

Determination of Trace Amounts of Phosphorus in Silicate Materials by Simultaneous Inductively Coupled Plasma Atomic Emission Spectrometry

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Inductively coupled plasma atomic emission spectrometry (ICP-AES) implemented on a simultaneous spectrometer was used for the determination of trace amounts of phosphorus in rocks, soils and sediments. Samples were decomposed by using a mixture of concentrated hydrofluoric, perchloric and nitric acid in Teflon beakers. Overlap of emissions from other elements present in the samples with the polychromator spectral line at 214.914 nm for phosphorus was studied. The corresponding inter-element coefficients for overlap correction were determined and the optimum position for background readings was established. The limit of determination achieved was 24 ppm referred to the solid sample. The proposed method was tested by applying it to the analysis of rock, soil and sediment certified reference materials, and determining its short-time precision and method precision, which turned out to be 0.24–0.68% and 1.70–4.16% (relative standard deviation), respectively.

Keywords: Inductively coupled plasma atomic emission spectrometry; phosphorus determination; rock; soil; sediment

Introduction

Phosphorus occurs widely in the Earth's shell, which contains approximately 0.23% of P_2O_5 . Most of the phosphorus is present in minerals of the apatite $[3Ca_3P_2O_5Ca(Cl,F)_2]$ group. Small amounts of VO_4 , AsO_4 and SiO_4 can substitute for PO_4^{3-} , and Na, Sr, U, Th and lanthanoid metals may replace Ca in the parent structure.

In relation to the natural geological sedimentation cycle,¹ apatite is relatively insoluble in alkaline or neutral waters. Its solubility increases with increasing acidity and decreasing temperature. Thus, although apatite may be residually concentrated in the first stages of weathering, it eventually breaks down. The soluble phosphorus from apatite binds to organic matter during sedimentation to produce primarily amorphous and black shales.

The phosphorus content in igneous rocks ranges from 600 ppm in granites to 1200 ppm in the most alkaline rocks. Exceptionally, some alkaline igneous rocks and highly developed granites may contain even higher proportions.

Phosphorus is of great geochemical interest for several reasons. Thus, (i) it is a good lithological indicator associated with magmatic rocks, (ii) it separates certain facies between alkaline rocks or granites and (iii) it differentiates clay lithologies (black lutites or amorphous) from other types of lithology in sedimentation media.

For the above reasons, the inclusion of phosphorus determinations in geochemical programmes is of great significance.

The general literature on silicate materials abounds with references to the determination of phosphorus. The instrumental techniques most frequently used for this purpose include spectrophotometry,^{2–12} atomic absorption spectrometry^{13–15} and X-ray fluorescence spectrometry.^{16–20} Direct current plasma (DCP) and inductively coupled plasma atomic emission spectrometry (ICP-AES) have also been used for determining phosphorus in silicate materials despite their poor sensitivity to this element and the weak emission lines²¹ typically obtained (phosphorus is one of the elements with the highest ICP detection limits.²² Bankston *et al.*²³ used the 214.914 nm line for the DCP determination of phosphorus following sample dissolution by $LiBO_2$ fusion in a graphite crucible. Cook and Miles,²⁴ Burman²⁵ and McLaren *et al.*²⁶ used the lines at 178.287, 213.618 and 214.914 nm, respectively, to determine phosphorus by ICP after dissolving samples with HF in a platinum or (PTFE) vessel. Xu *et al.*²⁷ investigated spectral interferences in the determination of phosphorus in steel by using an échelle spectrometer. They found the P I 213.618 nm line to be free from any interference from the iron line, but partly overlapped with the wing of the Cu II 213.598 nm line. Kanda and Taira²⁸ suggested that the 214.914 nm line is suitable for soil and sediment analysis. However, the line is subject to a severe background shift interference from the Cu II 214.897 nm line.

The excellent multi-element excitation capabilities of the ICP technique have been shown also to result occasionally in serious spectral interferences arising from line overlap and background shifts in geochemical determinations of phosphorus. Background shifts can often be corrected by making off-line background measurements using a quartz refractor technique. In those instances where some spectral interference is unavoidable, correction coefficients are determined by nebulizing ultrapure matrix solutions following calibration under the same working conditions as those to be used in the subsequent analyses. If the interference originates in a polychromator channel, then the correction factor can readily be applied to the interfered element.

The most sensitive lines for phosphorus determination by air-path ICP-AES are the UV lines at 213.618 and 214.914 nm. The purpose of this work was to investigate spectral interferences with the emission line of phosphorus at 214.914 nm (*viz.*, the phosphorus line set in our spectrometer polychromator) and obtain a coincidence scan for those of the interferents. Interferent scans were required for appropriate selection of a corrective procedure.

Experimental

Apparatus and Operating Conditions

A Jarrell Ash ICAP-62 simultaneous spectrometer equipped with an ICP source was used. The spectrometer was furnished with a polychromator of 1500 grooves mm^{-1} accommodating 35 photomultiplier tubes. The specifications of the ICP source were as follows: frequency, 27.12 MHz; induced power, 1000 W; and reflected power, <5 W. The operational conditions used were as follows: plasma gas (argon) flow rate, 171 min^{-1} ; aerosol carrier gas (argon) flow rate, 0.75 l min^{-1} ; coil cooling water flow rate, 1.5 l min^{-1} ; sample introduction, pneumatic cross-flow nebulizer; sample delivery, Gilson Minipuls-2 peristaltic pump (2 ml min^{-1}); measurement time, 10 integrations at 5 s per step; and observation height, 16 mm.

Reagents

Stock standard solutions of high purity (Riedel-de Haën), HF, HNO_3 and HCl (Suprapur grade, Merck) and distilled water of resistivity less than 18 $\text{M}\Omega \text{ cm}$ were utilized to make all standard and sample solutions.

Samples

The samples tested were eight different certified silicate reference materials supplied by the National Bureau of Standards (NBS), the Canadian Certified Reference Materials Project (CCRMP), the Rat Für Gegenseitige Wirtschaftshilfe (RGW), the Bureau of Standards of the Republic of South Africa (SAR) and the Bureau of Standards of the Republic of China (GBW).

Decomposition of Samples

Samples were decomposed by using the following procedure. In a 100 ml Teflon beaker were placed 1.0000 g of finely ground, previously dried (110°C) sample, 10 ml of concentrated HF, 5 ml of concentrated HClO_4 and 5 ml of concentrated HNO_3 . The mixture was heated to dryness at 190°C on a hot-plate to remove tetrafluorosilicate and residual HF. Then, 5 ml of concentrated HCl and 5 ml of distilled water were added and the residue was heated at 100°C until complete dissolution. The resulting solution was finally diluted to 100 ml with distilled water and stored for subsequent analysis.

Preparation of Standard solutions for Spectral Interference Study

Thirteen single-element working standard solutions were prepared by diluting to 100 ml previously prepared stock standard solutions containing 2 ml of concentrated HNO_3 and 10 ml of concentrated HCl. The element concentrations were as follows: aluminium 1000, iron 1000, calcium 1000, magnesium 500, titanium 250, tungsten 100, cadmium 100, silver 100, zirconium 100, copper 12, vanadium 10, niobium 10, tin 10 and phosphate 3.26–10 $\mu\text{g ml}^{-1}$ (as P). These concentrations are similar to or slightly higher than those typically encountered in silicate rocks, soils and sediments.

Results and Discussion

Spectral Interferences

Spectral interferences were identified and appropriate wavelengths for background correction were chosen by performing short-wavelength scans near the analyte wavelength. Figs. 1–4 show typical overlapped scans obtained for phosphorus at 214.914 nm.

Fig. 1 includes the scans for pure solutions of aluminium, iron, phosphorus and a blank reagent. As can be seen, aluminium caused a strong interference. Pritchard and Lee²⁹ ascribed this effect of aluminium to two complementary effects, namely (i) the presence of a doublet at 214.874 and 214.964 nm that is only observed above 3500 $\mu\text{g ml}^{-1}$ Al and (ii) the presence of a broad, unbroken band that extends to higher wavelengths. The effect of iron was less marked: one of its lines (214.917 nm) overlapped that of phosphorus, and another three lines (214.850, 214.962 and 215.018 nm) only affected its spectral background structure. Calcium, magnesium and titanium gave a flat background that raised that of the phosphorus line slightly but did not alter its measurement position (the scans have been omitted from the figure).

Fig. 2 shows the scans obtained for the copper, vanadium and niobium solutions. Even though these three elements gave peaks near the phosphorus emission line (*viz.*, 214.842 nm for V, 214.872, 214.903 and 214.950 nm for Nb and 214.897 for Cu), only the Cu line and the Nb line at 214.903 nm overlapped that of phosphorus (the others only affected the background measurement position).

Fig. 3 shows the scans for tungsten, zirconium and tin. As can be seen, the emission of phosphorus overlapped two W lines (214.914 and 214.885 nm) and one, uncatalogued Zr line (approximately 214.89 nm). However, the emission of Sn (214.874 nm) and an unknown W line at approximately 214.96 nm should only affect background measurements. While the W, Zr and Sn concentrations in the standards used allowed their interfering effects to be revealed, these levels are hardly reached in rocks, soils and sediments (with the exception of specific mineralization zones).

Fig. 4 shows the spectral scans obtained for a certified soil sample, a blank reagent and two multi-elemental standards of the following compositions: standard A, 1000 $\mu\text{g ml}^{-1}$ Al, Fe and Cu, 500 $\mu\text{g ml}^{-1}$ Mg, 250 $\mu\text{g ml}^{-1}$ Ti and Na, 12 $\mu\text{g ml}^{-1}$ Cu and 10 $\mu\text{g ml}^{-1}$ Nb and V; and standard B, the same as standard A plus 3.26 $\mu\text{g ml}^{-1}$ P.

The similar characteristics of the spectra for both standards and the soil sample and the difference between the two

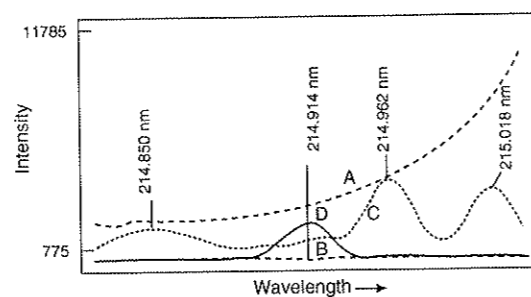


Fig. 1 Spectral scans in the vicinity of the P I 214.914 nm line for A, Al (1000 $\mu\text{g ml}^{-1}$); B, blank; C, Fe (1000 $\mu\text{g ml}^{-1}$); and D, P (3.26 $\mu\text{g ml}^{-1}$).

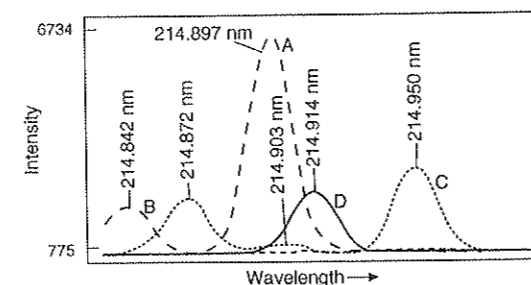


Fig. 2 Spectral scans in the vicinity of the P I 214.914 nm line for A, Cu (12 $\mu\text{g ml}^{-1}$); B, V (10 $\mu\text{g ml}^{-1}$); C, Nb (10 $\mu\text{g ml}^{-1}$); and D, P (3.26 $\mu\text{g ml}^{-1}$).

standards in the P emission region, confirm that the model used to study potential interferences was correct.

The determination of phosphorus entailed a prior selection of the background measurement position and the establishment of correction coefficients for inter-elemental effects.

Measurement of Background

Irrespective of the contribution from the attacking reagents used, the spectral background in the vicinity of the 214.914 nm line for phosphorus was essentially provided by Al and Fe and, to a lesser extent, the Cu, V, Nb, W and Zr lines mentioned in the previous section.

Despite the complexity of ICP emission, accurate background measurements could be made by using a quartz refractor plate behind the spectrometer entrance slit. The plate shifted the beam falling on the grating, thereby effectively changing the angle of incidence. The magnitude of the shift was proportional to the refractor plate angle relative to the incident beam. This allowed spectral scans of ± 0.1 nm to be achieved in the vicinity of the line to be measured.

As can readily be seen from the spectral region provided by this procedure (Fig. 4), the optimum position for measuring the spectral background was 214.876 nm. The background intensity was subtracted from that of the analytical line by using a multiplexed analogue-to-digital converter and computerized data processing.

Correction of Spectral Overlap

In order to overcome spectral overlap of the phosphorus line with those of some interferences that resulted in spurious analyte concentrations, the following equation was used:

$$c_p = A_p - \sum_j K_{pj} c_j$$

where c_p is the corrected concentration of phosphorus, A_p its apparent concentration, c_j the concentration of interferent j ; and K_{pj} the inter-element correction coefficient of element j for phosphorus. K_{pj} values (Table 1) were calculated by

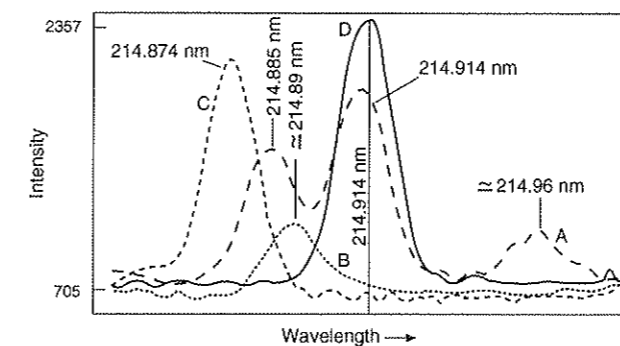


Fig. 3 Spectral scans in the vicinity of the P I 214.914 nm line for A, W (100 $\mu\text{g ml}^{-1}$); B, Zr (100 $\mu\text{g ml}^{-1}$); C, Sn (100 $\mu\text{g ml}^{-1}$); and D, P (3.26 $\mu\text{g ml}^{-1}$).

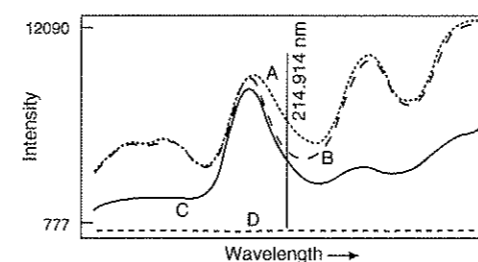


Fig. 4 Spectral scans in the vicinity of the P I 214.914 nm line for A, standard A; B, standard B; C, sample GSD-12; and D, blank.

measuring the emission intensities at 214.914 nm for the single-element solutions. K_{PCu} takes into account the interference of Cu 214.897 nm in the locations of peak and background measurements. The negative K_{PNb} value is due to the interference of Nb 214.872 nm, which affects the background more than the interference of Nb 214.903 nm, which, in turn, affects the peak.

Limit of Determination for Phosphorus

The limit of determination for phosphorus was considered, with a probability greater than 99.87%, as the concentration providing ten readings whose average intensity, decreased by three times their standard deviation, was not overlapped with the average blank intensity (standard A) increased by three times its standard deviation.

In order to calculate both the determination limit and the phosphorus concentration in rocks, soils and sediments that the proposed method could discriminate, nine standards containing 0, 0.25, 0.50, 0.75, 1.00, 1.25, 1.50, 1.75 and 2.00 $\mu\text{g ml}^{-1}$ of phosphate were analysed under the optimum experimental conditions. Table 2 shows the average intensity, standard deviation and variation range for the readings obtained according to the $\pm 3s$ criterion. As can be seen, there was an essentially linear relationship between the average intensity and its standard deviation (Fig. 5). Such a linear relationship allowed a determination limit of 0.75 $\mu\text{g ml}^{-1}$ (equivalent to 24 ppm solid sample) for phosphorus to be calculated and confirmed that attacking solutions applied to the materials studied differing by as little as 0.25 $\mu\text{g ml}^{-1}$ (equivalent to 8 ppm solid sample) in their phosphorus contents could be discriminated with a probability greater than 99.87% over the concentration range 24–100 ppm. The inflection point in the curve of concentration *versus* intensity (0.75 $\mu\text{g ml}^{-1}$ of P) is explained because the lower concentrations are below the limit of determination and, in practice, they do not produce variations in the intensity reading.

Table 1 Inter-element correction coefficients for some of the interferences studied relative to the 214.914 nm line for phosphorus

Interferent	λ/nm	K_{pj} ($\mu\text{g ml}^{-1}$ P/ mg ml^{-1} interferent)
Al	Background continuum	1.6×10^{-3}
Fe	214.917	1.5×10^{-4}
Cu	214.897	0.005
Nb	214.872	-0.05
	214.903	
Zr	214.889	0.003
W	214.914	0.016

Table 2 Data used to establish the limit of determination for phosphorus and the concentration that the proposed method can discriminate in silicate materials

Standard concentration/ $\mu\text{g ml}^{-1}$		Average reading	Standard deviation	Reading variation range
PO_4^{3-}	P			
0	0	23 300	120	22 940–23 660
0.25	0.082	23 500	122	23 134–23 660
0.50	0.163	23 700	125	23 325–24 075
0.75	0.245	24 100	130	23 710–24 490
1.00	0.326	25 100	141	24 523–25 523
1.25	0.408	26 100	150	25 650–26 550
1.50	0.484	27 200	159	26 723–27 677
1.75	0.571	28 200	170	27 790–28 571
2.00	0.652	29 300	181	28 757–29 843

Precision and Accuracy

The precision and accuracy of the proposed method were determined by analysing the above-mentioned eight certified reference materials. Table 3 summarizes the results obtained in the short-term precision testing (ten successive determinations on one sample specimen) and method precision testing (ten determinations on ten different sample specimens). The mean value (\bar{x}), standard deviation (s) and relative standard deviation (s_r) for each assay are given. The precision and accuracy obtained with the two test series were acceptable.

Conclusion

The proposed ICP-AES method allows the determination of phosphorus in silicate materials by use of a direct-reading spectrometer. By correcting spectral line overlap (using empirical inter-elemental coefficients) and choosing an appropriate spectral background measurement position, the

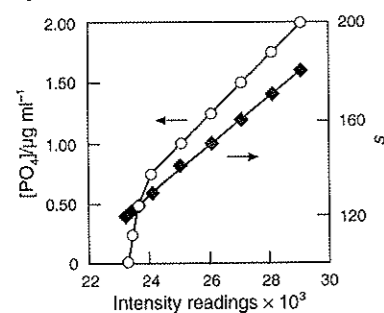


Fig. 5 Variation of the standard deviation (s) with the average intensity readings for the standards.

Table 3 Results obtained in the determination of phosphorus (as % P_2O_5) in eight rock, soil and sediment certified reference samples

Standard	Concentration (%)		Short-term precision, s (%)	Method precision, s_r (%)
	Certified	Found		
SO-1	0.062	0.061	0.62	1.70
SO-2	0.300	0.305	0.54	2.30
GBWO 7401 (GSD-1)	0.0735	0.0695	0.24	1.95
GBWO 7307 (GSD-7)	0.0820	0.0755	0.57	2.81
GBWO 7312 (GSD-12)	0.0235	0.0212	0.62	4.16
NBS 2704	0.10	0.097	0.68	3.32
BM	0.046	0.044	0.49	3.10
SARM 3	0.026	0.023	0.60	3.55

element can be determined with adequate accuracy and precision from the 214.914 nm line in the spectrometer polychromator, which is subject to severe interference from other elements.

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Atomic Absorption Spectrometric Determination of Copper and Lead in Silicon Nitride and Silicon Carbide by Direct Atomization

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A method involving atomic absorption spectrometry (AAS) with direct atomization has been developed for the determination of trace amounts of Cu and Pb in Si_3N_4 and SiC using a graphite furnace. The samples were ground to a particle size of less than 20 μm and mixed with 2.5 times the amount of graphite powder in a corundum mortar. A 0.5–3 mg amount of the mixed sample was weighed in a tared graphite cup and atomized in a cup-type graphite furnace. The absorbance was determined by integration of the spectral lines in the absorbance versus time spectrum. Calibration was effected using aqueous standard solutions. The absorbance versus time spectrum of Cu shows double peaks. On the basis of the X-ray diffraction patterns and scanning electron micrographs of the Si_3N_4 -graphite powder mixture heated at the temperature of each atomization step, the first peak could be assigned to the Cu vaporizing from Si, and the second peak to the Cu vaporizing from SiC. The results for 12 α - Si_3N_4 , two β - Si_3N_4 and two SiC samples were in good agreement with the values obtained by electrothermal AAS for liquid samples. The relative standard deviations for Cu (0.11–53.7 ppm m/m) and Pb (0.36–1.55 ppm m/m) are 1.6–39% ($n = 5$) and 4.3–17% ($n = 5$), respectively. The determination limits, corresponding to twice the standard deviation of the blank measurements, are 92 μg for Cu and 53 μg for Pb.

Keywords: Silicon nitride; silicon carbide; copper and lead determination; direct atomization; electrothermal atomic absorption spectrometry

Introduction

Silicon nitride ceramics have high chemical resistance and excellent mechanical properties at high temperature, and they are used as heat-resisting high-strength materials for turbine blades and ceramic engines. As the existence of the β -phase in raw α - Si_3N_4 powder and the presence of metallic impurities such as Ca, Ti and Fe at the grain boundary of the crystallites of Si_3N_4 ceramics adversely affects the mechanical properties of these ceramics, such as the three-point bending strength at high temperature,¹ trace amounts of metallic impurities have routinely been determined by inductively coupled plasma atomic emission spectrometry (ICP-AES),² particle-induced X-ray emission spectrometry (PIXE)³ and atomic absorption spectrometry (AAS).^{4–6} Usually, ICP-AES and AAS require complicated and tedious procedures for the dissolution of Si_3N_4 samples. Also, these processes are sometimes accompanied by accidental contamination.

Many attempts have been made to carry out analyses by direct atomization of solid samples, and the sampling method has been reviewed by Langmyhr⁷ and Ng.⁸ Katsukov *et al.*⁹ determined Fe in Si_3N_4 and SiC by direct atomization using

electrothermal atomic absorption spectrometry (ETAAS). Graule *et al.*¹⁰ determined 13 trace elements in slurry samples by direct atomization ICP-AES. Because the direct atomization method is free from dissolution and dilution procedures, the development of a highly sensitive and rapid determination, with few contamination problems, is feasible.

However, as a solid or liquid matrix usually generates considerable amounts of background absorption, the effective reduction of the background requires the use of further techniques, including the Zeeman effect method^{11,12} and the Smith-Hieftje method¹³ in correcting the interference. The difficulties in the introduction of samples into the furnace have been reduced by the insertion of (1) a platform into the graphite tube,¹⁴ (2) a cup into the graphite tube furnace¹⁵ and (3) a graphite cup into the cup-type furnace.¹⁶ The selection of calibrating materials is important when a solid sample is atomized directly. Few investigations have been carried out into the use of aqueous standards for calibration, with the exception of Cu in carbonate rocks,¹⁷ minor elements in silicate rocks,¹⁸ Mn and Cu in quartz and rock crystals¹⁹ and metals in ZrO_2 .²⁰ Headridge and Nicholson,²¹ and Atsuya and Itoh,²² however, have used synthetic solid standards having similar compositions to actual samples, which gave more accurate results than those obtained with aqueous standards. The chemical and physical state of the determinants at the atomizing temperature, on which the absorbance and absorbance versus time profiles of the determinant depend significantly, have not yet been investigated, with the exception of Zn, Ag, Pb and Bi in steel²³ and Cu in Al metal.²⁴ Although the absorbance versus time profile for liquid samples shows a single peak, determinants in nitrides, carbides and oxides having high melting-points give complicated profiles.

The present investigation is concerned with a further application of direct atomization AAS to determine Cu and Pb in Si_3N_4 and SiC ceramics by using a graphite cup and a graphite furnace. Discussion will be extended to: (1) particle size of sample, (2) amount of graphite powder to be added, (3) chemical state of samples at pyrolysis and atomization steps, (4) optimization of atomization programme, (5) effectiveness of chemical modifier and (6) optimum amounts of samples to be introduced. The effectiveness of aqueous standard solutions for the calibration of solid samples will also be discussed. The proposed method was applied successfully to the determination of Cu and Pb in several Si_3N_4 and SiC ceramic powders.

Experimental

Apparatus

A Hitachi Z-8000 polarized Zeeman atomic absorption spectrometer was used in conjunction with monoatomic