

## Short Communication

### Elimination of sodium interference in flameless atomic absorption spectrophotometric determination of lead in potable water by matrix modification

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#### Abstract

A flameless atomic absorption spectrophotometric method which provides a useful, rapid, direct, accurate and precise determination of lead in water and uses matrix modifying reagent consisting of ammonium nitrate and ammonium dihydrogen phosphate is reported. The interference due to sodium salt is minimized by the presence of ammonium ions. Presence of phosphate helps in charring of samples at higher temperatures without the loss of lead. Sample treatment is minimum and pre-concentration is not required which reduces the time of analysis and risk of contamination. The minimum detection limit of the method is  $1 \mu\text{g Pb}$  per litre.

**Key words:** Lead, flameless atomic absorption spectrophotometry, graphite furnace, ammonium nitrate, ammonium dihydrogen phosphate.

#### 1. Introduction

Flameless atomic absorption spectrophotometry provides ultra trace methods for the determination of lead ; however, presence of sodium causes major interferences. The method of standard addition is found to be prone to errors in this technique<sup>1</sup>. Ediger<sup>2</sup> introduced the technique of matrix modification to remove complex matrices during charring stage and advocated the use of ammonium nitrate as matrix modification reagent. Others demonstrated that the addition of ascorbic acid was

beneficial when performing analysis of lead using flameless atomic absorption spectroscopy<sup>3</sup>. It has also been shown that addition of ascorbic acid results in the production of lead atomic vapour by a consistent single mechanism<sup>4</sup>. However, it was observed by us that sodium interferes in lead atomization significantly. Its interference is completely removed by these matrix modifications. A method which utilizes a matrix modifying reagent consisting of mixture of ammonium nitrate and ammonium dihydrogen phosphate is, therefore, developed for lead determination in water.

## 2. Materials and methods

### 2.1. Stock lead solution (1000 mg/l)

Lead nitrate,  $\text{Pb}(\text{NO}_3)_2$  (0.1599 g) was dissolved in about 20 ml of deionised distilled water. Nitric acid (1 ml) was added to it and then the volume was made up to 100 ml with deionised distilled water.

### 2.2. Standard lead solutions

Standard lead solutions of 1, 5, 10, 25 and 50  $\mu\text{g/l}$  concentration were prepared by two step dilutions of stock lead solution with 1% nitric acid prepared in deionised distilled water.

### 2.3. Matrix modifying reagent

Ammonium nitrate (4 g) plus ammonium dihydrogen phosphate (4 g) were dissolved in 20 ml of double distilled water. The volume was then made up to 100 ml with double distilled deionized water.

### 2.4. Equipment

A Perkin-Elmer model-372 atomic absorption spectrophotometer equipped with a Perkin-Elmer model HGA-2200 graphite furnace, a deuterium background corrector and a hollow cathode lamp of lead was used. The lead hollow cathode lamp was operated at 10 mA lamp current with wavelength setting of 283.3 nm and spectral slit width of 0.7 nm. The spectrophotometer was operated in peak height mode. A Perkin-Elmer model 56 recorder on 10 millivolt span was used to record the absorbance signals. Nitrogen was used as purge gas at a flow rate of 50 ml/min. Pyro-coated graphite tubes were used for sample atomization and ML Pipettes with disposable tips were used to inject the sample into the graphite tubes.

### 2.5. Analytical procedure

Water sample for the determination of total lead was prepared by taking 50 ml homogenised water sample in an Erlenmeyer flask. Its pH was adjusted to 2-3 with reagent grade nitric acid and then it was boiled 3-5 minutes vigorously. The content was allowed to cool, filtered through 0.45 micron filter paper and transferred to a



50 ml volumetric flask. The volume was made up with 1% nitric acid. Parallel blanks were prepared by taking deionized distilled water. Standards were also subjected to treatment similar to sample. A 2 ml aliquot was taken in a 5 ml volumetric flask and 2 ml of matrix modifying reagent was added to it. The contents were swirled to mix homogenously and then made up to 5 ml with 1% nitric acid. A 20  $\mu$ l of this matrix modified sample was injected in graphite tube. Graphite furnace was then programmed for drying at 100° C for 30 seconds, charring at 750° C for 30 seconds and atomization at 2300° C for 10 seconds. Background corrector was used throughout the analysis. Results were computed from peak height readings for standards, blank and samples.

### 3. Results and discussion

#### 3.1. Optimum programming of graphite furnace

It is desirable in flameless atomic spectroscopy that matrices present in the sample are completely removed or modified so that only the atoms of analyte are evolved during atomization. In order to achieve this condition an optimum atomization temperature was selected. Plain samples and matrix modified water samples were subjected to analysis at varying charring temperature. It was observed that loss of lead started after charring temperature exceeded 450° C in case of plain water samples. However, no significant loss of lead occurred up to 750° C in matrix modified water samples (Fig. 1). This charring temperature was fixed and atomization temperature was allowed to vary. Results showed that lowest optimum atomization temperature for matrix modified water sample was 2300° C. It was also observed that no spurling of samples occurred during drying stage by fixing drying temperature at 100° C for 30 seconds without ramping.

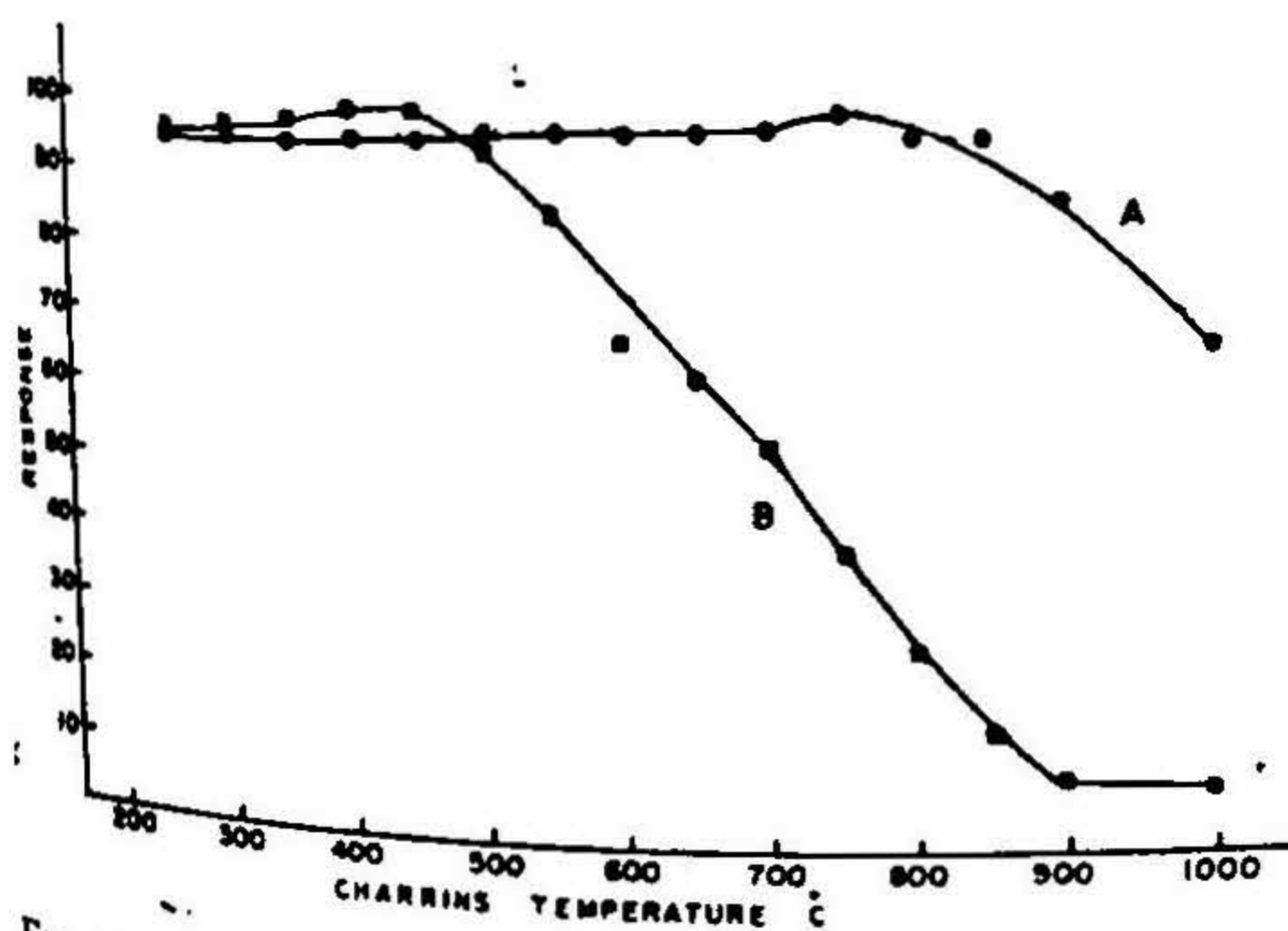


FIG. 1. Effect of matrix modifying reagent on AAS response of 10  $\mu$ g Pb/l. (A) with matrix modifying reagent; (B) without matrix modifying reagent.

### 3.2. Sodium interference

Spiking of lead standard ( $100 \mu\text{g Pb/l}$ ) with varying amounts of sodium showed that sodium suppressed the signal of lead. A suppression of 25.6% was observed at a sodium concentration of 100 ppm. However, when standards spiked with sodium were analysed after matrix modification, almost insignificant interference was observed up to 500 ppm of sodium (Table I). Interference at higher concentration of sodium was also insignificant. Sodium never occurs above 500 ppm in potable water; however, sodium levels may be quite high in sea water. This method may be easily adopted for sea water after optimizing atomization conditions for the matrices present in sea water.

Presence of sodium suppresses the formation of gaseous atom of lead during atomization. However, ammonium nitrate helps in the ashing during charring stage and thus expels sodium at this stage. Formation of lead phosphate enables charring at higher temperature without the loss of lead atoms. Actually, in the presence of phosphate matrix, lead forms lead hydrogen phosphate. This compound on heating decomposes at  $350^\circ\text{C}$  to lead pyrophosphate which is stable up to  $933^\circ\text{C}$ . Formation of lead pyrophosphate is responsible for consistent atomization of lead<sup>5</sup>. The matrix was also found to completely remove the interference due to 500 ppm of potassium, 10 ppm of calcium, 100 ppm of magnesium, 100 ppm of chloride and 500 ppm of sulphate.

Table I

Variation in lead response in the presence of varying amounts of sodium  
(Response for 10 ppb lead standard is taken as 100)

Sodium conc. (ppm)	AAS response without matrix modifying reagent	AAS response with matrix modifying reagent
10	110.9	103.9
20	100.7	101.6
30	95.5	103.5
40	99.3	107.0
50	82.3	101.4
100	74.4	104.5
200	66.9	108.8
500	67.3	114.8



### 3.3. Accuracy, precision, detection limit and sensitivity

Accuracy of the method was found between 90.1–115.6% for lead concentrations varying between 10 and 200  $\mu\text{g Pb/l}$ . Precision which determines the relative scattering of replicate analysis was within 6%. Limit of detection and sensitivity were 1  $\mu\text{g Pb/l}$  and 0.4  $\mu\text{g Pb/l}$  respectively.

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