

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

Accurate analysis of soils by the Agilent 5900 ICP-OES

#### Authors

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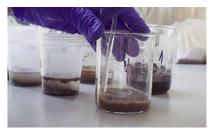


Figure 1. Soil samples.

## 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  $4.25 \pm 0.05$  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 P_A 177.434 nm 213.618 nm ppm ppm		P_Axial 213.618nm P2O5 ppm			
6	Standard 5	25.00	25.00				
7	Standard 6						
8	Rinse	0.27	-0.06 u		-0.5		
9	Sample 27 4	Сору	0.05	Ctrl+C	712		
10	Sample 2	Copy (with h	neaders)	Ctrl+Shift+C	ft+C 6.62		
11	Sample 2	Add Custom			6.0 <sup>-</sup>		
12	Sample 2	Edit Custom Column Remove Custom Column Column Properties					
13	Sample 2						
14	Sample 2	Export selected solutions					

Figure 2. Custom column applied for P 213.618 nm axial view, automatically reporting the effective  $P_2O_5$  concentration.

Edit Custor	m Column					
Name	P_Axial 21	3.618nm	205			
Equation	① P_A 213. ③	618 *	20 / 5 ) *	( 141.9445 )	61.9474 )	
Elemen	ts					
Element	Wavelength (nm)	Label	Туре			
Р	177.434	P_R	Analyte			
р	177.434	P_A	Analyte			
Р	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.

#### www.agilent.com/chem/5900icp-oes

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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  $P_2O_5$  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 <sub>2</sub> O <sub>5</sub> in CRM (mg/kg)	Viewing Mode	Measured P in Extracted Soil (mg/kg)	Calculated $P_2O_5$ 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
		Axial	8.83	80.9	1.12	104
P 213.618		Radial	8.24	75.4	2.05	97
		SVDV	8.79	80.5	1.53	104
	CRM 28					
	- 74.1 ± 10.3	Axial	8.19	75.1	0.26	101
P 177.434		Radial	7.71	70.6	1.14	95
		SVDV	8.12	74.4	0.88	101
		Axial	8.23	75.4	0.48	102
P 213.618		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  $P_2O_2$ . 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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