

Mario Veicht, Ionut Mihalcea*, Đorđe Cvjetinovic and Dorothea Schumann

Radiochemical separation and purification of non-carrier-added silicon-32

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Abstract: ^{32}Si ($T_{1/2} = 153(19)$ y) is an extremely rare, naturally-occurring isotope that has been considered as a geochronometer suitable for radiometric dating over the time span from 100 to 1000 years ago – a time span that has proved rather difficult to explore in this manner. Past attempts to determine the ^{32}Si half-life have resulted in a wide range of values possessing significant uncertainties because only low-activity samples could be made available for such measurements. Utilizing the 590 MeV ring cyclotron at PSI, megabecquerel quantities of ^{32}Si have been produced by exposing metallic vanadium discs to high-energy protons in order to induce spallation. A radiochemical separation procedure has been successfully developed and applied to the irradiated discs as part of the SINCHRON project, based on a combination of ion-exchange and extraction resins. The process was shown to be reliable and robust with a high chemical yield. Radiochemically pure ^{32}Si solutions with activity concentrations of up to several kBq/g can be produced to perform individual measurements (AMS, ICP-MS, LSC) for various studies. Thus, a careful redetermination of the ^{32}Si half-life has become feasible to begin the first steps toward the confident implementation of this radionuclide for geochronological purposes.

Keywords: extraction chromatography; ion exchange chromatography; Si-32 radiochemical separation.

1 Introduction

Radiometric dating has proved to be a versatile tool, which is well-known, for instance, for radiocarbon (^{14}C), potassium-argon ($^{40}\text{K}/^{40}\text{Ar}$), and uranium-lead (e.g., $^{238}\text{U}/^{206}\text{Pb}$) dating. The nuclides are specifically chosen based on their different half-lives ($T_{1/2}$) to account for the timescale over which they can be accurately employed, but also related to their typical accumulation in the environment. As an example, radiometric dating of sediments is considered as an archive that provides extensive information [1]. Typically, ^{210}Pb ($T_{1/2} = 22.20(22)$ y) and ^{14}C ($T_{1/2} = 5700(30)$ y) are used to extract such information. However, because of their individual half-lives, an accurate age determination in the range of 100–1000 years has not yet been established, resulting in a visible dating gap (Figure 1a).

That time span is of particular interest, as sedimentary reconstructions potentially reveal anthropogenic activities during the industrialization period, or generally allow focusing on age horizons associated with human activity ([1] and references therein).

Since, the cosmogenic isotope ^{32}Si ($T_{1/2} = 153(19)$ y [12]) can fill the sediment geochronology gap between ^{210}Pb and ^{14}C , serious limitations are to be found in the high uncertainty as well as in the wide range of published half-life values (Figure 1b), which was highlighted by Fifield and Morgenstern [11], and generally related to the lack of sufficient ^{32}Si sample material.

Already in the mid-1980s, Polak et al. [13] reported on the artificial production of ^{32}Si through proton-irradiation of vanadium. Furthermore, proton-irradiation not only produces ^{32}Si , but also other rare radionuclides, such as ^{26}Al , ^{41}Ca , and $^{44}\text{Ti}/^{44}\text{Sc}$.

Due to the long-term exposure of the V-discs at the PSI facility, extensive amounts of these nuclides were produced, with particular interest focused on ^{32}Si . Here, we report on the artificial production of a significant amount of ^{32}Si , and the subsequent development of a reliable, robust chemical separation procedure to provide radiochemically pure ^{32}Si solutions with high activity concentrations.

Consequently, the SINCHRON project (^{32}Si – a new chronometer) aims to redetermine the half-life of this cosmogenic nuclide. One approach is to determine the decay of the radionuclide sample after a given time interval,

***Corresponding author: Ionut Mihalcea**, Laboratory of Radiochemistry, Paul Scherrer Institut (PSI), Forschungsstrasse 111, CH-5232 Villigen, Switzerland, E-mail: ionut.mihalcea@psi.ch

Mario Veicht, Laboratory of Radiochemistry, Paul Scherrer Institut (PSI), Forschungsstrasse 111, CH-5232 Villigen, Switzerland; and École Polytechnique Fédérale de Lausanne (EPFL), Route Cantonale, 1015 Lausanne, Switzerland

Đorđe Cvjetinovic, Faculty of Physical Chemistry, University of Belgrade, Studentski Trg 12-16, 11158 Belgrade, Serbia

Dorothea Schumann, Laboratory of Radiochemistry, Paul Scherrer Institut (PSI), Forschungsstrasse 111, CH-5232 Villigen, Switzerland

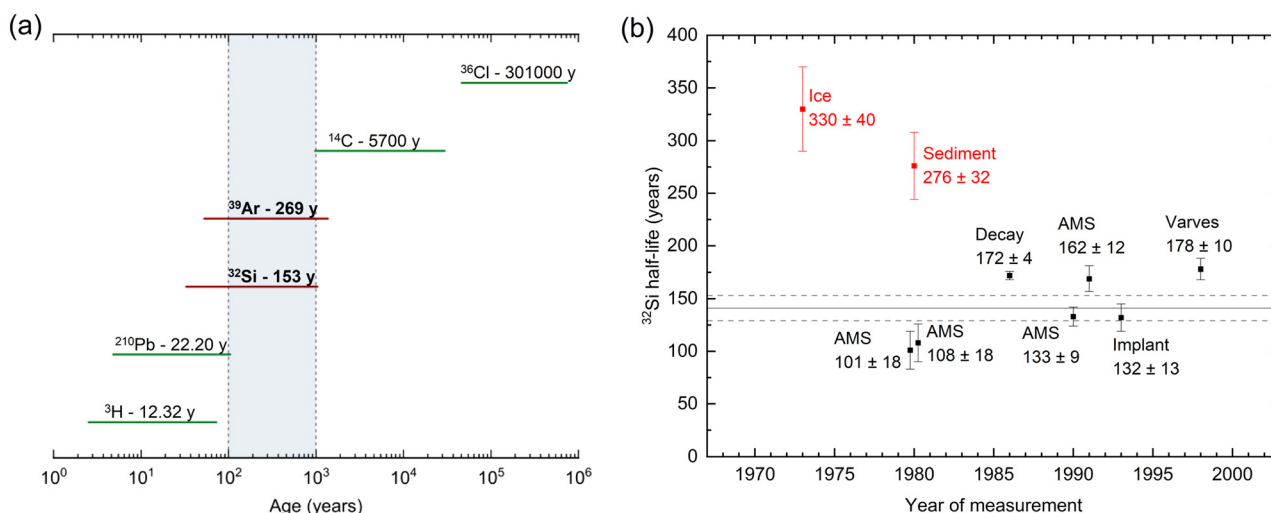


Figure 1: (a) Weakness found in dating capabilities between 100 and 1000 years, and (b) compilation of previous ^{32}Si half-life determinations [2–10], modified after Fifield & Morgenstern [11].

where the decay constant (λ) can be calculated from fitting procedures that fit the data to exponential decays, and thus the $T_{1/2}$ can be derived according to: $T_{1/2} = \ln(2)/\lambda$. For such measurements, the long-term stability of both the sample and the measurement device (e.g., ionization chamber) is vital. Another approach concerns the measurement of the specific nuclide's number of atoms (N) by means of mass spectrometry (ICP-MS, AMS), and additionally, the activity (A) determination, through LSC, for the identical sample. Here, the $T_{1/2}$ can be determined from the relationship: $T_{1/2} = N \ln(2)/A$. Finally, with complementary measurements, the ^{32}Si $T_{1/2}$ can be validated. As a result, our aim in the future will be to provide a new recommended half-life value with a relative standard uncertainty of less than 5%.

2 Experimental studies

Unless otherwise stated, all declared chemical reagents are commercially available and were used without further purification. All required solutions were prepared by dissolution or dilution of commercial products with ultra-pure water (utp- H_2O , 18.2 M Ω cm, Veolia S.A.) and brought to desired concentrations volumetrically. Moreover, any glassware was avoided, and instead, plastic (e.g., polypropylene (PP), polyethylene (PE), polymethyl methacrylate (PMMA)) was used.

2.1 Isotope production

The initial, inactive natural metallic vanadium discs (typical mass \approx 420 mg, diameter \approx 9.20 mm, thickness \approx 1.0 mm

chemical purity of vanadium: 99.8%) were provided by Goodfellow Cambridge Limited (U.K.). The manufacturer's certificate states typical impurities (upper limit in %) to be: Si (<0.2), Nb (<0.1), Mo (<0.1), Ta (<0.1), Al (<0.05). In order to determine the silicon content more precisely, ICP-OES measurements were employed (see Supplementary Material), and a Si content of 200 ppm per V-disc (0.02%) on average was determined. Although working without silicon-carrier, we refer deliberately to non-carrier added (nca) ^{32}Si because of the unavoidable initial presence of Si in the discs and subsequent ^{32}Si sources.

In total, 150 vanadium discs were used as targets in the SINQ (the Swiss Spallation Source) Target Irradiation Program (STIP) [14], using PSI's High-Intensity Proton Accelerator (HIPA), and all simultaneously exposed to high-energy protons ($E_{\text{max}} = 590$ MeV) over two years (2010–2012). The irradiated vanadium discs were then recovered from SINQ, and stored at PSI to allow the short-lived isotopes to decay.

2.2 Radiochemical separation procedure

The separation of nca ^{32}Si from the vanadium matrix required a step-wise approach in order to account for its complexity and to separate other valuable radionuclides. For the radiochemical separations, we chose ion-exchange chromatography in combination with extraction/chelating resins that allowed us to design a highly-selective and robust separation procedure.

Generally, the procedure is composed of three steps: (1) a Loading Step (Load) in which the initial solution is brought

into contact with the stationary phase; (2) a Washing Step (Wash), designed to remove all remaining non-binding elements from the resin, and (3) an Elution Step (Elute), employed to gather the fractions, e.g., for further processing. While the separation procedure was developed to process approximately 150 proton-irradiated vanadium specimens, we also focused firmly on high reproducibility and a generalized purification procedure to account for the different irradiation conditions, leading to a diverse nuclide inventory for each of the vanadium discs. Peristaltic pumps (REGLO Digital MS-2/8, Cole-Parmer Instrument Company, LLC., U.S.A.) were used for the separations, with the same squeeze tubes (Ismaprene PharMed[®], SC0307, Cole-Parmer Instrument Company, LLC., U.S.A.). The method development focused primarily on cold non-active model solutions matching precisely the chemical composition of the active solutions. However, the procedure was ultimately also applied to active solutions in order to study and to confirm the element's behavior in the respective matrix.

2.3 Instrumental analysis

The cold solutions were analyzed using ICP-OES (Agilent 5110, Agilent Technologies, U.S.A.). When measuring samples containing hydrofluoric acid (HF), an HF-resistant setup from the supplier was utilized. Single-element standards (1000 mg/L, TraceCERT[®], Merck KGaA, Germany) in various dilutions were used to prepare the calibration curves for the measurements.

γ -ray spectroscopic studies of the active solutions were undertaken by means of a high-purity germanium (HPGe) detector (Mirion Technologies, California, U.S.A.). A Packard Tri-Carb[®] 2250CA was used for liquid scintillation counting (LSC) to identify pure beta-emitting nuclides (e.g., $^{32}\text{Si}/^{32}\text{P}$).

2.4 Initial preparation for the chemical separation procedure

Before each dissolution, an irradiated vanadium disc was pre-cleaned in 5 mL 8 M HCl to remove a blackish layer that was present on the surface of the disc and contained mostly ^{60}Co , as confirmed by γ -ray spectroscopy. Subsequently, a single disc was placed into a 15 mL PP-tube before adding an acidic cocktail composed of 2.5 mL 8 M HCl and 2.5 mL 8 M HNO_3 for dissolution. During this dissolution process, the PP-tube was placed in an ice-water mixture to quench the exothermic dissolution reaction. This step was necessary, as previous tests with unquenched dissolution

experiments showed a solid residue, indicating the likely formation of an insoluble vanadium compound (e.g., V_2O_5). Thus, the addition of HCl is of further advantage because vanadium(V) forms various chemically stable and soluble chloride complexes [15], which prevents the formation of any solid residues.

In order to diminish the escape of gaseous species (NO_x , $^3\text{H}_2$, HCl(g) , ^{39}Ar , ^{42}Ar) that are created during the dissolution procedure, a cylindrical cap filled with activated carbon was screwed on the PP-tube and replenished after each dissolution procedure. Upon the complete dissolution, a dense, dark-blue-colored solution resulted that was added to 50 mL utp- H_2O . The PP-tube was thoroughly rinsed with 12 mL utp- H_2O . Afterwards, 8 mL 0.5 M N_2H_4 solution was added, and the solution immediately stirred. The addition of hydrazine was necessary to reduce V(V) to the preferred oxidation state of V(IV), which generally shows a higher affinity regarding the cation exchanger [16]. Finally, the volume of the solution was adjusted to 100 mL, and the resulting pH was typically in the range of 1.25 ± 0.05 .

2.5 Separation from the bulk vanadium matrix

Cation exchange chromatography (Dowex[®] 50WX8-200, 200–400 mesh; Sigma-Aldrich, Germany) was initially applied, hosting the resin in tailor-made PMMA columns (length = 300 mm; inner diameter = 10 mm) to a height of 220 mm. Before use, a typical resin-batch was pre-cleaned with 25 mL 6 M HCl, further treated with 50 mL 3 M HNO_3 , and washed with 50 mL utp- H_2O . Consequently, the matrix solution was loaded at a fixed flow-rate of 1.50 mL min^{-1} . The Wash consisted of 20 mL utp- H_2O and was collected together with the initial Load, which was treated as ^{32}Si fraction ($V_{\text{total}} = 120 \text{ mL}$). An Elution stage of 50 mL 3 M HNO_3 followed, and was stored for further radiochemical separations of other products of interest from the initial matrix. Finally, post-conditioning of the resin-batch was undertaken with utp- H_2O until pH reached 4. Each resin batch was used on average six times before replacement with a fresh batch.

2.6 Purification steps to remove trace impurities

After the cation-exchange chromatography, traces of ^{22}Na , $^{42}\text{Ar}/^{42}\text{K}$, $^{44}\text{Ti}/^{44}\text{Sc}$, ^{60}Co , ^{94}Nb , $^{172}\text{Hf}/^{172}\text{Lu}$ and ^{173}Lu were typically noticeable, as confirmed by γ -ray spectroscopy.

Subsequent purification steps were implemented to remove these radionuclides selectively.

2.6.1 LN (extraction) resin: $^{44}\text{Ti}/^{44}\text{Sc}$, ^{173}Lu and $^{172}\text{Hf}/^{172}\text{Lu}$

Extraction chromatography was initially applied by means of LN resin (20–50 μm TrisKem SAS, Bruz, France), which is composed of di(2-ethylhexyl)orthophosphoric acid (HDEHP) impregnated onto an inert support. As only trace impurities had to be removed, a shorter in-house-made PMMA column (length = 100 mm; inner diameter = 10 mm) was chosen and filled with 35 mm resin. Before separation, the resin was pre-conditioned with 20 mL 0.10 M HCl/HNO_3 to account for the composition of silicon solution since the pH was typically measured to be 1.00 ± 0.05 . The ^{32}Si fraction was loaded at a fixed flow-rate of 1.25 mL min^{-1} . Due to the short resin height, the Wash consisted of only 5 mL $\text{utp-H}_2\text{O}$, and was collected together with the Load. Each resin batch was used for six independent purification procedures before being replaced with a fresh one.

2.6.2 Monophos (chelating) resin: ^{94}Nb

Monophos (100–200 mesh; TrisKem SAS, Bruz, France) was used in the next step as the chelating ion-exchange resin. The preparatory steps for the system were identical to the LN purification, except that the PMMA column was changed to a smaller ISOLUTE[®] PE column (length = 60 mm; inner diameter = 9 mm, Biotage Sweden AB, Sweden), and the height of the resin bed was reduced to 30 mm. A 130 mL ^{32}Si fraction was obtained as a consequence of this particular separation procedure.

2.6.3 Evaporation to dryness: ^3H and $^{39,42}\text{Ar}$

Dissolved radioactive gases were removed by evaporating the ^{32}Si fraction to dryness in a PTFE evaporating dish (flat shape, 25 mL) on a hot plate (Fisherbrand[®] Isotemp[®] 4 \times 4, Thermo Fisher Scientific Inc., U.S.A.) with the temperature set to $175 \pm 5^\circ\text{C}$. After reaching complete dryness, 25 mL $\text{utp-H}_2\text{O}$ were added and re-evaporated. The procedure was repeated nine consecutive times to reach a total evaporated volume of 250 mL. Finally, the ^{32}Si recovery was achieved by washing the dish twice with 1 mL 1 M HF, topped up to 10 mL with $\text{utp-H}_2\text{O}$, and eventually transferring into a 50 mL PP-Tube for intermediate storage of the solution.

2.6.4 Dowex[®] 1X8: preparation of the final ^{32}Si solution

Generally, with each processed vanadium disc, a 20 mL 0.1 M HF ^{32}Si solution is obtained. Initially, the LN and Monophos resins removed those ions known to form stable, anionic fluoride complexes. Consequently, similar chemical behavior to Si will hinder further separation as anion exchange is applied. Ideal separation conditions for Si were investigated by employing dedicated batch studies for the expected matrix, including known trace impurities (see Supplementary Material). A similar approach is also described in the literature [17] as providing the basis for the separation from alkali and alkaline-earth metal cations, but also from cobalt and vanadium by using a mixture of HF/HCl. A ISOLUTE[®] PE column (length = 60 mm; inner diameter = 9 mm, Biotage Sweden AB, Sweden) was filled with 30 mm anion exchange resin (Dowex[®] 1X8, 200–400 mesh; Sigma-Aldrich, Germany). Before processing each ^{32}Si solution, the column was washed with 50 mL 0.10 M HF/0.10 M HCl, 50 mL 0.10 M HF/0.13 M HCl, 25 mL 0.10 M HF/0.16 M HCl, and 20 mL 0.50 M HCl. The column was subsequently washed with $\text{utp-H}_2\text{O}$ until pH reached 4, after which 50 mL 0.10 M HF were used to pre-condition the column for the Load. The different initial pre-conditioning steps account for an implemented procedure, which will ensure the removal of any sulfur present in the solution, which is vital for ICP-MS and AMS measurements. 50 mL 0.10 M HF/0.10 M HCl ($m(^{\text{nat}}\text{S}) = 30 \text{ mg}$) were added, followed by 50 mL 0.10 M HF/0.13 M HCl. Sulfur was quantitatively removed from the column in the last step using 25 mL 0.10 M HF/0.16 M HCl, and monitored by ICP-OES measurements. Consequently, the ^{32}Si Elute was obtained within 20 mL 0.50 M HCl.

3 Results and discussion

^{32}Si has been previously produced by various nuclear reactions, such as non-carrier free by double neutron capture $^{30}\text{Si}(\text{n},\gamma)^{31}\text{Si}(\text{n},\gamma)^{32}\text{Si}$ [18] using natural or enriched silicon samples [11], and also with tritons according to $^{30}\text{Si}(\text{t},\text{p})^{32}\text{Si}$ [6, 18, 19]. However, these reactions suffer from a relatively low ^{32}Si production yield, caused by the low natural abundance of ^{30}Si ($=3.092\%$) and low cross-section values for ^{30}Si and ^{31}Si , respectively. Further, phosphorous was studied as a source for ^{32}Si , utilizing the reaction $^{31}\text{P}(\text{n},\gamma)^{32}\text{P}(\text{n},\text{p})^{32}\text{Si}$ [20, 21], as a production route. Proton-induced spallation reactions on heavier elements such as chlorine [22–25], iron [26], sulfur [27], and vanadium [13] have also been

investigated, and the authors have reviewed the production of ^{32}Si . Other products of interest were created during these spallation processes.

As for the metallic vanadium discs used in this work as targets, additional security concerns were necessary, since salts such as $\text{Mg}_2\text{P}_2\text{O}_7$ and KCl are forbidden as targets in SINQ due to their water solubility, so that the use of metal was vital. Although successfully used in previously reported vanadium dissolution experiments [4], H_2SO_4 was avoided in our method due to $^{32}\text{S}/^{32}\text{Si}$ isobar interferences in ICP-MS and AMS measurements.

The first separation step is designed to isolate silicon from the bulk of the matrix materials, while the dilution to 100 mL is needed to adjust the acidity, and thereby enhance the affinity of all cations in solution toward the cation exchange resin. The addition of hydrazine not only reduces V(V) to V(IV) , but as a weak base also increases the pH. When applying cation exchange chromatography under these conditions, most of the matrix components are removed (V , Na , Fe , Co , Sc , and Ti), while Si passes through as Si(OH)_4 . Washing the column with 20 mL $\text{utp-H}_2\text{O}$ ensures that silicon is quantitatively separated. Although using a strong reducing agent, there is still an equilibrium between V(V) and V(IV) species. Therefore, traces of vanadium were usually found in the ^{32}Si solution, and were determined in cold experiments to be in the range of 1–2 μg per Si fraction (see Supplementary Material). Furthermore, the ^{32}Si fraction typically contained several other nuclides, which were identified by means of γ -ray spectroscopy (Figure 2). As a result, extraction resins were explicitly chosen based on their affinity towards these nuclides. LN is an extraction resin designed especially for lanthanide separation [28]. However, this resin exhibits a high affinity towards $^{44}\text{Ti(IV)}/^{44}\text{Sc(III)}$, $^{173}\text{Lu(III)}$, $^{172}\text{Hf(IV)}/^{172}\text{Lu(III)}$, and lower affinity regarding $^{94}\text{Nb(V)}$ while showing no retention of $^{32}\text{Si(IV)}$ (see Supplementary Material), which is present as the hydrolyzed species Si(OH)_4 . A typical spectrum after LN-separation is presented in Figure 2.

The Monophos resin used in the second subsequent purification step is commercially designed for the actinide and polyvalent cations separation [29]. This particular resin possessed a high affinity towards $^{94}\text{Nb(V)}$ (Figure 3), while showing (in a similar manner to the LN resin) no retention regarding Si (see Supplementary Material).

Evaporation to complete dryness was required to remove the volatile β^- -emitting ^3H and $^{39,42}\text{Ar}$ radionuclides. The removal of these radionuclides was monitored by LSC, along with confirming the absence of the short-lived daughter (^{42}K) of ^{42}Ar by searching for the possible

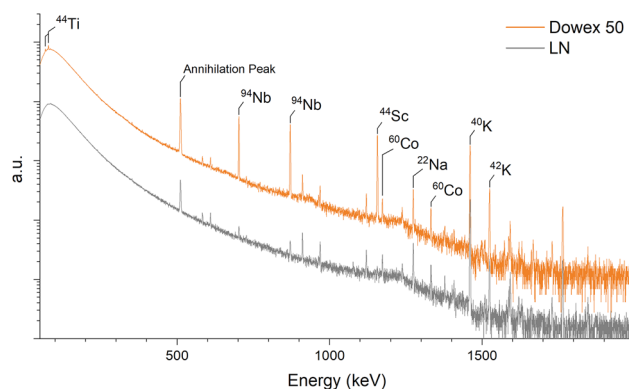


Figure 2: Example γ -ray spectra of a ^{32}Si fraction after processing through Dowex[®] 50 (orange), and the subsequent first purification using the LN resin (gray).

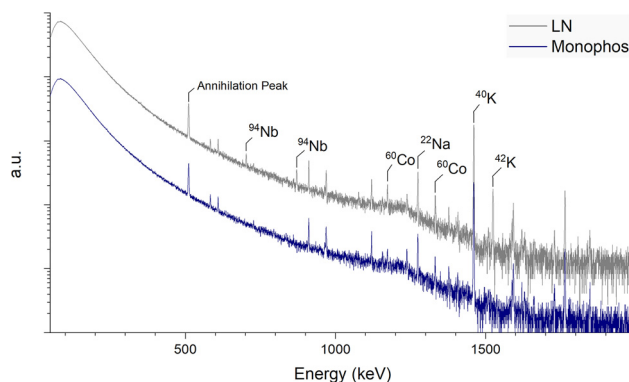


Figure 3: Example γ -ray spectra of a ^{32}Si fraction after LN (gray), and the second stage of purification by means of the Monophos resin (blue).

emission of 1525 keV gamma rays in order to demonstrate the successful removal of ^{42}Ar .

While silicon was present as hydrolyzed species during the separation procedure, the recovery from the evaporation dish using HF changed the silicon speciation into hexafluorosilicate (SiF_6^{2-}), according to Eq. (1):



Batch studies were performed in order to define the optimum conditions to achieve high retention ($\log(K_d) > 10^3$) of SiF_6^{2-} in 0.10 M HF and low retention ($\log(K_d) \approx 1$) when using 0.50 M HCl . Generally, this separation step was designed to ensure the successful occurrence of the following stages: (a). production of ^{32}Si solutions with differing activity concentrations – up to ten ^{32}Si fractions can be processed ($V_{\text{total}} = 200 \text{ mL}$) for subsequent concentration on the anion exchange resin; (b). trace impurities such as

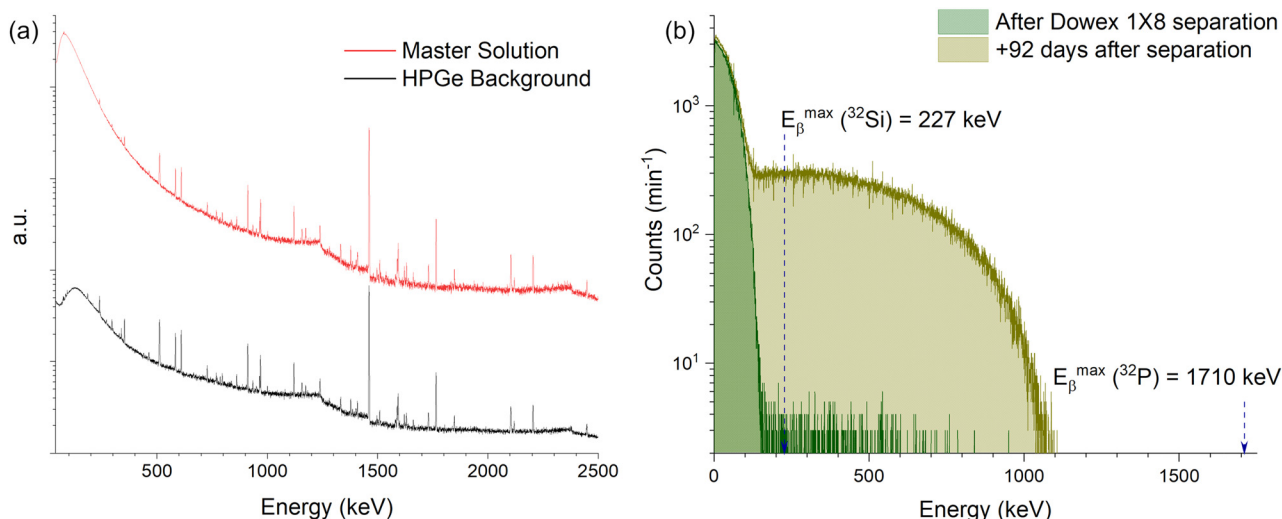


Figure 4: Spectral comparison of (a) 95 h γ -ray measurements, and (b) LSC spectra of ^{32}Si master solution directly after separation, and 92 days later to allow in-growth of ^{32}P daughter.

^{22}Na , V(IV, V) , and ^{60}Co have a low or no affinity towards the resin, and can also be removed. However, as both V(IV) and V(V) tend to form fluoride complexes, care was taken to elute vanadium while not eluting silicon from the column. Moreover, a third step was implemented to remove the sulfur isobar ($m(^{32}\text{S}) = m(^{32}\text{Si})$) that would adversely affected the ICP-MS measurements. Finally, ^{32}Si was quantitatively removed from the column with 20 mL 0.50 M HCl . Subsequent long-term HPGe γ -ray measurements (>90 h) and β -counting by LSC of the final silicon fraction confirmed the absence of any γ -emitting nuclides (Figure 4a) and the presence of $^{32}\text{Si}/^{32}\text{P}$ (Figure 4b), respectively. The complete separation scheme is summarized in Figure 5.

4 Conclusions and outlook

Following the proton-irradiation of the metal vanadium targets, a series of subsequent purification steps were designed and implemented to obtain a radiochemically pure ^{32}Si solution. The results demonstrated that the separation scheme provides a robust and reliable method with a high-yield of ^{32}Si recovery, which will allow the content of up to 150 active vanadium discs to be separated and purified. Consequently, sufficient amounts of ^{32}Si can now be provided to perform a range of individual measurements, focusing initially on a precise determination of the half-life of ^{32}Si . Furthermore, the proton-induced spallation reactions within the vanadium discs are considered to be a

valuable source for other rare carrier-free radionuclides (such as ^{26}Al , ^{41}Ca and $^{44}\text{Ti}/^{44}\text{Sc}$) that can also be recovered from the existing metal matrix.

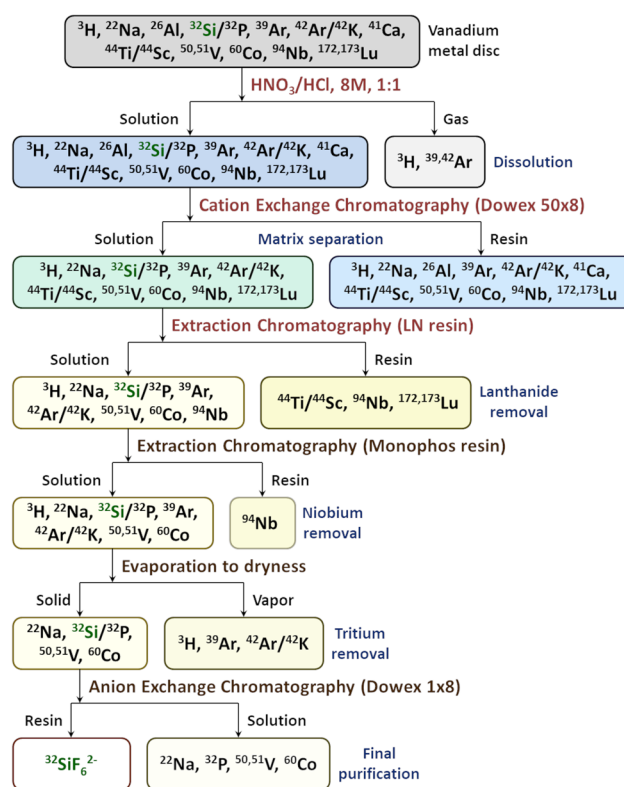


Figure 5: Step-wise separation of nca ^{32}Si from proton-irradiated vanadium matrix.

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