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The dissertation of ***Harb Ahmed Hamdy Aly*** on the topic: "**Determination of strontium-90 by its daughter isotope yttrium-90 in alkaline carbonate medium**", submitted for the degree of candidate of chemical sciences in a scientific specialization 1.4.13 Radiochemistry

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The relevance of this work: ^{90}Sr is the most important strontium isotope, which has a relatively long half-life of ($t_{1/2}$ = 28.9 years). Radio-strontium (^{90}Sr) has existed globally since the early beginning of the nuclear weapons tests and locally from authorized (nuclear facilities and nuclear fuel reprocessing industries) or unauthorized releases (nuclear power plant accidents). ^{90}Sr can cause great external radiation doses to humans and other living things. In addition, ^{90}Sr easily enters the food chain and accumulates in the bones and teeth of the human body, causing a chronic internal radiation dose due to its long biological half-life in the human body and the production of the daughter nuclide ^{90}Y through β-decay.

^{90}Y extraction and separation from ^{90}Sr have been studied in acidic media with diversified extractants: dioctyl-phosphoric acid, β-diketone and D_2EHPA were used as extractants to separate ^{90}Y from ^{90}Sr, crown ethers are also extensively studied. The fact that most of the nuclear wastes processing comes from acidic media (mainly nitric acid), but wastes from alkaline media are gaining attention with alkaline processes in nuclear technology (ex. Caustic-Side Solvent Extraction (CSSX) process for Cs removal from HLW and potential wastes of the reprocessing CARBEX process).

This dissertation's scientific novelty was its systematic solvent extraction investigation and separation of Y/Sr from carbonate media, which indicated a synergistic interaction between methyl tri-octyl ammonium carbonate (MTOAK) and several ligands. 2,3DHN and 8HQ possessed a good synergism with MTOAK.

The practical significance of this dissertation concerns with $^{90}Y/^{90}Sr$ separation from alkaline carbonate media by liquid-liquid extraction in order to validate

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of a new and fast radiometric method for ^{90}Sr determination in soil samples by its daughter ^{90}Y. In instead of the conventional methods for determining ^{90}Sr in soil samples, a novel approach was validated. The main advantage of carbonate media is less corrosive and low hazards by-products, avoiding handling difficulties, accompanied hazards of classical acid extraction products.

The dissertation comprises six chapters that contain 41 figures, 22 tables and 118 references. A brief description of these chapters is given in the following sections.

Chapter 1: Introduction

This chapter consists of topic relevance, research purpose and objectives, scientific novelty and practical significance of the work, methodology and method of research, and eventually work approbation and publications.

Chapter 2: Literature review

Literature on the isotopes of strontium and yttrium as well as analytical radio-strontium determination, including analytical strategies, sample preparation, sample pre-treatment, chemical separation, and determination. The advantages and disadvantages of the Sr chemical separation techniques, such as selective precipitation, ion-exchange, extraction chromatography, and liquid-liquid extraction, are also well-explained. This chapter also examines how ^{89}Sr and ^{90}Sr were determined.

Chapter 3: Experimental

The chemical structure of the chelating compounds studied in the dissertation is introduced in this chapter, along with the inorganic reagents used. This chapter also covers the detailed spectrophotometric arsenazoIII procedures for determining yttrium. Experiments on extraction and stripping as well as regeneration studies are described at the end of this chapter.

Chapter 4: yttrium (III) solubility in carbonate media

This chapter contains the results obtained for the solubility and recovery of stable yttrium in alkaline media at different conditions. The author (Harb) found that sodium

carbonate and ammonium carbonate have a considerably higher solubility for yttrium than potassium carbonate at room temperature. Different alkaline salts are used for the roasting process; the roasting temperature was chosen based on their melting points. The eutectic mixtures of $NaOH/Na_2CO_3$ and Na_2CO_3/K_2CO_3 have promising results for yttrium solubility and recovery. The yttrium recovery after roasting was studied in the presence of alumina and silicate; the recovery of yttrium after roasting with the eutectic mixture Na_2CO_3/K_2CO_3 possessed a good yttrium recovery in most conditions. In most conditions, satisfactory recovery percentages for yttrium have been achieved by the author. In addition, the yttrium oxide dissolution rates are much closer; the kinetic orders were $(NH_4)_2CO_3$, K_2CO_3 and Na_2CO_3.

Chapter 5: liquid-liquid extraction of Y(III) and Y/Sr separation

In this chapter, 14 hydroxy organic compounds evaluated as ligands for the extraction of yttrium from carbonate media. It is found that 8-hydroxyquinoline and 2,3-dihydroxyphthalene possessed good D_Y. The D_Y was studied as a function of different parameters: pH, types of diluents, carbonate media, and contact time. The crystal structure and several extracted species were detected using XRD, ATR-IR, and ESI-MS (-). The XRD patterns of Y-2,3DHN showed that the crystal structure appeared like a sandwich-like structure, where Y(III) was located between the layers of 2,3DHN. The binary systems 2,3DHN/MTOAC and 8HQ/MTOAC were used for yttrium separation from carbonate media. The lg $SF_{Y/Sr}$ of 2,3DHN/MTOAC reached 5,5 in BuAc and toluene, and the lg $SF_{Y/Sr}$ of 8HQ/MTOAC was 2.96 and 1.67 in toluene and BuAc, respectively.

Chapter 6: Determination of ^{90}Sr in soil sample by its daughter ^{90}Y

The results presented in this chapter indicate that yttrium was successfully leached and extracted from a sand sample with different leaching mixtures. Then three systems were used to determine ^{90}Sr in the soil. Harb succeeded in accurately determining ^{90}Sr by its daughter ^{90}Y from soil samples after alkaline roasting mixture

1:1:1 (soil:sodium carbonate:potassium carbonate), leaching mixture of 1 mol/L potassium carbonate/0.3 mol/L citric acid, and extraction with 2,3-dihydroxynaphthalene + methyltrioctylammonium carbonate in toluene with an error less than 5%.

Comments and recommendations

1. In the page 29: Why did the author use 40 mg/L for Y and 4 mg/L for Sr?
2. Page 43: The author concluded that the high adsorption percentages obtained for Y(III) (92.2% and 59% using alumina and silica, respectively) decreased the recovery percentage of Y(III). This conclusion does not consistent with the data given in Table 4.8 (Y recovery is increased, not decreased, in presence of alumina or silica). Also, why Y(III) recovery in presence of Silica is lower than that in presence of alumina (tables 4.6 – 4.8) although silica had lower adsorption capability (59%) than alumina (92.2%)?
3. The concentration of 8HQ is ten times higher than that of Y(III). On what basis this ratio was chosen?
4. The D_Y decrease after pH 13 in 2-nitrotoluene might be due to hydroxide precipitation). Why there was no precipitation in case of butyl acetate at pH 13?
5. Page 48, Figure 5.1 shows about 23% of 8HQ transferred into aqueous at pH 13.5, which might explain the D_Y decrease after pH 13). Did the remaining 77% of 8-HQ be insufficient to extract Y(III)?
6. Unlike what obtained in Figure 5.1, why there is no extraction achieved for either Y(III) or Sr(II) using BuAC at the studied pH range? (Figure 5.3).
7. The mineralogical characteristics and composition of the soil used for strontium determination studies should be specified because it has a big impact on the results of the study.
8. There are also a number of misprints and minor inaccuracies, particularly the ionic radius for Y, Ho and Er; the symbol of maximum wavelength; and the Angstromm, which should be corrected to A^{o}, etc.

These remarks do not reduce the overall positive feedback on the work.

Dissertation of ***Harb Ahmed Hamdy Aly*** on the topic: "Determination of strontium-90 by its daughter isotope yttrium-90 in alkaline carbonate medium" *meets* the basic requirements established 19.11.2021 by Order No.11181/1. "On the procedure for awarding academic degrees at St. Petersburg State University". The applicant ***Harb Ahmed Hamdy Aly*** *deserves* to be awarded the academic degree of candidate of chemical sciences in a scientific speciality 1.4.13 Radiochemistry. No violations of paragraphs 9 and 11 of the specified order have been detected.

Mamdoh R. Mahmoud

Mamdoh
30/7/2023

Professor of Radiochemistry

Nuclear Chemistry Department

Atomic Energy Authority, Cairo, Egypt