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Dispersive micro-solid phase extraction combined with dispersive liquid-liquid microextraction for speciation analysis of antimony by electrothermal vaporization inductively coupled plasma mass spectrometry

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ABSTRACT

A method was developed for speciation analysis of antimony by electrothermal vaporization inductively coupled plasma mass spectrometry (ETV-ICP-MS) after preconcentration/separation using dispersive micro-solid phase extraction (DMSPE) and dispersive liquid-liquid micro-extraction (DLLME). In DMSPE, titanium dioxide nanofibers were used for preconcentration and separation of analytes. The upper aqueous phase and elution solution from DMSPE were used for further preconcentration and separation of Sb(III) and Sb(V) by DLLME without any pre-oxidation or pre-reduction operation, respectively. The extracts from DLLME were used for ETV-ICP-MS determination with APDC as a chemical modifier. Under optimal conditions, the detection limits of this method were 0.019 and 0.025 pg mL^{-1} with relative standard deviations of 5.7% and 6.9% for Sb(III) and Sb(V) (c=1.0 ng mL^{-1}, n=9), respectively. This method was applied for speciation analysis of Sb and its distribution in the tea leaves and the tea infusion, including total, suspended, soluble, organic and inorganic Sb as well as Sb(III) and Sb(V). The results showed that the contents of Sb are 62.7, 12.9 and 47.3 ng g^{-1} in the tea leaves, tea residue and tea soup, respectively; those of soluble, organic, inorganic, Sb(III) and Sb(V) are 0.41, 0.11, 0.29, 0.21 and 0.07 ng mL^{-1} in the tea soup, respectively. A certified reference material of tea leaves (GBW 07605) was analyzed by this method with satisfactory results.

Keywords: Antimony speciation; Tea leaves; Dispersive micro-solid phase extraction; Dispersive liquid-liquid microextraction; Electrothermal vaporization inductively coupled plasma mass spectrometry

1. Introduction

Antimony is a cumulative toxic element and may cause a wide variety of adverse health effects [1, 2]. In particular, the physicochemical and toxic properties of Sb strongly depend on its chemical forms. The inorganic species of Sb are more toxic than its organic ones [3]. The toxicity of Sb(III) is higher than that of Sb(V) [4, 5]. Tea has become a beverage widely consumed in vast quantities worldwide (only surpassed by water). Thus, it is necessary not only to determine the total Sb, but also to determine Sb speciation and its distribution in tea

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