



## Separation of Sc and Ti using cation exchange resin

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### 1. Introduction

In Brazil, 704 thousand new cancer diagnoses are projected annually during the period of 2023 to 2025 [1], with other studies suggesting a potential 42% increase in cancer cases. This poses challenges for patients, healthcare systems, and society and early diagnosis accompanied by treatment significantly reduces mortality rates [2].

The early diagnosis and treatment can be achieved with the assistance of theranostic radionuclides. Among them, <sup>47</sup>Sc has gained prominence in recent years due to its excellent nuclear properties, including its 3.35 days of half-life,  $\beta^-$  energy of 162 keV, suitable for targeted therapy in small tumors, and  $\gamma$  energy of 159 keV, suitable for imaging. One method of obtaining <sup>47</sup>Sc is through nuclear reactors via the reaction: <sup>47</sup>Ti (n,p) <sup>47</sup>Sc [3].

The chemical behavior of Sc is like that of yttrium and lanthanides. Therefore, its purification can be achieved through chromatographic separations, such as ion exchange or extraction techniques. Resins like AG50W-X8, TRU, DGA, or UTEVA are commonly used for this purpose [4-8].

The objective of this study is to verify the feasibility of separating Ti and Sc using DOWEX 50 W-X8 resin. The separation yield was determined using instrumental neutron activation analysis, INAA.

### 2. Methodology

#### 2.1. Materials

It was used a glass chromatography column with a diameter of 1.75 cm. The resin used was the Dowex 50W-X8 cation exchange resin.

In this study it was used certified solutions of Ti and Sc from Spex CertiPrep, USA. The Ti solution had a concentration of 1000  $\mu\text{g mL}^{-1}$  and the Sc solution had an original concentration of 1000  $\mu\text{g mL}^{-1}$ , where it was diluted to a concentration of 100  $\mu\text{g mL}^{-1}$ .

#### 2.2. Sample preparation

For this procedure, 4 identical samples were prepared as repetitions.

The initial sample, prepared non-radioactively, consisted of aliquots of 300  $\mu\text{L}$  of Sc and 400  $\mu\text{L}$  of Ti from the certified standard solutions, diluted to a volume of 5 mL with purified water.

The final concentration obtained was 80  $\mu\text{g mL}^{-1}$  of Ti and 6  $\mu\text{g mL}^{-1}$  of Sc. These concentrations were determined considering the isotopic abundance of <sup>47</sup>Ti (7,44%), therefore simulating the production of <sup>47</sup>Sc

using natural Ti. To verify these concentrations and calculate the separation yield, a 500  $\mu\text{L}$  aliquot of the sample was pipetted onto a polyethylene capsule and evaporated for subsequent concentration analysis.

Additionally, two diluted standards were prepared using the certified standard solutions. One standard contained 50  $\mu\text{L}$  of the original Ti certified solution, resulting in 50  $\mu\text{g}$  of Ti in the capsule. The other standard contained 10  $\mu\text{L}$  of the diluted Sc certified solution, resulting in 1  $\mu\text{g}$  of Sc in the capsule.

### 2.3. Chromatography column preparation

The chromatography column was prepared with 5 g of Dowex 50W X8 cation exchange resin that was pre-conditioned by sequentially passing 350 mL of distilled water, 200 mL of 2 mol  $\text{L}^{-1}$  HCl, and 50 mL of distilled water through the column.

### 2.4. Chemical separation by ion exchange chromatography

The method used in this article for the separation of Ti from Sc was described by Deilami-Nezhad [9]. The initial solution was passed through the column at a rate of 1 drop every 5 seconds, allowing retention of Ti and Sc ions while other ions passed through. Ti ions were eluted using 50 mL of 2 mol  $\text{L}^{-1}$   $\text{HNO}_3$ , while Sc ions were eluted with 100 mL of a mixture of 4 mol  $\text{L}^{-1}$  HCl and 0.1 mol  $\text{L}^{-1}$  HF, both elutions occurring at the same rate. The eluted solutions were evaporated to approximately 5 mL on a hot plate, then transferred to 5 mL volumetric flasks and diluted with distilled water.

An aliquot of 500  $\mu\text{L}$  from each solution was pipetted onto polyethylene capsules and evaporated for a later concentration analysis.

### 2.5. INAA procedure

The mass fractions of Ti and Sc were determined using the comparative method of activation analysis. This involves irradiating the samples simultaneously with standards of the element of interest and measuring them with the same geometry. Two separate irradiations were conducted: a short-scheme and a long-scheme irradiation.

Using the short-scheme irradiation the concentration of Ti was determined. The capsules containing the samples and standards were enclosed in polyethylene containers and placed inside another polyethylene device for irradiation. The irradiation was conducted using the pneumatic station at the IEA-R1 nuclear research reactor, lasting for 2 minutes under a thermal neutron flux of  $1.9 \times 10^{12} \text{ n cm}^{-2} \text{ s}^{-1}$ .

The long-scheme irradiation was utilized to determine the concentration of Sc in the solution. The capsules containing the samples and standards were wrapped in aluminum foil and placed inside an aluminum cylinder, and irradiated for 8 hours under a thermal neutron flux of  $4.5 \times 10^{12} \text{ n cm}^{-2} \text{ s}^{-1}$  at the IEA-R1 nuclear research reactor.

After irradiation, all capsules containing the samples and standard solutions were individually mounted on stainless steel planchets for subsequent gamma activity measurement. In the case of short-scheme irradiation, samples were measured as soon as possible, due to the short half-life of  $^{51}\text{Ti}$  (5.76 minutes) [10]. In contrast, for the long-scheme irradiation, samples were allowed to decay for a few days to reduce the radiation dosage taken and eliminate potential contaminants with short half-lives. Given that the half-life of  $^{46}\text{Sc}$  is 83.8 days, excessive decay of this radioisotope was not a concern [10].

The gamma activity measurements were done using a Hiperpure Germanium semiconductor detector (HPGe) coupled with a spectrum digital analyzer, both from Canberra, and attached to a microcomputer.

To determine Ti, it was used the transition energy of its radioisotope  $^{51}\text{Ti}$  (320 keV), formed through the nuclear reaction  $^{50}\text{Ti}(n, \gamma)^{51}\text{Ti}$  [10]. As for the Sc, it was determined using the transition energy of its radioisotope  $^{46}\text{Sc}$  (889 keV), formed by the nuclear reaction of  $^{45}\text{Sc}(n, \gamma)^{46}\text{Sc}$  [10].

## 3. Results and Discussion

In Table I, the concentrations of Sc and Ti are presented for the samples at each stage of the separation procedure, determined using INAA and employing the comparative method as described earlier.

Table I: Determination of Sc and Ti in the samples

Sample	Sc		Ti	
	M $\pm$ SD	RSD, %	M $\pm$ SD	RSD, %
Initial, %	78.1 $\pm$ 3.0	3.8	-	-
Ti eluate, %	2.3 $\pm$ 1.7	73.7	85.8 $\pm$ 7.1	8.5
Sc eluate, %	100 $\pm$ 30	30.4	12.4 $\pm$ 4.9	40
Waste, %	-	-	15 $\pm$ 13	86

where M  $\pm$  SD=arithmetic mean and standard deviation; RSD = relative standard deviation. The "-" in the table represents the results that are still in progress, therefore, at the moment of the publishing of this extended summary, the results have not been concluded.

In Table I, the initial solution was found to contain only 78% of the theoretically calculated mass, necessitating adjustments in subsequent concentration calculations. Although most of the Sc was correctly eluted at the Sc eluate, a small amount was also detected at the Ti eluate.

Results from the Ti eluate indicated a minor presence of Sc, with high standard deviation of 73% due to its low quantity. It's noteworthy that the INAA method's detection limit for Sc is in the nanogram range, allowing even trace amounts to be detected, potentially leading to higher error margins.

Regarding the concentration of Sc obtained at the Sc eluate, it can be concluded that a good yield was achieved, albeit with a standard deviation of 30%. This is because the individual concentration values are in the range of 70 %, but a possible outlier, out of 4 samples, decreased the precision value.

In Bokhari [11] work, a literature review is conducted comparing the separation of radioactive Sc from Ti using different resins. One of the mentioned resins was the Dowex 50-X8, where the yield of Sc in the Sc eluate was over 76%.

In Deilami-Nezhad [9], the yield obtained of  $^{47}\text{Sc}$  using the resin Dowex 50W-X8 was over 85% using a mixture of fluoridric and chloridric acid to elute Sc. These results show a separation efficiency over 90% also considering the short dissolution procedure, thus preserving the  $^{47}\text{Sc}$  activity.

In Table I, it is evident that most of the Ti was correctly eluted at the Ti elution. However, there was slight contamination observed at the Sc eluate and in the waste. The contamination in the waste is likely due to the amount of resin used; hence, increasing the resin quantity (mass) may enhance Ti adsorption efficiency. Concerning the Ti contamination at the Sc eluate, a small volume of eluent was used for Ti elution, leaving residues on the resin to be eluted along with the Sc. Therefore, increasing the eluent volume is necessary for complete Ti elution.

#### 4. Conclusions

By the results obtained, it was possible to verify the effectiveness of the separation procedure using DOWEX 50W-X8 resin matches the literature for Ti and Sc separation. The procedure was satisfactory regarding the yields of separation and that this process can be satisfactorily applied for  $^{47}\text{Sc}$  production by the activation of Ti.

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