22nd Iranian Seminar of Analytical Chemistry
26-28 January 2016
Chemistry & Chemical Engineering Research Center of Iran

Multivariate Data Analyses Intertwined with Electrochemical Hydride Generation Connected µ-Dynamic Extraction to Investigate Simultaneous Spectrophotometric Trace Assessment of As(III) and Sb(III)

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As (III) is one of the most important toxic trace elements, which can cause serious health problems owing to its carcinogenic effects. On the other hand Sb (III) is in-group of non-essential elements and its toxicity is comparable in behavior to arsenic. In the present research, a selective and sensitive method for simultaneous quantification of As (III) and Sb (III) is proposed. An automated dynamic headspace liquid phase micro extraction technique has been used in combination with electrochemical hydride generation to determine As (III) and Sb (III) - in binary mixture- by UV–Vis spectrophotometry. This method is based on electrochemical reduction of As to Arsine (AsH_3) and Sb to Stibine (SbH_3) in acidic media and on the subsequent reaction of AsH_3 and SbH_3 with silver diethyl dithiocarbamate (AgDDC) to give red complexes that their absorption spectra are so closely overlapping. The effects of various parameters affecting the electrochemical hydride generation and extraction efficiency were studied using a central composite design (CCD) under response surface methodology (RSM). Various linear and non-linear multivariate calibration methods, including multiple linear regression(MLR), Inverse classical least squares(ILS), factor analysis regressions (FARs) and optimized artificial neural networks were compared and evaluated for quantification of binary mixtures of As(III) and Sb(III). As a result, this study showed, electrochemical hydride generation connected with µ-Dynamic extraction is a very successful technique for selective and green chemical sampling of trace amount of As(III) and Sb(III). Chemometrics strategies allow to design a few experiments for concurrent optimizing intently and exactly multi-factor and multi-response (recoveries of As and Sb). Utilizing first order calibration methods assisted with signal processing fulfills conveniently simultaneous UV-Vis spectrophotometric determination of As(III) and Sb(III) (with complete and severe overlapping spectra) without any pre-separation, however in routine analysis it is done with the aid of atomic spectroscopy.

Keywords: Antimony; Arsenic; Chemometrics; µ-Dynamic Headspace extraction; Electrochemical Hydride Generation