Abstract: Divalent metal complexes of ligand 2-methoxybenzylidenepyruvate with Fe, Co, Ni, Cu and Zn as well as sodium salt were synthesized and investigated in the solid state. TG curves of these compounds were obtained with masses sample of 1 and 5mg under nitrogen atmosphere. Different heating rates were used to characterize and study these compounds from the kinetic point of view. The activation energy and pre-exponential factor were obtained applying the Wall-Flynn-Ozawa method to the TG curves. The obtained data were evaluated and the values of activation energy (E_a / kJ mol^{-1}) was plotted in function of the conversion degree (α). The results show that due to mass sample, different activation energies were obtained. The results are discussed mainly taking into account the linear dependence between the activation energy and the pre exponential factor, where was verified the effect of kinetic compensation (KCE) and possible linear relations between the dehydrations steps of these compounds.

Keywords: bivalent metal ions, 2-methoxybenzylidenepyruvate, non-isothermal kinetics, dehydration

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MATHEMATICAL EQUATION CORRECTION TO SPECTRAL AND TRANSPORT INTERFERENCES IN HIGH-RESOLUTION CONTINUUM SOURCE FLAME ATOMIC ABSORPTION SPECTROMETRY: DETERMINATION OF LEAD IN PHOSPHORIC ACID

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Abstract: In this work, a new mathematical equation correction approach for overcoming spectral and transport interferences was proposed. The proposal was applied to eliminate spectral interference caused by PO molecules at the 217.0005 nm Pb line, and the transport interference caused by variations in phosphoric acid concentrations. Correction may be necessary at 217.0005 nm to account for the contribution of PO, since $A_{total}^{217.0005\ nm} = A_{Pb}^{217.0005\ nm} + A_{PO}^{217.0005\ nm}$. This may be easily done by measuring other PO wavelengths (e.g. 217.0458 nm) and calculating the relative contribution of PO absorbance (A_{PO}) to the total absorbance (A_{total}) at 217.0005 nm: $A_{Pb}^{217.0005\ nm} = A_{total}^{217.0005\ nm} - A_{PO}^{217.0005\ nm} = A_{total}^{217.0005\ nm} - k\ (A_{PO}^{217.0458\ nm})$. The correction factor *k* is calculated from slopes of calibration curves built up for phosphorous (P) standard solutions measured at 217.0005 and 217.0458 nm, *i.e.* k = (slope$^{217.0005\ nm}$/slope$^{217.0458\ nm}$). For wavelength integrated absorbance of 3 pixels, sample aspiration rate of 5.0 ml min^{-1}, analytical curves in the 0.1 – 1.0 mg L^{-1} Pb range with linearity better than 0.9990 were consistently obtained. Calibration curves for P at 217.0005 and 217.0458 nm with linearity better than 0.998 were obtained. Relative standard deviations (RSD) of measurements (*n* = 12) in the range of 1.4 – 4.3% and 2.0 – 6.0% without and with mathematical equation correction approach were obtained respectively. The limit of detection calculated to analytical line at 217.0005 nm was 10 µg L^{-1} Pb. Recoveries for Pb spikes were in the 97.5 – 100% and 105 – 230% intervals with and without mathematical equation correction approach, respectively.

Keywords: mathematical equation correction approach; high-resolution continuum source flame atomic spectrometry; lead, phosphoric acid

Introduction

Among many factors affecting the determination of lead (Pb) in phosphoric acid by flame atomic absorption spectrometry spectral and transport interferences are considered the more important ones [1], since the analytical line at 217.0005 nm overlaps with PO molecular absorption bands at 216.99 nm in the air-acetylene flame [2], the alternate line at 283.3060 nm is recommended

[3,4]. However both main and secondary lines for Pb are interfered if variations in phosphoric acid concentrations occur, changing the aspiration rate of the nebulizer.

Correction of spectroscopy interferences by mathematical equations in inductively coupled plasma mass spectrometry (ICP-MS) technique has already been established [5,6]. Nevertheless the adaptation of such mathematical equations to atomic absorption spectrometry for spectral interference correction was not feasible due to the lack of a truly simultaneous multi-element system. Recently and upon the introduction of a high-resolution continuum source atomic absorption spectrometry (HR-CS AAS) [7], this problem has been overcome as the whole spectral environment around the analytical line can be acquired [8,9]. Using this technique, the interference caused by PO molecules at the main line for Pb at 217.0005 nm may be eliminated using the least-squares background correction (LSBC) [1]. The alternate line at 283.3060 nm is unaffected by PO, but both main and secondary lines are interfered by transport effects. A method using the LSBC plus internal standardization was recently proposed to minimize these interferences in the determination of Pb in phosphoric acid by HR-CS FAAS [1].

In this paper, a new approach to overcome spectral and transport interferences associated with the determination of Pb in phosphoric acid by HR-CS FAAS has been proposed. In principle, spectral overlap can be corrected providing that another wavelength of the interfering element is itself free from interference. The feasibility of using mathematical equation correction previously employed in ICP-MS, to eliminate spectral interference caused by PO molecules at the 217.0005 nm Pb line and transport interference caused by variations in phosphoric acid concentrations in the determination of Pb in phosphoric acid has been investigated.

Materials and methods

Instrumentation

All measurements were carried out using an Analytik Jena ContrAA 300 high-resolution continuum source flame atomic absorption spectrometer equipped with a xenon short-arc lamp XBO 301 [10] with a nominal power of 300 W operating in a hot-spot mode as a continuum radiation source. This new equipment presents a compact high-resolution double-Echelle grating monochromator correspondent to a spectral band width < 2 pm per pixel in the far ultraviolet range and a charge-coupled device (CCD) array detector.

High-purity (99.7%) acetylene (Air Liquid, Brazil) was used as fuel gas. Air-acetylene oxidizing flame was used for analyte atomization. Solutions were directly aspirated by the burner / nebulizer system of the spectrometer using an injection module (SFS 6) enabling the computer-controlled aspiration of blanks, analytical solutions and samples.

Reagents, analytical solutions and samples

High purity de-ionized water (resistivity 18.2 MΩ cm) obtained using a Millipore Rios 5® reverse osmosis and a Millipore Milli-Q Academic® deionizer system (Bedford, MA, USA), and Merck Suprapur® nitric acid (Darmstadt, Germany) were used throughout to prepare all solutions. All chemical reagents used were of analytical grade.

Reference solutions containing 0.10, 0.20, 0.40, 0.80 and 1.00 mg L^{-1} Pb were daily prepared by appropriate dilution of the 1000 mg L^{-1} single stock standard solutions (Normex ™, Carlo Erba, Italy) and acidified to 1% (v/v) with HNO_3.

Working standard solutions containing 1.0, 2.5, 5.0 and 7.5% (m/v) H_3PO_4 were prepared by dissolving 1.18, 2.48, 5.88 and 8.82 g of analytical reagent grade phosphoric acid 85% (Mallinckrodt, Xalostoc, Mexico) in 1% (v/v) solution. Blank solution was prepared containing only HNO3 in 1% (v/v).

Test solutions containing 100 or 400 µg L^{-1} Pb in presence of 1.0, 5.0 and 7.5% (m/v) H_3PO_4 were used to illustrate the interference effects and the effectiveness of the proposed approach. These solutions were prepared by appropriate dilution of the Pb stock standard solution and concentrated phosphoric acid in 1% (v/v) HNO_3.

Measurement procedure

The measurement procedure is relatively simple. The absorbance for blank [1% (v/v) HNO_3), reference standards [0.10 - 1.00 mg L^{-1} Pb in 1% (v/v) HNO_3] and phosphoric acid sample solutions were measured at the main line for Pb at 217.0005 nm. The absorbance of interfering species (PO) must be measured in the interfering and non-interfering wavelengths: in the present work, absorbances for all phosphoric acid working standard solutions [1.0 - 7.5% (m/v) H_3PO_4] were measured at 217.0005 nm and 217.0458 nm. At 5.0 mL min^{-1} of aspiration flow rate, fuel/oxidizing ratio 0.126, 6 mm of burner height and wavelength-selected absorbance equivalent to 3 pixels, the equipment was adjusted under optimum conditions. All measurements were carried out in triplicates.

Results and discussion

Spectral and transport interferences on Pb: qualitative and quantitative data

Spectral interferences caused by phosphoric acid on Pb determination at the main line 217.0005 nm are due to the PO molecular absorption bands at 216.99 nm formed in the air-acetylene flame. This spectral interference was evaluated by calculating the apparent Pb concentration at the main line for Pb at 217.0005 nm for different phosphoric acid solutions. When the acid concentration was varied from 0% to 7.5 (m/v), the apparent Pb concentration varied from 0 to around 300 µg L^{-1}, as well as the sample flow-rate reduced from 5.0 to 4.0 ml min^{-1}. In earlier work, it was demonstrated that the LSBC technique is an efficient tool to eliminate spectral interference caused by PO on Pb determination, but this correction technique did not avoid the transport effects caused by variations in phosphoric acid concentrations [1].

Determination of the correction factor for mathematical equation

If PO interfering molecule can be measured at a different wavelength free of any interference (and this is feasible in HR-CS FAAS), the correction factor can be calculated from the ratio of slopes of analytical curves for phosphoric acid built up at interfering (217.0005 nm) and non-interfering (217.0458 nm) wavelengths and the absorbance at this last wavelength. The total absorbance (A_{total}) measured at 217.0005 nm corresponds to the contribution of Pb plus PO, so that:

$A_{total}^{217.0005\ nm} = A_{Pb}^{217.0005\ nm} + A_{PO}^{217.0005\ nm}$ Eq. (1)

To obtain the net absorbance for Pb, the PO absorbance must be subtracted from the total absorbance at 217.0005 nm:

$A_{Pb}^{217.0005\ nm} = A_{total}^{217.0005\ nm} - A_{PO}^{217.0005\ nm}$ Eq. (2)

If absorbance of pure phosphoric acid standard solutions at the interfering (217.0005 nm) and non-interfering (217.0458 nm) wavelengths are measured, it is possible to estimate the $A_{PO}^{217.0005\ nm}$ by the application of Beer's Law [11] in both situations as bellow:

$A_{PO}^{217.0005\ nm} = A_{PO}^{217.0458\ nm}\ (slope^{217.0005\ nm}/slope^{217.0458\ nm})$ Eq. (3)

So, the term $(slope^{217.0005\ nm}/slope^{217.0458\ nm}).\ A_{PO}^{217.0458\ nm}$ is the correction factor to be applied in Eq. (2) to obtain only the net absorbance for Pb:

$A_{Pb}^{217.0005\ nm} = A_{total}^{217.0005\ nm} - (slope^{217.0005\ nm} / slope^{217.0458\ nm}).\ A_{PO}^{217.0458\ nm}$ Eq. (4)

For wavelength integrated absorbance equivalent to 3 pixels, aspiration rate of the nebulizer fixed initially at 5.0 ml min^{-1}, analytical curves in the 0.1 – 1.0 mg L^{-1} Pb concentration range was always attained with linearity better than 0.9990. Calibration curves for phosphoric acid (PO) at 217.0005 and 217.0458 nm presented plots within the 1.0 – 7.5% H_3PO_4 with linear coefficient better than 0.9980. Alternate wavelengths were tested for PO at 216.9020 nm, 216.9940 nm and 217.0458 nm, but only the last one furnished the best linear coefficient for calibration curves.

Additionally, it should be stressed that the line for PO at 217.0458 nm is within the spectral window for Pb at 217.0005 nm (Figure 1). The ratio between slopes of curves for PO (slope $^{217.0005\ nm}$/slope $^{217.0458\ nm}$) was calculated daily taking into consideration that changes in the operating parameters alter the measured absorbance. However, ratios obtained in different days were usually within 1.23 – 1.29, but were not significantly different at the 95% confidence level (paired *t-test*).

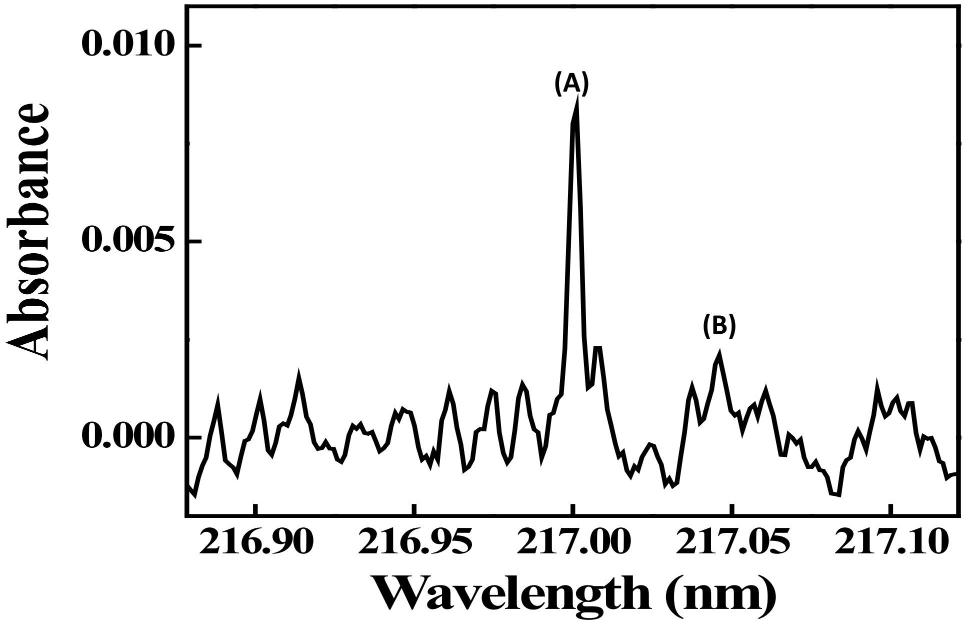

Figure 1. Spectrum recording for spiked Pb in phosphoric acid. (A) and (B) correspond to lines at 217.0005 nm and 217.0458 nm, respectively.

Analysis of test solutions and samples

The feasibility of the mathematical correction for spectral and transport interferences was verified in the determination of Pb in test solutions and two phosphoric acid samples (Table 1). Results for samples were in agreement at a 95% confidence level (paired *t-test*) with those obtained by HR-CS FAAS using LSBC plus internal standardization. Precision and accuracy studies were also carried out using test solutions, or else, recovery experiments for diluted [1% – 7.5% (m/v)] phosphoric acid samples spiked with 100 and 400 µg L^{-1} Pb.

If no correction method is applied, the concentrations of Pb found were in the 140 – 230 µg L^{-1} and 420 – 480 µg L^{-1} intervals for spiked samples with 100 and 400 µg L^{-1} Pb, respectively. These results showed severe spectral interference caused by PO affected measurements at 217.0005 nm causing a positive interference much more pronounced than the interference caused by the transport effect. On the other hand, when the mathematical equation approach was employed, the found concentrations of Pb were close to Pb spikes (recoveries in the 97.5 – 100% range), suggesting the usefulness of the proposal to eliminate spectral and transport interference. Relative standard deviations of measurements varied from 1.4 to 4.3% and 2.0 to 6.0% (n = 12) without and with mathematical equation correction approach, respectively. The limit of detection calculated to analytical line at 217.0005 nm was 10 µg L^{-1} Pb.

Table 1 Comparative results for Pb (n = 3) in commercial phosphoric acid samples and spiked Pb in test solutions (mean ± standard deviation) determined by HR-CS FAAS at atomic line 217.0005 nm with and without mathematical equation approach.

Samples	[Pb], µg L^{-1} Without correction	Proposed technique
Phosphoric acid		
1	94.4 ± 3.5	51.2 ± 2.9
2	91.2 ± 4.2	49.8 ± 3.2
Test solutions		
1.0% (m/v) H_3PO_4 + 100 µg L^{-1}	140 ± 2	100 ± 2
5.0% (m/v) H_3PO_4 + 100 µg L^{-1}	200 ± 7	100 ± 5
7.5% (m/v) H_3PO_4 + 100 µg L^{-1}	230 ± 9	100 ± 6
1.0% (m/v) H_3PO_4 + 400 µg L^{-1}	420 ± 13	400 ± 13
5.0% (m/v) H_3PO_4 + 400 µg L^{-1}	440 ± 19	390 ± 19
7.5% (m/v) H_3PO_4 + 400 µg L^{-1}	480 ± 18	390 ± 19

Conclusions

The accurate determination of Pb in high P content samples such as phosphoric acid by flame AAS requires an efficient method of interference correction. The alternate line for Pb at 283.3060 nm is usually recommended for the analyte determination free of PO interference, but it is interfered by transport effects. With the mathematical equation approach proposed, the main line for Pb at 217.0005 nm was efficiently employed for this analytical task. The mathematical equation correction approach is obviously not limited to the analyte and sample selected in this work.

Additional studies with others elements and samples are necessary to corroborate the general application in flame AAS.

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Resumo: Um método de correção de interferência espectral e de transporte é proposto, e foi aplicado para minimizar interferências por moléculas de PO produzidas em chama ar-acetileno e de transporte causada pela variação da concentração de ácido fosfórico. Átomos de Pb e moléculas de PO absorvem a 217,0005 nm, então $A_{total}^{217,0005\ nm} = A_{Pb}^{217,0005\ nm} + A_{PO}^{217,0005\ nm}$. Monitorando o comprimento de onda alternativo de PO em 217,0458 nm, é possível calcular a contribuição relativa de PO na absorbância total a 217,0005 nm: $A_{Pb}^{217,0005\ nm} = A_{total}^{217,0005\ nm} - A_{PO}^{217,0005\ nm} = A_{total}^{217,0005\ nm} - k\ (A_{PO}^{217,0458\ nm})$. O fator de correção *k* é a razão entre os coeficientes angulares de duas curvas analíticas para P obtidas a 217,0005 e 217,0458 nm ($k = b^{217,0005\ nm}/b^{217,0458\ nm}$). Fixando-se a taxa de aspiração da amostra em 5,0 ml min^{-1}, e integrando-se a absorbância no comprimento de onda a 3 *pixels*, curvas analíticas para Pb (0,1 - 1,0 mg L^{-1}) foram obtidas com coeficientes de correlação típicos $\geq 0,9990$. As correlações lineares entre absorbância e concentração de P nos comprimentos de onda 217,0005 e 217,0458 foram $\geq 0,998$. O limite de detecção de Pb foi 10 µg L^{-1}. O método de correção proposto forneceu desvios padrão relativos (n=12) de 2,0 a 6,0%, ligeiramente menores que os obtidos sem correção (1,4-4,3%). As recuperações de Pb adicionado às amostras de ácido fosfórico variaram de 97,5 a 100% (com correção pelo método proposto) e de 105 a 230% (sem correção).

Palavras-chave: método de correção por equação matemática; espectrometria de absorção atômica com fonte contínua e de alta resolução; chumbo; ácido fosfórico.

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PHOTO-FENTON PROCESS FOR TREATING BIOLOGICAL LABORATORY WASTEWATER CONTAINING FORMALDEHYDE

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Abstract: Laboratories consume great amounts of hazardous chemicals substances and consequently generate wastewater containing them, for example formaldehyde. This substance is widely utilized to preserve biological samples generating many liters of this residue every year. The present work proposes the use of the photo-Fenton process to treat formaldehyde wastewater using sunlight irradiation. Some aspects were investigated such as the iron source, sample and hydrogen peroxide concentration and also the use of stirred systems. The use of ferrioxalate (0.5 mmol L-1) improved the efficiency of the process in relation to the use of iron nitrate, while at least 1.0 mol L-1 H2O2 is necessary to treat the sample of the 500 mg C L-1. Under these conditions, every formaldehyde detectable was degradeted and 89% of the dissolved organic carbon was removed in two hours of exposure to sunlight. These results are satisfaction considerate for São Paulo State Environmental Agency.

Keywords: laboratory wastewater treatment, formaldehyde, photo-Fenton

Introduction

A large number of biological laboratories use solutions of formaldehyde to preserve anatomic pieces, generating many liters of formaldehyde waste every year. In according to the literature formaldehyde is genotoxic in exposed cells in vitro or exposed tissues in vivo [1,2,3]

Although the concentration of formaldehyde is just 3.7% w/w, discharging formaldehyde without treatment can cause serious damage from aquatic environment, due to its carcinogenicity [4] and there is no safe limit to exposition for this class (carcinogenic). Hence, these wastes require an appropriate treatment to avoid risks to the environment and human health.

The adequate treatment of wastes generated by industrial, agricultural or domestic activities is regulated by environmental agencies throughout the world, [5,6] which the levels of specific parameters are limited and checked in the discharged wastes. This is necessary due to large volumes of toxic generated wastes and consequently to the impacts that this cause. The formaldehyde, for example, is able to react with DNA, RNA and proteins, damaging cells and causing the death of microorganisms present in biological wastewater treatment plants [7,8].

Furthermore, the residues generated by Universities and Research Centers offers a special challenge: present specific treatments of different wastes. The ideal solution would be an *in situ* treatment of these wastes, avoiding storage and