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Two-phase liquid-liquid extraction in monosegmented flow analysis. Determination of cadmium with 1-(2′-pyridylazo) naphthol

Ileana Facchin, Celio Pasquini *

Instituto de Química, Universidade Estadual de Campinas, C.P. 6154, CEP 13083-970 Campinas SP, Brazil

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Abstract

A monosegmented flow system has been developed to perform liquid-liquid extraction. Monosegmented samples are introduced in a glass tube between two air bubbles. The volume of organic phase, containing an extracting reagent, is added after the second bubble by switching on a solenoid valve using an optical sensor and a timer circuit. It was observed that the transference of the analyte from the aqueous to the organic phase is achieved by adsorption on the surface of the tube followed by desorption into the organic phase. The system was evaluated for Cd(II) extraction at pH 9.9, employing chloroform-1-(2′-pyridylazo)naphthol as organic phase. The extraction efficiency is 75% and can be improved to be near 100% if the direction of the carrier flow is changed for a number of cycles in the same injection. Spectrophotometric detection was made without phase isolation at the end of the extraction tube. The detection limit (signal-to-blank = 3) for Cd(II) is 85 ng l^{-1} and a linear calibration graph was obtained for concentrations of the metal up to 2.0 mg l^{-1}. Samples can be introduced in the system at 30 h^{-1}. Other metals such as Cu(II) and Zn(II) can be also extracted employing the proposed methodology.

Keywords: Flow system; Liquid-liquid extraction; Cadmium

1. Introduction

Since its first appearance in the literature [1] the monosegmented continuous flow system (MSFS) has been applied to a number of determinations taking advantage of its low sample dispersion, high sample throughput and high residence time [1-5]. Simple equipment, similar to that employed in flow-injection analysis (FIA), and easy operation are achieved by the technique. Most of the procedures described for MSFS are all adaptations of well known spectrophotometric methodologies performed in homogeneous solutions. It was only recently that the MSFS has proved to be useful for determinations requiring the presence of two non-miscible phases in the system [6]. The single-phase liquid-liquid extraction procedure recently reported demonstrates the use of a three-component homogeneous mixture that allows full contact between the analyte and the extracting reagent [6]. Rapid reaction is accomplished and the ternary mixture is thereafter split in two phases. The organic phase retains the analyte in a suitable form for detection.

Although presenting some advantages, the lack of data on single-phase extraction procedures in the

* Corresponding author.

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