World Academy of Science, Engineering and Technology
International Journal of Biomedical and Biological Engineering
Vol:10, No:07, 2016

Miniaturizing the Volumetric Titration of Free Nitric Acid in U(vi) Solutions: On the Lookout for a More Sustainable Process Radioanalytical Chemistry through Titration-On-A-Chip

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Abstract : A miniaturized and automated approach for the volumetric titration of free nitric acid in U(VI) solutions is presented. Free acidity measurement refers to the acidity quantification in solutions containing hydrolysable heavy metal ions such as U(VI), U(IV) or Pu(IV) without taking into account the acidity contribution from the hydrolysis of such metal ions. It is, in fact, an operation having an essential role for the control of the nuclear fuel recycling process. The main objective behind the technical optimization of the actual 'beaker' method was to reduce the amount of radioactive substance to be handled by the laboratory personnel, to ease the instrumentation adjustability within a glove-box environment and to allow a high-throughput analysis for conducting more cost-effective operations. The measurement technique is based on the concept of the Taylor-Aris dispersion in order to create inside of a 200 µm x 5cm circular cylindrical micro-channel a linear concentration gradient in less than a second. The proposed analytical methodology relies on the actinide complexation using pH 5.6 sodium oxalate solution and subsequent alkalimetric titration of nitric acid with sodium hydroxide. The titration process is followed with a CCD camera for fluorescence detection; the neutralization boundary can be visualized in a detection range of 500nm-600nm thanks to the addition of a pH sensitive fluorophore. The operating principle of the developed device allows the active generation of linear concentration gradients using a single cylindrical micro channel. This feature simplifies the fabrication and ease of use of the micro device, as it does not need a complex micro channel network or passive mixers to generate the chemical gradient. Moreover, since the linear gradient is determined by the liquid reagents input pressure, its generation can be fully achieved in faster intervals than one second, being a more timely-efficient gradient generation process compared to other source-sink passive diffusion devices. The resulting linear gradient generator device was therefore adapted to perform for the first time, a volumetric titration on a chip where the amount of reagents used is fixed to the total volume of the micro channel, avoiding an important waste generation like in other flow-based titration techniques. The associated analytical method is automated and its linearity has been proven for the free acidity determination of U(VI) samples containing up to 0.5M of actinide ion and nitric acid in a concentration range of 0.5M to 3M. In addition to automation, the developed analytical methodology and technique greatly improves the standard off-line oxalate complexation and alkalimetric titration method by reducing a thousand fold the required sample volume, forty times the nuclear waste per analysis as well as the analysis time by eight-fold. The developed device represents, therefore, a great step towards an easy-to-handle nuclear-related application, which in the short term could be used to improve laboratory safety as much as to reduce the environmental impact of the radioanalytical chain.

Keywords : free acidity, lab-on-a-chip, linear concentration gradient, Taylor-Aris dispersion, volumetric titration
Conference Title : ICMN 2016 : International Conference on Microfluidics and Nanofluidics
Conference Location : Prague, Czechia
Conference Dates : July 07-08, 2016