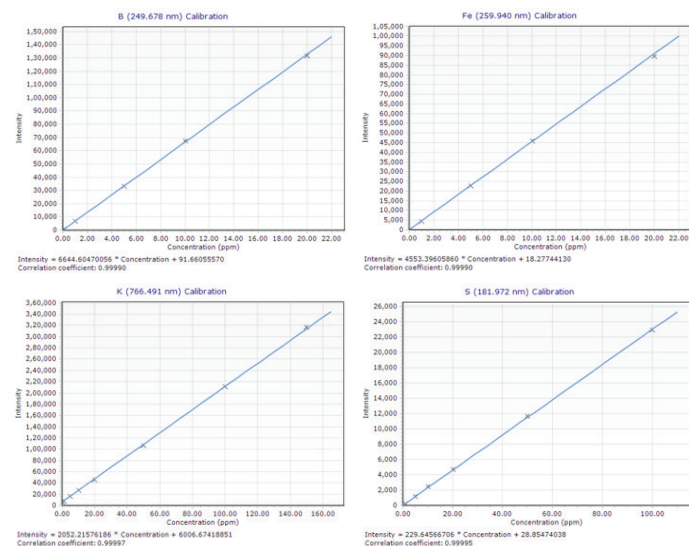| Standard | Elements | Calibration Range (ppm) |
|---|---|---|
| 1 | Cu, Fe, Mn, Zn, B | 0.1 to 20 |
| 2 | P, S | 1.0 to 100 |
| 3 | K | 1.0 to 150 |



**Figure 2.** Calibration curves for B 249.678 nm, Fe 259.940 nm, K 766.491 nm, and S 181.972 nm.

# Results and discussion

## Method Detection Limits (MDLs)

The Method Detection Limits (MDLs) in Table 3 were based on 3 sigma of 10 replicate measurements of the blank AB-DTPA extraction solution taken during analytical run.

**Table 3.** Method detection limits.

| Element | Wavelength (nm) | MDL (µg/g) | Correlation Coefficient |
|---|---|---|---|
| Boron | 249.678 | 0.017 | 0.99990 |
| Copper | 327.395 | 0.009 | 0.99991 |
| Zinc | 202.548 | 0.003 | 0.99970 |
| Manganese | 259.372 | 0.003 | 0.99973 |
| Iron | 259.940 | 0.001 | 0.99990 |
| Phosphorus | 213.618 | 0.020 | 0.99995 |
| Sulfur | 181.972 | 0.021 | 0.99995 |
| Potassium | 766.491 | 0.730 | 0.99997 |

## Sample analysis

The eight nutrient elements were measured in an extracted soil sample, as shown in Table 4. The extracted soil sample was also spiked with 2 mg/kg B, Cu, and Zn; 4 mg/kg Mn, Fe, and P; and 5 mg/kg S and K. All spike recoveries were between 90 to 110%, demonstrating the capability using the 5110 VDV ICP-OES to analyze multiple nutrient elements with good accuracy.

**Table 4.** Spike recovery results for extracted soil nutrients.

| Element | Wavelength (nm) | Extracted Soil Sample Concentration (mg/kg) | Spiked Concentration (mg/kg) | Measured Spiked Concentration (mg/kg) | Recovery (%) |
|---|---|---|---|---|---|
| Boron | 249.678 | 1.21 | 2 | 3.26 | 102.5 |
| Copper | 327.395 | 2.43 | 2 | 4.56 | 106.5 |
| Zinc | 202.548 | 1.42 | 2 | 3.47 | 102.5 |
| Manganese | 259.372 | 20.4 | 4 | 24.6 | 105.0 |
| Iron | 259.940 | 9.07 | 4 | 13.3 | 105.8 |
| Phosphorus | 213.618 | 5.96 | 4 | 10.2 | 106.0 |
| Sulfur | 181.972 | 35.2 | 5 | 39.9 | 94.0 |
| Potassium | 766.491 | 102.7 | 5 | 107.7 | 100.0 |

## Long-term stability

A long-term stability test was carried out by analyzing a spiked sample solution continuously over three hours. The % relative standard deviation (% RSD) of the measured element results is shown in Table 5. The %RSD of all elements was less than 2%. The results demonstrate the robustness and precision of the method over the extended run, which is due to the SSRF system and vertically oriented torch on the 5110 VDV ICP-OES.

**Table 5.** Long-term stability results (% RSD) for extracted soil elements.

| Element | Wavelength (nm) | % RSD |
|---|---|---|
| Boron | 249.678 | 1.72 |
| Copper | 327.395 | 1.62 |
| Zinc | 202.548 | 1.63 |
| Manganese | 259.372 | 1.82 |
| Iron | 259.940 | 1.60 |
| Sulfur | 181.972 | 1.56 |
| Phosphorus | 213.618 | 1.64 |
| Potassium | 766.491 | 1.65 |

## Sample analysis time

Sample-to-sample analysis time was only 31 seconds for all elements using the 5110 VDV ICP-OES with SPS 4 autosampler. The fast analysis time and subsequent low gas consumption is due to many features of the 5110, including the high-speed Vista Chip II CCD detector. The Vista Chip II detector reads all wavelengths simultaneously irrespective of elemental concentrations, rather than bracketing high emission wavelengths and low emission wavelengths into separate sequential measurements like other simultaneous detectors. Considering all gas flows into the ICP-OES, not just the torch gas flows, the total argon consumption was calculated at 9.0 L per sample.

## Conclusion

The Agilent 5110 VDV ICP-OES with SPS 4 autosampler was used for the determination of eight available nutrients in soils prepared using the AB-DTPA universal soil extraction procedure.

The excellent spike recovery test results of a soil extract showed the accuracy of the method for the measurement of macro, secondary, and micronutrients in soils. Also, the 5110 VDV ICP-OES achieved excellent stability over three hours of continuous measurement of a spiked soil sample. The analysis run time was only 31 s with 9.0 L of total argon consumption per sample.

Fast, robust, low-cost analysis is important for soil testing laboratories undertaking large-scale analysis of soil samples as required by the Indian Government's Soil Health Card (SHC) scheme. The data contained in the SHC enables farmers to use fertilizers more effectively and efficiently to maximize crop yields, while protecting the environment from overuse.

## References

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