AUTOMATED IN-SYRINGE MAGNETIC STIRRING ASSISTED LIQUID-LIQUID MICROEXTRACTION OF URANIUM(VI) COUPLED TO LIQUID WAVEGUIDE CAPILLARY CELL SPECTROPHOTOMETRIC

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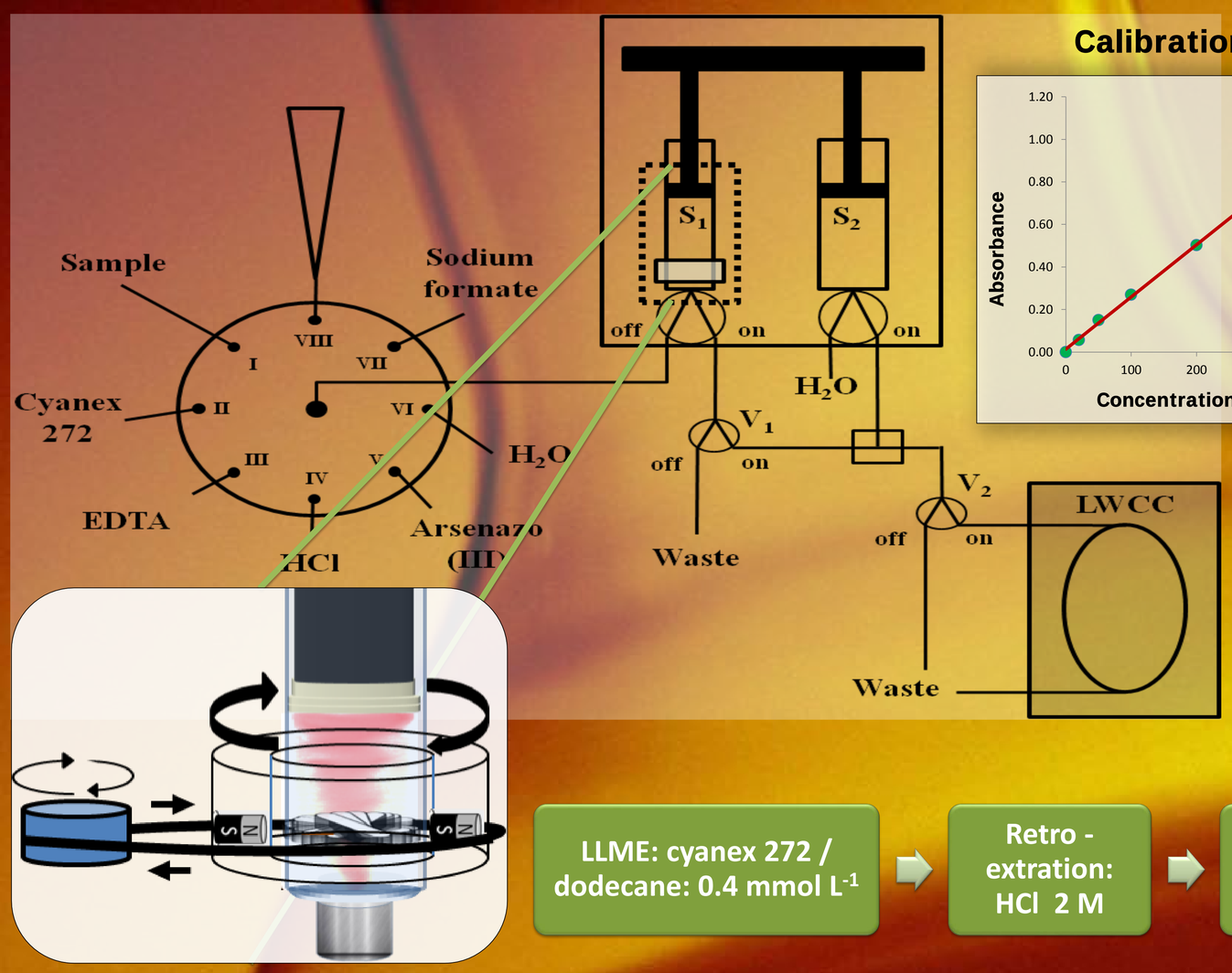
A fully automated in-syringe (IS) magnetic stirring assisted (MSA) liquid-liquid microextraction (LLME) method for uranium(VI) determination was developed, exploiting a long path-length liquid waveguide capillary cell (LWCC) with spectrophotometric detection. On-line extraction of uranium was performed within a glass syringe containing a magnetic stirrer for homogenization of the sample and the successive reagents: cyanex-272 in dodecane as extractant, EDTA as interference eliminator, hydrochloric acid to make the back-extraction of U(VI) and arsenazo-III as chromogenic reagent to accomplish the spectrophotometric detection at 655 nm. Magnetic stirring assistance was performed by a specially designed driving device placed around the syringe body creating a rotating magnetic field in the syringe, and forcing the rotation of the stirring bar located inside the syringe.

The detection limit (LOD) of the developed method is 3.2 µg L^{-1}. Its good interday precision (Relative Standard Deviation, RSD 3.3 %), intraday precision (RSD 3%) and its high extraction frequency (up to 6 h^{-1}) makes of this method an inexpensive, high precision and fast screening tool for monitoring uranium(VI) in environmental samples. It was successfully applied to different environmental matrices: channel sediment certified reference material (BCR-320R), soil and phosphogypsum reference materials, and natural water samples, with recoveries near to 100%.

ANALYTICAL PROCEDURE AND FEATURES



Calibration curve

Analytical features

Detection limit (µg L^{-1})	3.2
Quantification limit (µg L^{-1})	10.7
Intra-day precision (%) (n=10)	3.0
Inter-day precision (%) (n=5)	3.3
Sample volume (mL)	4
Enrichment Factor	8
Sensitivity (UA µg L^{-1})	0.0025
Linear working range (µg L^{-1})	10.7 – 400
Regression coefficient	0.9992
Injection throughput (h^{-1})	6

LLME: cyanex 272 / dodecane: 0.4 mmol L^{-1} → **Retro - extration: HCl 2 M** → **pH = 2: Sodium formate 2 M** → **Colorimetric reaction: arsenazo (III) 0.25 mL, 0.001%** → **Spectrophotometric detection**

Figure 1. Schematic diagram of the LLME-SIA-MSFIA system, procedure diagram and analytical features

CONCLUSIONS

The developed method takes advantage of the automation offered by flow analysis techniques, which combined with in-syringe magnetic stirring assisted exploits the full potential of the liquid-liquid microextraction as extraction technique.

The use of cyanex-272 for uranium extraction and its posterior derivatization with arsenazo-III allowed the satisfactory uranium determination in environmental samples with high variability in uranium(VI) content. Moreover, the implementation of a liquid waveguide capillary cell made possible to achieve the uranium reference values established by several regulatory organizations for drinking water analysis.

Thus, this method has several advantages such as simplicity, selectivity, sensitivity, low operational and instrumentation costs and robustness. Besides, it minimize considerably the use of sample and reagents, contributing to significantly reduce the environmental impact per analysis. The present system has been satisfactorily applied to a wide variety of environmental matrices proving to be a robust, fast and useful screening tool for uranium determination.

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