

UV-photochemical vapor generation of cobalt in biological samples with inductively coupled plasma optical emission spectrometry.

Honerio Coutinho de Jesus¹ (PQ), Ralph Edward Sturgeon² (PQ).

¹Universidade Federal do Espírito Santo, Departamento de Química, Av. Fernando Ferrari 514, Goiabeiras, Vitória - ES

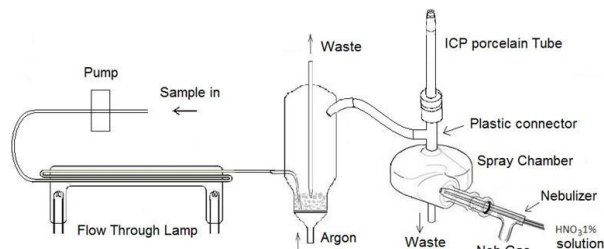
²Institute for National Measurement Standards, National Research Council Canada, 1200 Montreal Rd, Ottawa, ON, CA

Palavras Chave: Photochemical generation, cobalt, ICP-OES.

Introduction

Hydride generation is the most spread technique about Chemical vapor generation (CVG), where aqueous species can be transferred to the gas phase, but in the last decade, photo-CVG (or PVG) has increased due to its simplicity, versatility, cost effectiveness, greener analytical method and less interference¹. New elements such as Co could be determinate using this new technique in either flow through or batch reactor, employing low molecular weight organic acids (meanly formic acid) as photochemical agents on UV irradiation².

In this research, Co was examined using a high power Flow Through Lamp (20W) with 0.75 mL exposed internal volume in series with one fritted glass gas-liquid separator. The vapor phase was directed to a spray chamber installed bellow the torch assembly to allow the profiling of the cobalt line before the analysis (Figure 1). We used Perkin-Elmer Optima 3000 radial view spectrometer. Biological certified reference materials (Tort-2 and 3)



Results and Discussion

In the presence of LMW organic acids, the cobalt volatile species can be generated by UV irradiation of the reaction solution. Solutions of 100 µg L⁻¹ Co were prepared in formic, acetic and propionic acid, but only the Co formic acid solutions generated meaningful signals that increase with the increasing acid concentration (until 60% v/v was available). This result is different from Deng *et al.*² that establish 4% as best concentration. In the other hand there was agreement with the best buffer pH to improve the emission signal. In 50% v/v formic acid this pH was proximate at 3.3 as illustrated in Figure 2.

Besides the effect of the concentration of formic acid and pH of the solution other factors were evaluated. The signal increases with the flow rate of the solution in continuous mode of analysis, mainly due to the

higher mass transfer to the GLS. In the segmented mode the best irradiation time of the solution was 130 s (flow rate = 0.35 mL min⁻¹, similar to Deng *et al.*²) using mass flow rate = 4.3 mL min⁻¹. If the irradiation time is longer, the signal decrease, maybe due to decomposition of Co carbonyl compounds.

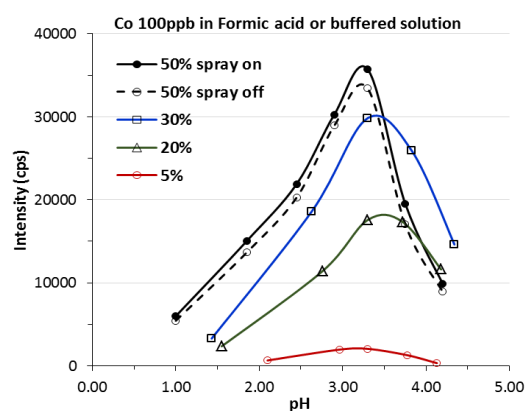


Figure 2. Emission signals to 100 µg L⁻¹ Co formic acid solutions buffered with NaOH. Flow rate in the PVG system = 3.4 mL min⁻¹.

Analytical figures of merit were evaluated under optimal experimental conditions. Good linear coefficient (>0.99), precision (2%) and LOD (<0.4 µg L⁻¹) was obtained for 100 µg L⁻¹ Co solution in 50% formic acid buffered at pH 3.3 using a sample flow rate of 3.4 mL min⁻¹ in continuous mode. The PVG efficiency was evaluated and was approximately 42% (the highest ever been publish to Co). But a poor recovery was obtained to real solutions (Tort-2 and 3) due to strong matrix interferences, such as nitrate, Cu and Fe investigated in this research. When the flow rate was decreased to range 1-2 mL min⁻¹ good accuracy was obtained (>90%), even though the sensitivity has decreased.

Conclusions

This work has demonstrated advantages to use the PVG technique to analyze the real samples, normally in different conditions from that in standard solutions.

Acknowledgements

I acknowledge financial support from CNPq and UFES and the technical support from NRC/Canada.

¹ He, Y.; Hou, X.; Zheng, C.; *Anal. Bional. Chem.* 2007, n° 388, 769.

² Deng, H.; Zheng, C.; Liu, L.; Wu, L.; Hou, X.; Lv, Y.; *Microchem. J.* 2010, 96, 277.