

Direct Determination of Phosphorus by Atomic Absorption

Application Note

Atomic Absorption

Introduction

The determination of phosphorus has traditionally been carried out via solution spectrophotometric techniques. The procedure usually involves formation of a phosphomolybdate complex and subsequent measurement at approximately 650 nm.

Direct atomic absorption phosphorus measurements have not been routinely made because of a number of difficulties:

- 1. The primary resonance lines (most sensitive) are in the vacuum UV at 177.50, 178.29, and 178.77 nm. Measurements at these wavelengths require an evacuated monochromator and a nitrogen purge between the source and flame (or furnace), and between the flame and monochromator. There are no commercial AAs available with this capability.
- 2. It is not currently possible to manufacture a hollow cathode lamp with an intense, narrow line spectrum and acceptable stability.

Recent advances in spectroscopic light sources have brought us electrodeless discharge lamps (EDLs), and phosphorus is one of those elements for which an EDL is available. Due to the high Intensity of the phosphorus EDL, the secondary resonance line doublet at 213.62 and 213.55 nm may be used in place of the primary resonance lines. This allows measurement on conventional AA instruments.



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Phosphorus is determined in the nitrous-oxide/acetylene flame using typical conditions listed in Table 1.

 Table 1.
 Instrumental Conditions for the Determination of Phosphorus by
 Flame AA

Wavelength	213.6 nm
Spectral band width	0. 2 nm.
Flame condition	Strongly reducing, slightly luminous
EDL power	6 watts

Using these conditions, the characteristic concentration (sensitivity, expressed as ppm phosphorus/0.0044 absorbance) is approximately 125 pg/mL. The detection limit, based on 10 baseline-corrected measurements, is 40 pg/mL. A typical calibration curve obtained in the nitrous oxide/acetylene flame is shown in Figure 1.



Figure 1. Calibration curve for phosphorus nitrous-oxide/acetylene flame.

The limiting factor for the upper concentration range limit in the flame is the ability to successfully aspirate solutions at these concentrations. Fifty thousand ppm is equal to 5% phosphorus. Based on the molecular weight of $NH_4H_2PO_4$ (which was used for this study) of 115, the total dissolved solids in the 50,000 ppm phosphorus solution is 18.5% (W/V). Solutions with such high dissolved solids quickly clog the nebulizer after only a few seconds of aspiration. It is possible, however, to accommodate such high concentrations using flame microsampling techniques [1,2].

Phosphorus can also be determined with some success in the carbon atomizer. The analytical conditions are listed in Table 2.

 Table 2.
 Instrumental Conditions for the Determination of Phosphorus by Carbon Rod AA

Wavelength	213.6 nm
Spectral band width	0. 2 nm
Maximum ash temperature	1500 °C
Atomize temperature	2400 °C
Ramp rate	600 °C/sec
Background correction	Yes
Sheath gas	N ₂
Matrix	0.2% La (as the nitrate)
Measurement mode	Peak height

A typical calibration curve for phosphorus by carbon rod atomization is shown in Figure 2. The characteristic concentration is 14 ng (or 0.7 μ g/mL using a 20 μ L injection volume) and the detection limit is 4 ng (or 0.2 pg/mL using a 20 μ L injection volume). Actual chart recorder tracings showing response and precision for phosphorus by carbon rod atomization are shown in Figure 3.

The use of lanthanum as a matrix modifier is an important one for two reasons. First, the lanthanum-phosphorus complex, which is formed, permits a higher ash temperature without loss of phosphorus. Second, the addition of lanthanum stabilizes the phosphorus and allows reproducible signal responses independent of the other major matrix constituents and their corresponding interferences on phosphorus. This fact is very important, because the types of samples in which phosphorus is typically determined (agricultural, nutritional, biological) are often complex in composition with more than one interfering component.

In order to further assure the integrity of results for phosphorus by carbon rod AA, the method of standard additions is recommended. A typical determination of phosphorus in plant material is shown in Figure 4. The sample is NBS Standard Reference Material 1571 (orchard leaves). The determination was carried out on an AA-775 and CRA-90 using the built-in standard additions mode on the instrument. Both the computed results and graphical results compare favorably with the certified value.



Figure 2. Calibration curve for phosphorus carbon rod atomizer (CRA 90).



Figure 3. Phosphorus by carbon rod.



Figure 4. Phosphorus in SRM #1571.

A summary of the figures of merit for the determination of phosphorus by both flame and carbon rod AA is given in Table 3.

Table 3. Summary of Figures of Merit for Phosphorus

	Flame	Carbon rod
Characteristic concentration	125 mg/L	0.7 mg/L ¹
Detection limit	40 mg/L	0.2 mg/L ¹
Normal working range ²	400–40,000 mg/L	2–300 mg/L ¹
1 Perced on 20 ul inicat	ion volumo	

1 Based on 20 µL injection volume

2 The normal working range corresponds roughly to 10 times the detection limit, at the low end, to the concentration which corresponds to ~0.8 Abs.

References

- R. C. Fry, S. J. Northway, and M. B. Denton, Anal. Chem., 50, p1719 (1978).
- T. N. McKenzie, Varian Techtron Lecture Transcript, Part Number 85 100349-00, (1979).

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