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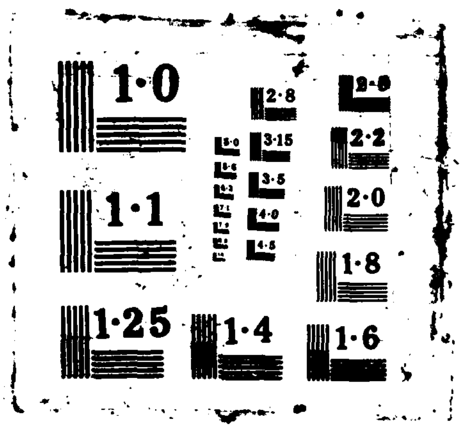
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DETERMINATION OF PHOSPHORUS IN WASTE WATER BY INDUCTIVELY COUPLED PLASMA-ATOMIC EMISSION SPECTROMETRY

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Under the EPA regulations, waste water should not contain more than 5-6mg/L of total phosphorus before it is discharged into a public sewer system. The conventional methods for determining phosphorus in waste are time consuming and also require pretreatment of samples to convert all the phosphorus to the ortho-phosphate form for analysis. Inductively coupled plasma-atomic emission spectrometric method (ICP-AES) has been successfully used to determine phosphorus in waste. This method is fast, accurate and does not require sample pretreatment prior to analysis. Keywords

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INTRODUCTION

Phosphorus is one of the important elements to be detected in environmental, biological, geochemical and metallurgical products. Murphy and Riley[1] proposed an application of phospho-melybdenum heteropoly method for the determination of phosphorus in sea water, and it became one of the most popular analytical techniques. The standard methods for phosphorus[2-4] determination are time consuming and require pretreatment of samples for conversion of all the phosphates to the ortho-phosphate form for analysis. Although either direct or indirect atomic absorption spectrometric method[5-10] is a fast method of analysis, the phosphorus concentration in some samples is too low to be detected by this method.

Inductively coupled plasma-atomic emission spectrometry (ICP-AES) method is characterized by a high detection efficiency and good reproducibility, and is an accepted technique for quantitative analysis. This method provides speed, accuracy and reliability for the determination of phosphorus in steel[11], rocks[12], water[6,13,14], waste[15] etc. Wings et.al[13] reported two wavelengths (213.618nm and 214.914nm) for phosphorus determination using ICP-AES method. It has been reported[16] that a partially resolved copper emission line at 214.90nm interferes with the 214.914nm phosphorus line. Other methods using ICP detector[17] and hyphenated ICP techniques[18-21] for phosphorus determination are also reported in the literature.

In this report ICP-AES method for the determination of phosphorus in waste water is presented. Phosphorus in 1-5 mg/L quantity can be determined with ± 1.2% error.

EXPERIMENTAL PROCEDURE

Reagents:

Phosphorus standard	1000 mg/L
Phenolphthalein indicator	
Sodium hydroxide	5.0 N
Sulfuric acid	10.0 N

Samples:
Sample solutions were identified as,

Raw sewage(RS)
Raw industrial waste (RIW)
Final effluent (FE)
Treated industrial waste (TIW)

Equipment:

The instrument used for all the measurements was a Perkin-Elmer ICP/6000 spectrometer equipped with a 7500 computer. The instrument set-up parameters for the phosphorus analysis were as described in Table 1. The analysis was performed with and without purging the ICP system.

Procedure:

Standard phosphorus solution was prepared from a stock solution of phosphorus (Spex Industries) by serial dilution with deionized distilled water. Deionized distilled water was also used as a blank and a rinse after each sample analysis to minimize the memory effect. Use of glassware was avoided as much as possible, if and when used it was rinsed with diluted hydrochloric acid.

Since the amount of phosphorus presented in the sample was very low as determined from the preliminary runs, a 20 mg/L phosphorus standard solution was used for all the analyses. Sample solutions were analysed in the as-received condition. Any solid residue if present in the sample solutions was removed by filtration or it would clog the nebulizer. It was found that the addition of a few ml of acid helped in dissolving the suspended solid. Colloidal solutions were easy to analyse without any interference. 10-15 sets of each sample were analyzed for consistent values. A standard addition method was used to make sure that the amount of phosphorus determined was correct. For comparison purposes samples were also subjected to pretreated as suggested under the Standard Methods for Phosphorus and then analyzed under the similar conditions.

RESULTS

The concentration of phosphorus found in raw and treated sewage water is reported in Table 2. An accurate determination of phosphorus was also made by the standard addition method and data obtained are presented in Table 3. The comparative values for phosphorus present in the sewage water by the direct analysis and the standard addition methods are reported in Table 4. The phosphorus concentration found in the treated and untreated samples was exactly the same.

A typical graphic spectrum is shown in Fig. 1 for 200, 100 and 20 mg/L and in Fig. 2 for 20, 10 and 5 mg/L phosphorus concentrations. A peak left of the standard phosphorus peak is identified as also due to phosphorus (identified as #5 in Figs. 1 & 2). Another peak (identified as #6 in Figs. 1 & 2) was observed to the right of the phosphorus peak and was identified by the Computer Search as due to As, Cu, Fe, Ni, V and Zn. No interferences were observed from any of these elements in the determination of phosphorus by this method.

CONCLUSION

Results obtained from the ICP-AES analysis of sewage water by direct analysis and the standard addition methods prove that this method can be used for routine, fast and accurate analysis of phosphorus. A quantitative value of 1 mg/L phosphorus can be obtained with ± 1.2% error.

Table 1-Instrumental Set-Up Parameters For Phosphorus Analysis

Plasma	Argon, high purity
Plasma height	15 nm
Plasma flow	12 L/min.
Auxillary flow	1 L/min.
Nebulizer	24-25 psi
RF generator	27.12 MHz
Incident power	1250
Reflectance	5 w
Purge	Nitrogen or argon gas
Wavelength	213.618 nm
Range	1.0 nm
Gain	Automatic
Background corr.	Both sides of the peak
Detection limits	0.076 mg/L at 213.618 nm 0.05 mg/L at 214.914 nm

Table 2 - Phosphorus in waste water

Sample name	Concentration of phosphorus found, mg/L	Standard deviation
Raw sewage (RS)	8.34 8.15 8.91	0.28 0.05 0.51
Final effluent (FE)	5.56 5.42 5.56	0.34 0.24 0.23
Raw industrial waste (RIW)	0.52 0.50 0.52 0.53	0.09 0.14 0.13 0.21
Treated industrial waste (TIW)	0.00	0.00

Table 3 - Phosphorus In Waste Water By Standard Addition Method

Sample name	Standard P added, mg/L	Total P found, mg/L	Actual P present mg/L	Standard deviation
RS	1.0	9.809	8.809	0.2
	1.0	9.589	8.589	0.5
	2.0	10.606	8.606	0.5
	2.0	10.525	8.525	0.5
	3.0	11.86	8.53	0.3
FE	2.0	7.53	5.53	0.2
	2.0	7.59	5.59	0.2
	2.25	7.84	5.59	0.4
	2.50	8.05	5.55	0.4
RIW	2.5	3.12	0.62	0.3
	4.0	4.792	0.79	0.2
	4.0	4.786	0.79	0.4
	5.0	5.634	0.63	0.4
	5.0	5.746	0.63	0.3
TIW	2.0	2.0	0.00	0.0
	2.0	1.8	-0.02	0.2
	2.5	2.5	0.00	0.0
	2.5	2.52	+0.02	0.2
	3.33	3.26	0.07	0.1

Table 4 - Comparative Values For Phosphorus

Sample name	Phosphorus in mg/L		Difference ±
	Direct analysis	Standard addition	
RS	8.67	8.61	-0.06
FE	5.53	5.56	+0.03
RIW	0.64	0.69	+0.05
TIW	0.070	0.00	±0.00

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Spectra Name: PHOSPHORUS Wavelength: 213.618 nm Range: 1:00 nm Gain: 538
Background Intervals: —0.045 nm + 0.035 nm Read Delay: 20
Remarks: Standard 200, 100 and 20 mg/L phosphorus solution

1 Standard 200
2 Blank
3 100 mg/L
4 20 mg/L
5
6
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11
12

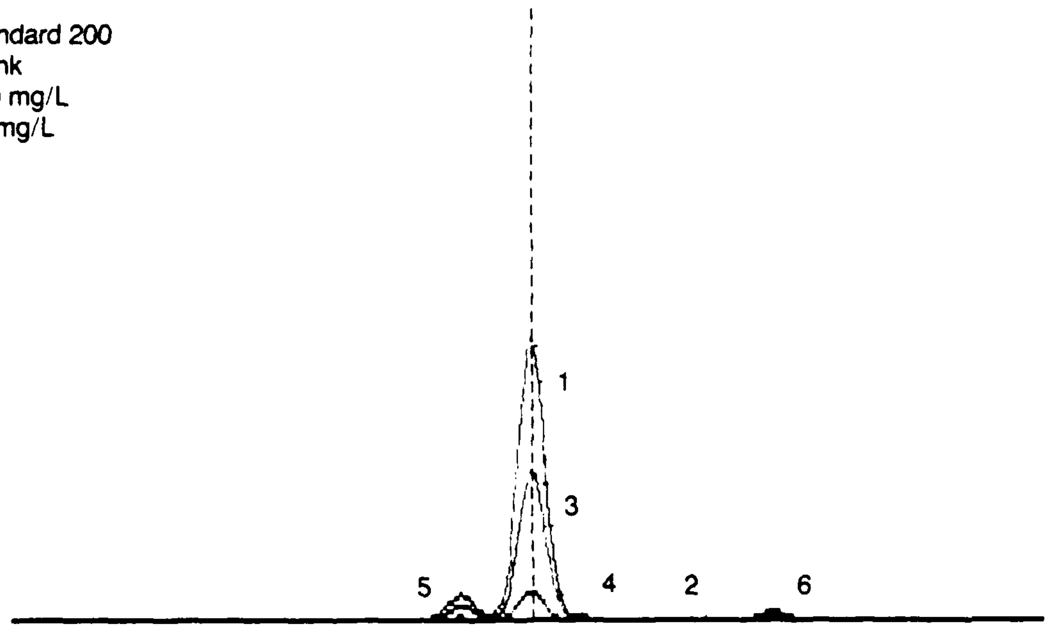

Fig. 1. Graphic Spectrum of Phosphorus, 200, 100 and 20 mg/L

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Spectra Name: PHOSPHORUS Wavelength: 213.618 nm Range: 1:00 nm Gain: 638
Background Intervals: —0.045 nm + 0.066 nm Read Delay: 20
Remarks: Standard phosphorus in 20, 10 and 5 mg/L concentration

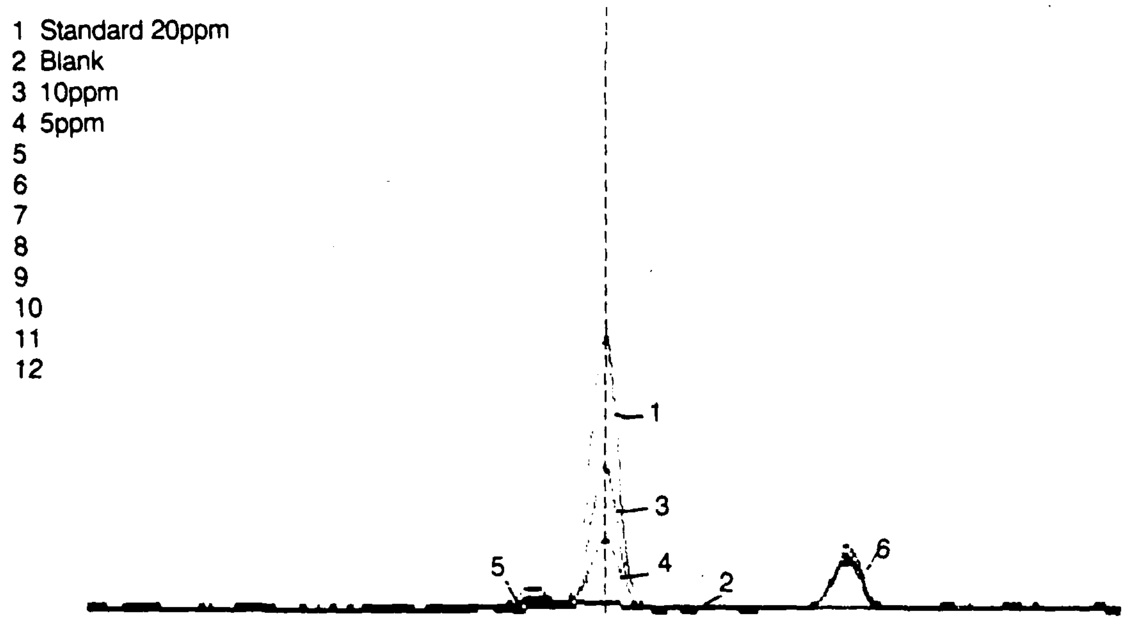

Fig. 2. Graphic Spectrum of Phosphorus, 20, 10 and 5 mg/L

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