Preparation for Liquid-Liquid Extraction Studies of Dubnium Using Homologue Models with the SISAK System

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Abstract

Tracers of Nb, Ta, and the pseudo-homologue Pa were used to model the chemical behaviour of dubnium in the development of suitable liquid-liquid extraction systems for the SISAK centrifuge system. Extraction from sulphuric acid solutions into trioctyl amine in toluene and into Aliquat 336 in toluene were investigated as chemical separation systems. Distribution ratios for the homologues and the protactinium tracer, 233Pa, were measured in batch experiments, whereas short-lived cyclotron-produced tracers of Nb and Ta were measured in on-line separations using the SISAK system. SISAK is an automated centrifuge system purpose built to perform fast liquid-liquid extractions of short-lived nuclei. The results establish the optimal conditions for a SISAK dubnium experiment to explore its chemical properties in solutions relating to those of niobium and tantalum.

Addition of H2O2 to the Aliquat 336/H2SO4 system was investigated as part of this work. The addition suppresses the extraction of Nb. And also Ta, but to a lesser extent. This selective hydrogen peroxide complex formation might serve as the basis for an alternative way to investigate the complexing behaviour of Db.

Part of the work presented here is from participation in a SISAK experiment on 257Rf. In this experiment the distribution ratio for extraction into trioctyl amine in toluene from sulphuric acid was determined. The practical experience gained from the Rf experiment was important to ensure that the liquid-liquid extraction system developed here for dubnium is realistic, and furthermore was invaluable in understanding all the necessary requirements and conditions for performing such a complex experiment. The Rf experiments used for the first time a two-step extraction procedure with direct detection of alpha activity in both organic phases. Consequently, the distribution ratio can be determined without knowledge of the amount of activity entering the SISAK system, thereby reducing the uncertainty in the distribution measurement significantly.

Due to the high amounts of liquid consumed by the SISAK system during transactinide experiments, which typically lasts several days, recycling of chemicals is necessary. This results in build-up of KCl in the aqueous phase (from the KCl-aerosol particles used as carriers in the gas jet). Thus, in a separate study the influence of KCl on the distribution ratios for zirconium and hafnium was determined to be negligible for the 5-time recycling currently used for the aqueous phase. It can, however, become a problem if the aqueous phase is more extensively recycled.

List of papers


Paper II. Zheng, L., Alstad, J., Bjørnstad, T., Opel, K., Sabelnikov, A. V., Omtvedt, J. P., SISAK Extraction of Nb and Ta, the Homologues of Db, into Aliquat 336 in Toluene from Sulphuric Acid with or without O22- Ligand, to be published.


Liquid-liquid extraction (LLE) is an introduction to this sample preparation technique. The extraction solvent should not be miscible with the eluent while solubilizing the maximum of solute. This mixture provides the extract (solvent extraction rich in solutes) and the raffinate (eluent depleted in solutes). Multiple steps extraction allows to deplete the eluent in solute in favor of enriching the extract. This technique is a very used step of sample preparation, but it presents multiple drawbacks when practiced with a separatory funnel: Multiplication of extraction steps to obtain optimum output. Use of large volumes of organic solvents, which the costs of recycling are becoming increasingly more expensive. In previous experiments it could be demonstrated that continuous liquid – liquid – extractions on a l-scale are possible with the MicroSISAK device [1]. Recently the IMM developed an improved version of this so called microreactor with two different mixer designs and up to three consecutive separation stages. A scheme of the setup for the experiments is shown in figure 1. in toluene from dilute sulphuric acid. It is reproducible with an accuracy of 5 mbar. For studies on the extraction yield for hafnium – the lighter homologue of rutherfordium (Rf) Z = 104 – we have chosen an extraction system, that has successfully been applied in chemical investigations of Rf with SiSAK [3]: Extraction of hafnium (Hf) with trioctylamine (TOA). Salting-Out Liquid–Liquid Extraction, Multiclass Pesticides, Alcoholic Beverages, High Performance Liquid Chromatography, Ultraviolet-Visible Detector. To cite this article. Tesfa Bedassa, Negussie Megersa, Abera Gure, Salting-out Assisted Liquid-Liquid Extraction for the Determination of Multiresidue Pesticides in Alcoholic Beverages by High Performance Liquid Chromatography, Science Journal of Analytical Chemistry. (2012). Sample-Preparation Methods for Pesticide-Residue Analysis in Cereals and Derivatives. Trends Anal. Chem. (2013). Salting-out Assisted Liquid-Liquid Extraction with the Aid of Experimental Design for Determination of Benzimidazole Fungicides in High Salinity Samples by High-Performance Liquid Chromatography. Talanta 106: 119–126. [37].