Iodide in urine

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Introduction

A method for the determination of iodide is developed using electrochemical detection (ECD) with a silver working electrode (WE). The method consists of ion exchange chromatography using a phosphate/citrate buffer at pH 6.5, followed by amperometric detection at 0.15 V.
Detection limit of iodide is 0.2-1 µmol/L (25 - 127 ng/mL) depending on the column performance. Reproducibility of the determination of iodide is concentration depended. At 50 µM RSD in peak area's and heights was better than 2%. Below 1 µM the RSD values increased to 10 – 22 %.

Method

The HPLC system consists of a anion exchange column with a phosphate/citrate mobile phase (Table I). The use of halide ions such as chloride and bromide must be avoided as they are reactive towards the silver electrode, causing a high background current and decreased sensitivity.
Di-sodium phosphate and citric acid (both 10 mM) are dissolved in 800 mL water, pH is set to 6.5 with NaOH and water is added to 900 mL. Finally 10% methanol (100 mL) is added.
Determination of analytes in complex matrices such as urine or plasma is often complicated by sample pre-treatment procedures to improve the selectivity of a method. Without suitable pre-treatment co-eluting peaks make reliable and reproducible analysis impossible.
An exception is the determination of iodide in urine. Due to the selectivity of the silver working electrode, urine could be injected directly. After dilution and filtration over a 0.2 µm membrane filter, urine samples have been injected onto the analytical column and analysed.

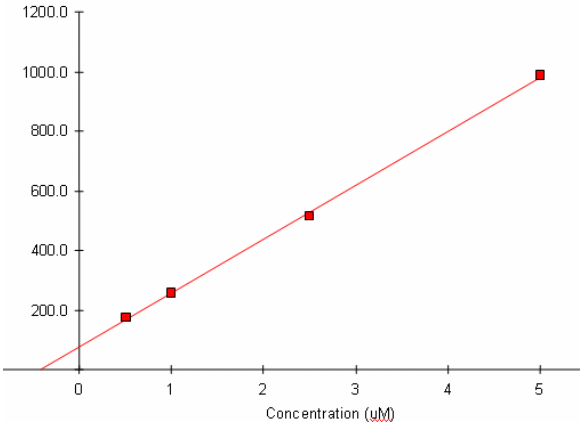

Fig. 1. Regression line of standard addition analysis of iodide in diluted and filtrated urine (see results in Fig. 2). Line: Y = 76.6 + 180.9B, r=0.9996.

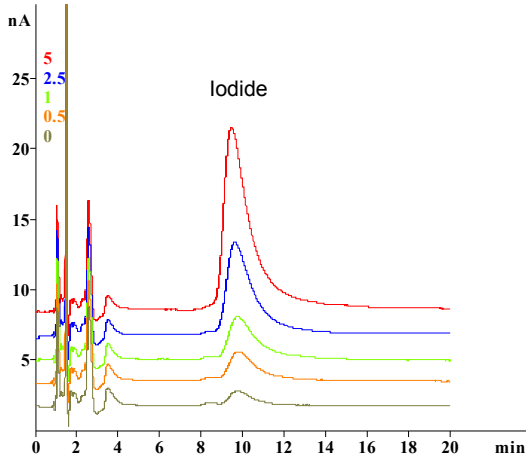

Fig. 2. Standard addition analysis of iodide in diluted (factor 10) and filtrated urine using ECD with a silver working electrode (20 µL). From top to bottom: urine with addition of 5, 2.5, 1, 0.5 and 0 µmol/L potassium iodide. Peak height of iodide (at t = 10 min) corresponds to a concentration of 4 µmol/l in urine.

LC-EC conditions

Column	ALD-510 anion exchange column, 100 x 4.6mm, 7um
Flow rate	1.3 mL/min
Mobile phase	Na_2HPO_4 and citric acid (both 10 mM), pH set to 6.5 with NaOH, 10 % MeOH.
Temperature	35 ºC (separation & detection)
Sample	urine is diluted (10x) and membrane filtrated (0.2 µm filter)
Range	500 nA/V
I-cell	30 - 50 nA
E-cell	150 mV vs HyREF

Part numbers and configuration used

180.0077	ALEXYS 100 Inorganic Ions II