

ICP-OES Analysis of Phosphorus in Soils Extracted using the Lancaster Leachate Method

Accurate analysis of soils by the Agilent 5900 ICP-OES

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Figure 1. Soil samples.

Measuring the total phosphorus content of soils

Phosphorus (P) plays a key role in plant growth and reproductive processes, so it is a vital nutrient—alongside nitrogen—for crop production. Traditionally, phosphate has been determined using colorimetric analysis and the molybdenum blue method. However, the analysis is time-consuming, cannot determine the other inorganic forms of P that may be present in soils, or quantify other elements in a single measurement (1, 2). Using the Lancaster leachate method (3), total P can be extracted and quantified using inductively coupled plasma-optical emission spectroscopy (ICP-OES). The acidic leaching solution increases the solubility of mineral P in order to extract it for analysis.

Lancaster leaching solution: The solution comprised 400 mL of acetic acid and 300 mL of 10 M lactic acid dissolved in 6 L of distilled water. 22.2 g of ammonium fluoride, 133.3 g of ammonium sulfate, and 170 g of sodium hydroxide were added. When cool, the solution was made up to 20 L with distilled water. The pH was adjusted to 4.25 ± 0.05 by adding solution 1 (to raise pH) or solution 2 (to lower pH). Both solutions 1 and 2 comprised 20 mL of acetic acid and 15 mL of 10 M lactic acid dissolved in 300 mL of distilled water. 1.11 g of ammonium fluoride, 6.665 g of ammonium sulfate, and 17 g of sodium hydroxide were added to solution 1, and left to cool before making up to 1 L with distilled water. The same method was used to prepare solution 2, except no sodium hydroxide was added.

Extraction procedure and analysis: 5 g of soil sample was combined with 20 mL of the leaching solution. The mixture was shaken for 10 minutes and filtered with grade 2 filter paper. The filtrate was analyzed using an Agilent 5900 Synchronous Vertical Dual View (SVDV) ICP-OES fitted with the standard sample introduction system.

Automatic calculation of concentration: The effective P_2O_5 concentration was calculated by accounting for the 5 g in 20 mL volume and the mass conversion from P to P_2O_5 . The conversion factor was 9.16. This conversion was performed automatically using the 'custom column' feature of the Agilent ICP Expert software (Figures 2 and 3).

Automatic calculations in ICP Expert

It is easy to add, edit, or remove custom columns in the ICP Expert software, as shown in Figures 1 and 2.

Tube	Solution Label	P_A 177.434 nm ppm	P_A 213.618 nm ppm	P_Axial 213.618nm P2O5 ppm
6	Standard 5	25.00	25.00	
7	Standard 6			
8	Rinse	0.27	-0.06 u	-0.58
9	Sample 27-1	0.02	0.05	78.79
10	Sample 2			6.62
11	Sample 2			6.01
12	Sample 2			
13	Sample 2			
14	Sample 2			4.02

Figure 2. Custom column applied for P 213.618 nm axial view, automatically reporting the effective P₂O₅ concentration.

Element	Wavelength (nm)	Label	Type
P	177.434	P_R	Analyte
P	177.434	P_A	Analyte
P	177.434	P_S	Analyte

Figure 3. Custom column editing window for the application of formulas and conversions, simplifying data analysis.

Method optimization

Two certified reference materials (CRMs), labeled 27 and 28, were provided by the Rural Development Administration in Korea. The CRMs were analyzed multiple times by the 5900 ICP-OES at different RF power and nebulizer flow rates. The nebulizer flow was a more important factor than RF power due to the high levels of salt in the leachate. RF power of 1.2 kW and 0.65 L/min nebulizer flow provided the best results for the determination of P in soil extracts.

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Evaluating the method

P was measured in two extracted soil CRMs using the 5900 in axial, radial, and SVDV mode. The results for P 177.434 and 213.618 nm are shown in Table 1. The RSDs for all measurements between 0.26 and 2.79% confirmed the robustness of the method, irrespective of viewing mode. Good accuracy was achieved for P₂O₅ in the two soil CRMs with a recovery within ±5% for both P wavelengths in all view modes.

Table 1. Recovery data for P in two soil CRMs, 27 and 28, n=5.

Element, Wavelength (nm)	Expected P ₂ O ₅ in CRM (mg/kg)	Viewing Mode	Measured P in Extracted Soil (mg/kg)	Calculated P ₂ O ₅ in Soil (mg/kg)	RSD (%)	Recovery (%)
CRM 27						
P 177.434	77.8 ± 9.3	Axial	8.80	80.6	1.16	104
		Radial	8.36	76.6	2.79	99
		SVDV	8.68	79.5	1.29	102
P 213.618		Axial	8.83	80.9	1.12	104
		Radial	8.24	75.4	2.05	97
		SVDV	8.79	80.5	1.53	104
CRM 28						
P 177.434	74.1 ± 10.3	Axial	8.19	75.1	0.26	101
		Radial	7.71	70.6	1.14	95
		SVDV	8.12	74.4	0.88	101
P 213.618		Axial	8.23	75.4	0.48	102
		Radial	7.72	70.7	1.47	96
		SVDV	8.22	75.3	0.97	102

Conclusion

The study showed the suitability of the Agilent 5900 SVDV ICP-OES for the measurement of available P in soils prepared using the Lancaster extraction method. The method provided fast, accurate, and precise results for P, reported as P₂O₅. Plus the 5900 can be used to quantify many more elements in soils, adding flexibility to the lab compared to single element colorimetric testing techniques.

References

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