IN-VESSEL HEADSPACE LIQUID PHASE MICROEXTRACTION COUPLED TO SPECTROPHOTOMETRY FOR IODATE DETERMINATION

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HS mode of LPME [Theis 2001] stands out clearly from other LPME methods. This approach allows the analyte to be easily and completely separated from complex matrices. In the HS mode of LPME, phase separation and following transfer to the instrument are greatly simplified. Unlike the other types of LPME, both water-immiscible and water-miscible solvents can be used to extract the analyte from the donor phase. Application of HS-SDME is limited to volatile or semi-volatile analytes. The most common approach in the HS-LPME is to use a microsyringe as a holder of microdrop. During the analysis.

HS-SDME is currently incompatible with most cuvettes and instruments used in spectrophotometry due to the very small volume of the extracting phase. The most common approach is to use micro-volume UV-vis spectrophotometers [Andruch 2012]. In this case, 1–2 μL of the extract is sufficient to measure the absorbance. The disadvantages of cuvetteless spectrophotometry are the short optical path of ~1 mm, fewer solvents, since they must have a suitable viscosity and volatility, and the high cost of the device.

In the present study, a new free space ME technique called intravascular liquid phase microextraction ((IV-HS-LPME)) was developed. The principal feature of this approach is that the acceptor phase is held in a homemade reactor, fixed in a free space above the analyzed solution in a closed vessel. The proposed approach is fully compatible with conventional microcuvettes and instruments used in spectrophotometry. The potential of the method was assessed by determining iodate by converting it to volatile iodine, then absorbing it with 1 % potassium iodide and finally measuring the absorption of the triiodide complex in a microcuvette.

To 15 mL clear or amber glass pharmacy vial add 5,5 mL of aqueous solution containing iodate (as IO_3^-) and 2.5 mL of 1M of sodium sulfate solution, After injecting 2 mL of 25 % sulfuric acid, a 50 μL of KI 1 % is exposed to the headspace of the sample and stirred at 1200 rpm for 25 min. and finally measuring the absorbance of the triiodide complex in a microcuvette. The calibration graph is linear in the range from 8.75 to 175 μg L^{-1} (as IO_3^-) with a detection limit of 4.5 μg L^{-1}. The developed method has a high precision comparable to conventional spectrophotometric methods (0.6–1.5 %). The extraction efficiency is comparable to other previously proposed approaches. the sensitivity and efficiency of the extraction is comparable with other earlier proposed approaches. The simple theoretical model was used to calculate the constants for the equilibria between donor and gas phase and gas and acceptor phase, respectively. For the volumes of acceptor phase less than 100 μL, the experimentally obtained efficiency of extraction was significantly higher than that calculated using the found distribution constants.

In the iodate determination, in comparison with cuvetteless SP the proposed method mode uses bigger volume of acceptor phase and consequently lower preconcentration factor. Nevertheless, this is compensated by greater path length and better reproducibility. The bigger volume and surface area of the acceptor phase also assist in the increase of the sensitivity and efficiency of the determination. It was shown that efficiency of extraction of iodine is not influenced by the matrix components of such analytical samples as mineral water, sea water and table salt.