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ORIGINAL PAPER

Headspace single-drop microextraction coupled with gas chromatography electron capture detection of butanone derivative for determination of iodine in milk powder and urine

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A new detection method using headspace single-drop microextraction (HS-SDME) coupled to gas chromatography (GC) was established to determine the iodine in milk powder and urine. The derivative from the reaction between iodine and butanone in the acidic media was extracted into a micro-drop then determined by GC-ECD. With the optimisation of HS-SDME and derivatisation, the calibration curve showed good linearity within the range of 0.004–0.1 μg mL^{-1} (0.004–0.1 μg g^{-1}) ($R^2 = 0.9991$), and the limits of detection for milk powder and urine were 0.0018 μg g^{-1} and 0.36 μg L^{-1}, respectively. The mean recoveries of milk powder and urine were 90.0–107 % and 89.4–101 % with mean RSD of 1.7–3.4 % and 2.7–3.3 %, respectively. This detection method affords a number of advantages, such as being simple, rapid, and inexpensive, with low organic solvent consumption, and is remarkably free from interference effects, rendering it an efficient method for the determination of iodine in milk powder and urine samples.

Keywords: headspace single-drop microextraction, gas chromatography-electron capture detection, iodine, butanone, milk powder, urine

Introduction

Iodine plays an important role as an essential life element in humans and animals. The main source of iodine is food, which accounts for more than 80 % of the total intake, followed by water, accounting for approximately 10–20 % of the total intake; in addition, a certain amount of iodine may be ingested from the air. As an important raw material in the synthesis of thyroid hormones, iodine deficiency disorders (IDD) result in stillbirths, abortions, endemic cretinism, and impaired mental functions in children and adults (Hetzel, 1983), especially in developing and less developed countries (de Benoist et al., 2004). In 1998 Delange (1998) showed that almost one billion people are at threat from iodine deficiency disorders, and exposed to endemic goitre, cretinism, and foetal abnormalities, etc. (DeLong et al., 1997). On the other hand, an excess of iodine is also harmful to health, although often neglected. Both iodine deficiency and excess have been associated with the development of goitre. Meanwhile, goitre has been linked to a risk of thyroid cancer, especially in women (Kolonel et al., 1990). Hence, the detection of iodine is imperative.

Several methods have been developed for the determination of iodine, including spectrophotometric methods (Gilfedder et al., 2007; Pena-Pereira et al., 2009), ion chromatography (Bichsel & von Gunten, 1999), use of ICP-MS and GC-MS (Reddy-Noone et al., 2007). These methods possess a number of disadvantages; for example, the homologous pre-treatment technique is problematic and time-consuming, requiring a large number of steps, and the selectivity is low.

Gas chromatography has been used for the determination of iodine based on derivatisation, and the normal derivable reagents include alkylating regents

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