Electrochemical Hydride Generation coupled with Automated Dynamic Headspace Liquid-phase Microextraction: A novel sample preparation method to improve spectrophotometrics ultramicro-analysis of Antimony(III)

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Antimony (Sb(III)) is an emerging element of environmental concern because of its alarming concentration is increasingly observed during the last years. This is due to the current extensive industrial use of antimony. In this study, an automated dynamic headspace liquid phase microextraction technique is coupled with electrochemical hydride generation to determine trace amount of antimony (Sb) by UV–Vis spectrophotometry. This method is subsequently based on electrochemical reduction of Sb to stibine (SbH_3) and reaction of SbH_3 with silver diethyl dithiocarbamate (AgDDC) -as the acceptor phase in headspace- to give a red complex. The electrochemical hydride generator consisted of a cathode cell separated from the anode cell by a porous glass frit and was operated with a constant direct current. A platinum disk was used as the anode and a pre-activated graphite rod as the cathode. A 100 µL syringe assisted with a homemade syringe pump has been used for dynamic micro extraction. Multi factor optimization of process was performed by simultaneous varying of influencing parameters (Current, Electrolysis time, Extraction time, catholyte –HCl- Concentration, Sample Volume) with the aid of response surface methodology (RSM). Under the optimized operating conditions, the detection limit has been measured to be 0.39 µg (39 ng ml^{-1}).The calibration curve was linear in the range of 120–1500 µg $L^{-1.}$ The relative standard deviations at different concentration levels were less than 5.4 %(n=5). To evaluate applicability of the proposed method, Sb(III) was measured in different environmental samples. To sum up, this research shows that dynamic head space is a successful technique for green chemical sampling of electrolytic metal-hydride generated associated with high concentration capability and good replicability. In the other words a selective, efficient, highly sensitive, and ultramicroscale sample preparation and analytical technique is developed to analysis of Sb(III).

Keywords: Automated dynamic headspace microextraction; Design of experiments; Electrochemical Hydride Generation; Sb(III).