

Isotope Harvesting with Hollow Fiber Supported Liquid Membrane (HFSLM)

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Abstract

The Facility for Rare Isotope Beams (FRIB) will generate many unique isotopes of scientific interest which are retained in the primary beam dump. This work uses Hollow Fiber Supported Liquid Membrane (HFSLM) for extraction of ultra-trace concentrations of short-lived radioisotopes from the large solution volumes present in the primary beam dump loop. Part per trillion levels of ^{48}V were successfully recovered from an aqueous solution spiked with predicted concentrations of chemically similar species, with an extraction efficiency of 71% in 60 minutes.

Key Words

Hollow Fiber Supported Liquid Membrane, HFSLM, Isotope Harvesting, ^{48}V

1. Introduction

The Facility for Rare Isotope Beams (FRIB) is a new national user facility in the United States currently under construction on the campus of Michigan State University (MSU). This facility will produce beams of radionuclides that have previously only existed in supernovae explosions and the crusts of neutron stars. As it creates and delivers beams of some of the most exotic nuclei in the universe, FRIB will simultaneously generate hundreds of other radionuclides that could be synergistically harvested without impact to the primary experiment. Many of these co-produced isotopes are of significant interest for research in biomedicine, energy, environmental studies, plant sciences, and materials science. For example, an experiment utilizing ^{58}Ni as the primary beam will create significant quantities of ^{44}Ti , ^{48}V , ^{48}Cr and ^{52}Fe over the course of a week (Severin et al., 2019). Vanadium-48 would be useful as a radiotracer for nitrogen fixation studies in plants, ^{48}Cr is needed as a precursor for generating isotopically pure ^{48}V for stockpile stewardship studies, and ^{52}Fe would be useful as a positron-emission tomography radiotracer for both medicine and plant sciences.

The FRIB heavy-ion beam begins with a Super Conducting Electron Cyclotron Resonance (SC-ECR) ion source (Machicoane et al., 2016). Ions of the appropriate energy are extracted and accelerated through the 400 kW, 200 MeV/nucleon superconducting-RF driver linear accelerator (Wrede, 2015). In-flight fission and nuclear fragmentation interactions with a light production target generate the primary beam which is directed through the A1900 fragment separator (Morrissey et al., 2003) to select the rare isotope ions of interest to the primary user for experimentation. Co-produced isotopes will be available for harvesting as both gaseous and dissolved species in various locations throughout FRIB, including the primary beam-dump, fragment catcher, and mass slit areas (Figure 1). The primary beam dump, a spinning water-filled Ti-6Al-4V alloy drum and associated water cooling loop, will contain the vast majority of co-produced isotopes and approximately 75% of the unreacted primary beam.

There are several challenges associated with isotope harvesting from the primary beam-dump loop. The loop itself consists of approximately 7000 liters of water circulating at 4.6 L/s through a mixed bed resin (Visser & Scielzo, 2016). The two 50 gal resin beds will only be accessible during major shutdown operations, effectively limiting harvesting operations from the resin beds to long-lived isotopes. The neutral pH environment along with the high flow rates and large volume of water will also limit the technologies available for isotope harvesting. Moreover, chemical separation schemes will have to be developed in order to harvest a specific element from the wide variety of species that will be present at ultra-trace concentrations in the circulating water system. The objective of this work was to investigate the use of Hollow Fiber Supported Liquid Membrane (HFSLM) extraction as a way to harvest co-produced isotopes in-situ from the primary beam-dump loop, unlocking the potential to harvest both short and long-lived isotopes. Without such an in-situ system, short-lived isotopes would be decayed away before any harvesting occurs.

Membrane contactors have been used for over 20 years in large volume systems to control dissolved gases in many applications, including power plants, pharmaceutical production, and food and beverage industries. A large number of studies using these membranes for membrane-based extraction or separation exists in the literature (Castro-Munoz and Fila, 2018; Pabby and Sastre, 2013; Zakrzewska-Trznadel, 2013). There is also considerable interest in using these membranes for treatment of wastewater containing metal ions, inorganic and organic compounds due to the large mass transfer area afforded (Sun et al., 2017). Membrane contactors consist of microporous polypropylene hollow fiber membranes wrapped around a central tube and contained within a contactor housing. During operation, applying vacuum or sweep gas creates a difference in gas concentration between the inside and outside of the membrane, which causes dissolved gas to enter or exit the liquid system. In the case of liquid extraction, the vacuum is replaced with a stripping solution and an organic liquid is embedded in the small pores of the polypropylene membrane and kept there by capillary forces (Kislik, 2010;

Kocherginsky et al., 2007). If the organic liquid is immiscible with the aqueous feed and strip streams, and contains the appropriate extractant, specific elements or groups of elements can be preferentially extracted. Some authors have reported issues with stability of liquid membranes at higher flow rates, i.e., 400 mL/min. but this may also be attributed to the decreased residence time in the module (Leepipatpaiboon et al., 2014; Pirom et al., 2018; Wongkaew et al., 2015).

In this work, we examined Hollow Fiber Supported Liquid Membrane (HFSLM) extraction as a novel method for in-situ recovery of ultra-trace concentrations of metals that will be present in the beam-dump loop. HFSLM extraction has the potential to be seamlessly integrated into the primary beam-dump loop, allowing recovery of rare isotopes while FRIB is in operation. This capability is essential for harvesting shorter-lived isotopes. One example of such a radioisotope is ^{48}V , which is important for stockpile stewardship (FRIB, 2012) and plant microbiome studies (Wichard, 2016; Kraipial et al., 2009). The 15.97 day half-life makes ^{48}V a good model for shorter-lived isotopes, and there are established HFSLM methods for extracting vanadium from aqueous solutions with Aliquat 336 (Palet et al., 1995; Rosell et al., 1997). According to Palet et al. (1995), the vanadium species is highly dependent upon solution pH and the metavanadate species $[\text{VO}_2(\text{OH})_2^-]$ in the neutral feed solution is favored to be transported, while the other oxoanions $[\text{VO}_2^+, \text{VO}(\text{OH})_3, \text{and } \text{VO}_3(\text{OH})^{2-}]$ show an inertness to membrane permeation. There may be a more selective interaction between Aliquat 336 and the monocharged anion than the other vanadium species. In this work, HFSLM was used to separate part per trillion levels of ^{48}V from aqueous solutions spiked with chemically similar species at predicted beam dump production ratios. Reported here are the results of those studies and the observation of the rapid recovery of ultra-trace ^{48}V from simulated aqueous projectile-fragment beam dump loop conditions.

2. Materials and Methods

2.1. General

All materials and reagents were used as received unless otherwise stated. All water used was 18M Ω grade or greater. Titanium foil, 0.127 mm thick, 99.99+% was obtained from Alfa Aesar. Metal free centrifuge tubes were obtained from Labcon. Single element ICP standards were obtained from High Purity Standards. Nitric acid (69%, Optima Grade), 60 mL borosilicate glass test tubes, ammonium nitrate (A.C.S. grade), ammonium hydroxide (Optima grade), sulfuric acid (A.C.S. grade), and hydrogen peroxide (30%, A.C.S. grade) were obtained from ThermoFisher Scientific. Aliquat 336 (MW 442) and dodecane (99%) were obtained from Acros Organics. AG50x8 resin (100-200 mesh) was obtained from Bio-Rad, USA. 1-Dodecanol (\geq 98%, A.C.S. grade) was obtained from Sigma. The Hollow Fiber Supported Liquid Membrane (Membrane Contactor) was a Liqui-Cel 2.5x8 manufactured by 3M.

2.2. Elemental Analysis

Elemental analysis of non-radioactive samples was performed on a PerkinElmer NexION 300X ICP-MS operated in KED (Kinetic Energy Discrimination) mode with a helium flow of 3.5 mL/min. Each isotope was acquired as 5 replicates of 10 sweeps, with 50 ms dwell time per sweep. Internal standards In and Tl were used to normalize data and account for instrument drift. Standards were prepared from dilutions of commercial High Purity Standards multi-element stock solutions.

Elemental analysis of radioactive samples was performed on a Perkin Elmer ICP-OES 2100 using axial plasma view, plasma gas flow of 15 L/min, auxiliary gas flow of 0.2 L/min, nebulizer gas flow of 0.7 L/min, and peristaltic pump rate of 0.40 mL/min. Each sample was acquired as 5 replicates with 7 points per peak, and utilizing Y as an internal standard. Standards were prepared from dilutions of commercial High Purity Standards stock solutions.

2.3. Gamma-Ray Spectroscopy

Radiotracers were evaluated on a 23% relative efficiency HpGe detector (Canberra Model GC2018S), calibrated with a NIST-traceable mixed-gamma standard. Vanadium-48 has significant

gamma emissions at 944, 984, and 1312 keV with abundancies of 7.8, 99.9, and 97.5%, respectively (Lund, 2018). Samples were counted for 10 min. with dead times below 10%, and the software for data acquisition and analysis was Canberra Genie PROcount 2000 V3.3. All samples for analysis were prepared in 20 mL scintillation vials mounted in an acrylic holder a fixed distance from the detector. This geometry was maintained for all measurements.

2.4. Cyclotron production of ^{48}V

The 16.5 MeV GE PETtrace cyclotron at the Missouri University Research Reactor (MURR) was employed to produce ^{48}V by proton irradiation of natural ^{48}Ti foils. The activity of ^{48}V was calculated based upon the following equation: $A = N I x \sigma (1 - e^{-\lambda t})$, where N is the number of target nuclei/cc, I is the intensity of the beam, x is the target thickness in cm, σ is the cross section from the excitation function for ^{48}Ti , λ is the decay constant, and t is the beam exposure time. The cyclotron was operated at 50 μA , and the natural Titanium foil target was a 38 mm-diameter disk with a thickness of 50 microns. Using these values and modeling the target as 10, 5 micron slices, and selecting the cross section in each slice to account for the stopping power of the target given by the NIST PSTAR database, a value of 0.02 mCi of ^{48}V per μA of beam per hour was predicted. For a 2-hour irradiation, this equates to a theoretical value of 2.23 mCi of ^{48}V .

2.5. ^{48}Ti Target Dissolution/Purification

A 38 mm diameter 50-micron ^{48}Ti foil was irradiated at 50 μA for two hours. The 12.7 mm diameter circle irradiated by the beam was removed from the foil with scissors and the vanadium was separated using the procedure found in Zeisler & Ruth (1995). Approximately 60 mg of foil was refluxed with 5 mL of 6 M H_2SO_4 (A.C.S. grade) for 5 hours. The resulting clear purple solution was taken to 70 mL with water and passed through a cation-exchange column (13 cm x 1.5 cm) containing 15 g of AG50-

X8 resin, 100-200 mesh (Bio-Rad, USA) for titanium removal. The ^{48}V was eluted with 3x10 mL fractions of 1% H_2O_2 (A.C.S. grade) in 0.01 M Optima HNO_3 .

2.6. HFSLM conditions

The HFSLM cartridge was a Liqui-Cel 2.5x8 manufactured by 3M (Table 1), installed in a horizontal orientation. The stripping solution travels parallel to the membrane which contains the complexing mixture in its pores. Membrane contactor flow rates were maintained with a Masterflex peristaltic pump 7-200 rpm, equipped with an Easy Load II pumphead. The liquid membrane was prepared by dissolving 0.15 M Aliquat 336 in 3% dodecanol/dodecane and this organic solution was circulated into the lumen side of the HFSLM system (under slight pressure) at 450 mL/min for at least two hours to ensure complete permeation of the membrane pores, while the shell side of the membrane remained open to air. After charging with the organic solution, the excess organic was pumped out of the system and the entire system was washed with deionized water three times for 30 minutes at 450 mL/min. Following rinsing, the feed solution was pumped into the lumen side and the stripping solution was pumped counter-currently into the shell side, operating the unit in recirculation mode. The feed solution was aqueous at neutral pH in a closed system (slightly pressurized at 3 p.s.i.). The strip solution was 0.5 M Ammonia in 0.1 M Ammonium Nitrate at pH 10, open to the atmosphere. Flow rates of both the feed and stripping solutions were kept the same in all experiments at 450 mL/min (6 gph) by flowmeter. Feed and strip reservoirs were stirred throughout the experiment and arranged as illustrated in Figure 2.

3. Results and discussion

3.1. Modeling of Beam Dump contents

The number of atoms present in the beam dump following a 1-week ^{58}Ni beam irradiation (275.3 MeV/u) of a 1.1 g/cm² carbon target at FRIB were calculated using the LISE++ nuclear reaction software

package (v.11.0.19) from Michigan State University (Kuchera et al., 2015; Tarasov & Bazin, 2018). The number of ^{48}V atoms produced through this irradiation was estimated to be $1.92\text{E}17$, end of bombardment. The number of atoms from the ^{58}Ni beam itself was estimated to be over 300 times greater. Other elements generated by irradiation which form similar species as ^{48}V (oxoanions) at neutral pH include: Ti, Cr, and Mn (formed at the same order of magnitude), and B and P (formed at 10 times less than ^{48}V level). These calculations were used to determine the concentrations of elements for use in competition studies (Table 2).

The radiation exposure to the polypropylene fiber is minimal in the proposed application as the HFM is exposed to a small fraction of the total activity in a first pass through the system; the bulk of the activity is trapped in a separate shielded resin bed. In addition, Rathore et al (2001) found no physical damage of the polypropylene hollow fiber upon examination with SEM at radiation levels up to 1 Mrad.

3.2. HFSLM Vanadium Separation

Experiments were performed to determine the feasibility of using HFSLM extraction for removal of vanadium from neutral pH beam dump solution. All experiments were performed at 20°C , although literature (Wongkaew et al., 2015; Pirom et al., 2014) suggests an increased extraction efficiency at temperatures up to 40°C at which point the lifetime of the liquid membrane is shortened. Initial HFSLM experiments were performed with natural vanadium in preparation for radioactive experiments with the following solution concentrations: feed solution of 1000 mL of 0.2 ppm ^{51}V at pH 6, strip solution of 300 mL of 0.5 M Ammonia in 0.1 M Ammonium Nitrate at pH 9, and organic solution of 0.15 M Aliquat 336 in 3% dodecanol/dodecane (Rosell et al., 1997). The feed and strip solutions were pumped at a constant 450 mL/minute. Periodic 1 mL samples were taken from the feed and strip solutions throughout the run and were analyzed by ICP-MS. Results of the natural vanadium experiments show feed vanadium concentration dropping from $0.2\text{ ppm}\pm 0.01\text{ ppm }^{51}\text{V}$ to below detection limit (1 ppb), and strip

vanadium concentration growing from zero to $0.4 \text{ ppm} \pm 0.02 \text{ ppm } ^{51}\text{V}$ in 60 minutes, see Figure 3. There was a higher (2x) final concentration of ^{51}V in the strip solution due to the concentrating effect of the reduced strip volume.

3.3. ^{48}V HFSLM Separation

With the success of the cold vanadium experiment, a radiotracer vanadium extraction experiment was performed. Titanium foil was irradiated at $50 \mu\text{A}$ for two hours and measured $2.55 \text{ mCi } ^{48}\text{V}$ using a Capintec CRC-Ultra at setting 569, which compares favorably to the theoretical value of 2.23 mCi of ^{48}V . The irradiated portion of the foil was then cut out with scissors. The irradiated foil was dissolved by refluxing with 5 mL of $6 \text{ M H}_2\text{SO}_4$ for 5 hours. The clear purple solution was taken to a 70 mL final volume with water. Thirty-five milliliters of the target solution was taken to 500 mL with water and pH adjusted to 6.8 for the feed solution. The conditions of the radiotracer vanadium experiment were the same as the cold experiment, except the feed and strip volumes remained equal at 500 mL . Initial and final 1 mL samples were taken from the feed solution, and periodic 1 mL samples were taken from the strip solution throughout the run. All samples were analyzed for ^{48}V by HPGe gamma spectroscopy. This experiment resulted in only 1% extraction of the ^{48}V into the strip solution, which was unexpected as we observed complete extraction of vanadium in the natural vanadium experiments. We hypothesized that the poor extraction performance of the radiotracer resulted from the introduction of competing titanium ions. Analysis of titanium in the feed solution showed 435 ppm from digestion of the Ti target.

3.4. AG50-X8 Resin for Titanium Removal

The initial pre-column sample of dissolved target had activities of $2562 \mu\text{Ci } ^{48}\text{V}$, $46 \mu\text{Ci } ^{47}\text{Sc}$, and $33 \mu\text{Ci } ^{44\text{m}}\text{Sc}$. One third of the sample was retained, and the remaining two thirds was passed through the AG50-X8 cation resin column. The retained sample showed activities of $734 \mu\text{Ci } ^{48}\text{V}$, $19 \mu\text{Ci } ^{47}\text{Sc}$, and

13 $\mu\text{Ci } ^{44\text{m}}\text{Sc}$, while the post-column sample showed 1626 $\mu\text{Ci } ^{48}\text{V}$ (92% recovery) and no detectable Sc activity.

3.5. ^{48}V HFSLM Separation after Titanium Removal

In a second vanadium radiotracer experiment, the same conditions were used as the initial experiment except that the ^{48}V tracer was separated from the titanium target material with AG50-X8 resin prior to incorporation into the feed solution. Feed and strip solution volumes remained equal at 500 mL. Initial and final 1 mL samples were taken from the feed solution, and periodic 1 mL samples were taken from the strip solution throughout the run. All samples were analyzed for ^{48}V by HPGe gamma spectroscopy. The extraction results are illustrated below (Figure 4), with activities converted to concentration units. The error bars on this experiment ($n=1$) are based upon the uncertainty in the counting statistics using the Canberra Genie software. Feed vanadium concentration went from 18.5 ppt ^{48}V to 1.6 ppt ^{48}V in 180 minutes. The concentration in the strip solution grew from zero to 13.2 ppt ^{48}V in 180 minutes, a 71% extraction. Removal of titanium with AG50x8 resin confirmed our hypothesis of titanium ion interference as illustrated by the increased extraction of ^{48}V to 71%. It should be noted that the high concentrations of titanium encountered in the first radiotracer (> 400 ppm) experiment will not exist in the beam dump loop at FRIB. The highest concentration of ions in the beam dump will come from the heavy ion beam, and the ^{58}Ni concentration for a 1-week irradiation is only estimated to be 900 ppt.

3.6. ^{48}V HFSLM Separation with Competitive Species

With the success of the radiotracer vanadium HFSLM extractions, further ^{48}V extraction experiments were performed to determine the impact of competitive chemical species that would be present in the FRIB beam dump following the ^{58}Ni irradiation of a carbon target. The testing was performed with the same conditions as previous experiments with the addition of: 10 ppt of Mn, Cr,

and Ti; 100 ppt of B, and P; and 10 ppb of Ni to the feed solution as expected to exist in the beam dump (Table 2). These concentrations were chosen based upon the ratio of predicted nuclides in the FRIB beam dump loop with a ^{58}Ni beam. Initial and final 1 mL samples were taken from the feed solution, and periodic 1 mL samples were taken from the strip solutions throughout the run. All samples were analyzed for ^{48}V by HPGe gamma spectroscopy. The results are illustrated below (Figure 5), with activities converted to concentration units. The error bars on this experiment ($n=1$) are based upon the uncertainty in the counting statistics using the Canberra Genie software. Feed vanadium concentration went from 8 ppt ^{48}V to 0.5 ppt ^{48}V at the end of the experiment. The strip vanadium activity grew from zero to 5.7 ppt ^{48}V in 60 minutes, a 71% extraction. The results demonstrate the successful (71%) extraction of 8 part per trillion ^{48}V in the presence of competitive species within the first 60 minutes of operation.

4. Conclusions

To the best of our knowledge, this is the first ever demonstrated HFSLM extraction of such low concentrations of metals, which are the theoretical abundances expected in the FRIB beam dump loop. In addition, the rapid (60 min) extraction unlocks the door to harvesting many short-lived isotopes that would be decayed away before any batch harvesting method could be implemented. The extraction method remained effective in the presence of Mn, Cr, Ti, B, P, and Ni ions at the expected concentrations for the application. Lastly, these short-lived isotopes could be concentrated through the use HFSLM. For example, the HFSLM module could be plumbed to use the 7000 L beam dump loop as the inlet for the feed, and a 1 L loop inserted for the strip solution. This would introduce a concentration factor of 7000 for the isotope of interest.

The success of HFSLM extraction gives the researcher another tool in the quest for As Low As Reasonably Achievable (ALARA) radioactive exposure. By plumbing the membrane contactor in a

location away from the 50-gallon resin tanks, researchers only need to account for the radiation field generated by the harvested isotope rather than the radiation from the entire range of beam fragments.

While this proof-of-principle experiment demonstrates the effective extraction of part per trillion level radioisotopes from an aqueous projectile-fragment beam dump, further research should focus upon radioisotopes that cannot be produced easily by other means. For example, the ^{48}V used for this study was produced on a small medical cyclotron, which could not easily produce isotopes such as ^{48}Cr , or ^{56}Ni . Due to the rapid extraction, it is likely that short-lived research isotopes will be the most promising targets for HFSLM extraction from projectile fragmentation facilities.

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Additional Information

Declarations of interest: None.

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Vitae

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Figure Captions

Figure 1 Schematic of harvesting locations in the FRIB beam line. Attributed to MSU/FRIB.

Figure 2 Counter-current flow diagram for recycle mode operation of HFSLM: 1)feed reservoir (pressurized) 2)peristaltic pumps 3)flow meter 4)pressure gauge 5)hollow fiber module 6)stripping reservoir

Figure 3 Trace (ppm) ^{51}V extracted from feed vs time. Feed pH 6, Strip 0.5 M Ammonia in 0.1 M Ammonium Nitrate pH 9, Organic 0.15 M Aliquat 336 in 3% dodecanol/dodecane, flowrate 450 mL/min. (n=1)

Figure 4 Ultra-trace (ppt) ^{48}V extracted from feed vs time. Feed pH 6.88, Strip 0.5 M Ammonia in 0.1 M Ammonium Nitrate pH 10, Organic 0.15 M Aliquat 336 in 3% dodecanol/dodecane, flowrate 450 mL/min (n=1, titanium removed)

Figure 5 Ultra-trace (ppt) ^{48}V extracted from feed vs time. Feed pH 6.88, Strip 0.5 M Ammonia in 0.1 M Ammonium Nitrate pH 10, Organic 0.15 M Aliquat 336 in 3% dodecanol/dodecane, flowrate 450 mL/min. 8 ppt ^{48}V , 10 ppt Mn, Cr, Ti; 100 ppt B, P; 10 ppb Ni (n=1)

Table 1 Hollow Fiber Supported Liquid Membrane Specifications

Table 2 Estimated beam dump concentrations and experimental concentrations

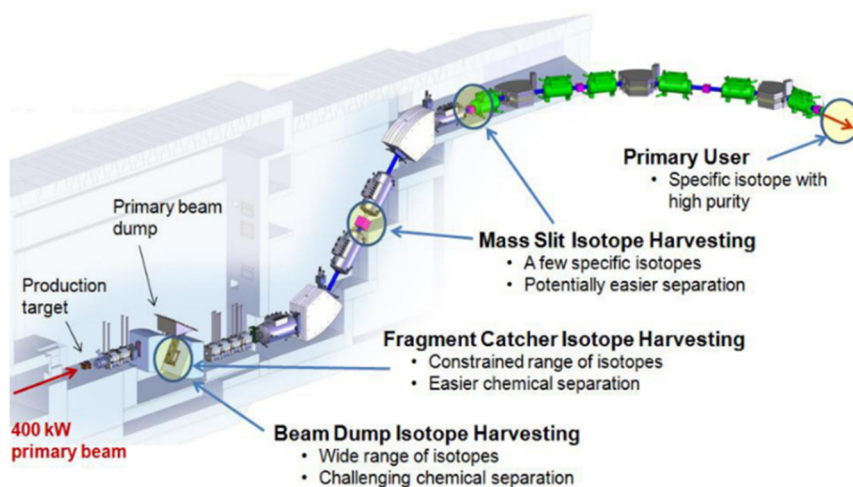


Figure 1

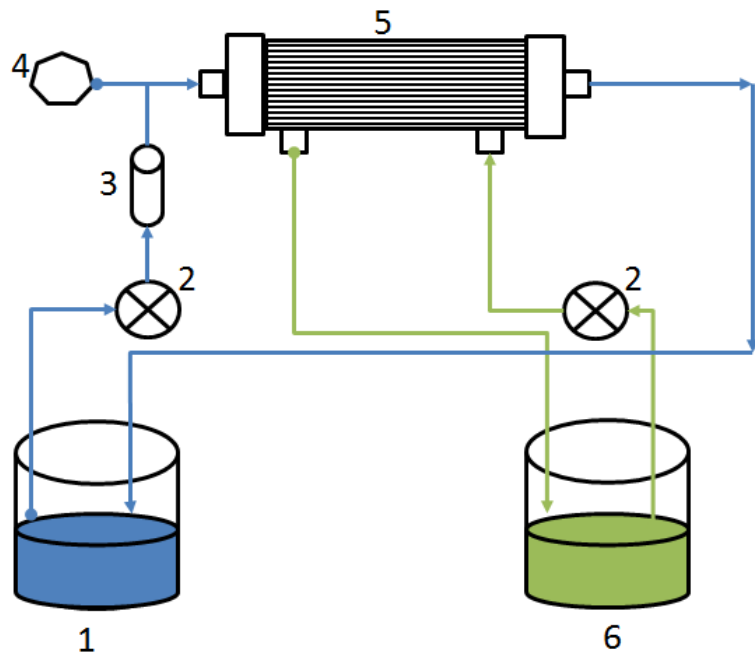


Figure 2

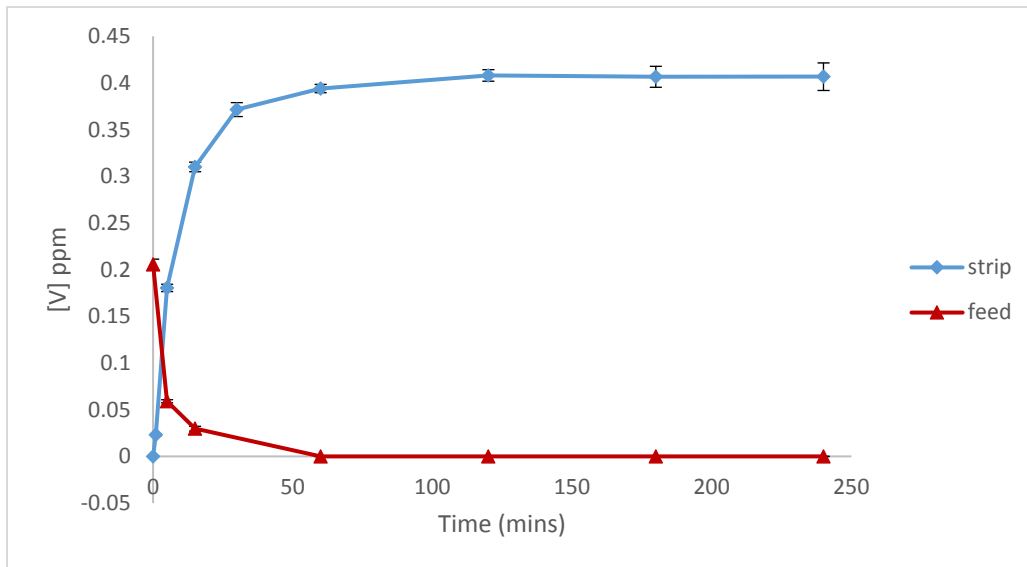


Figure 3

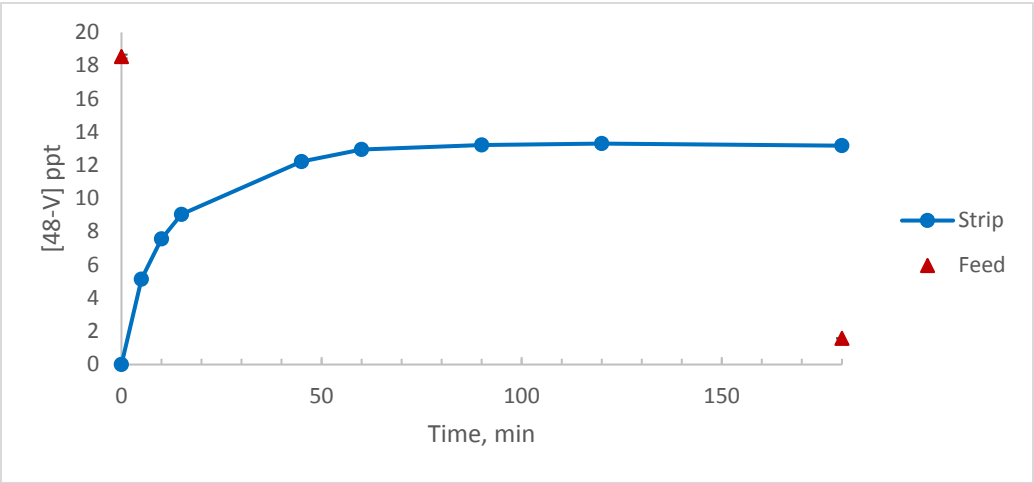


Figure 4

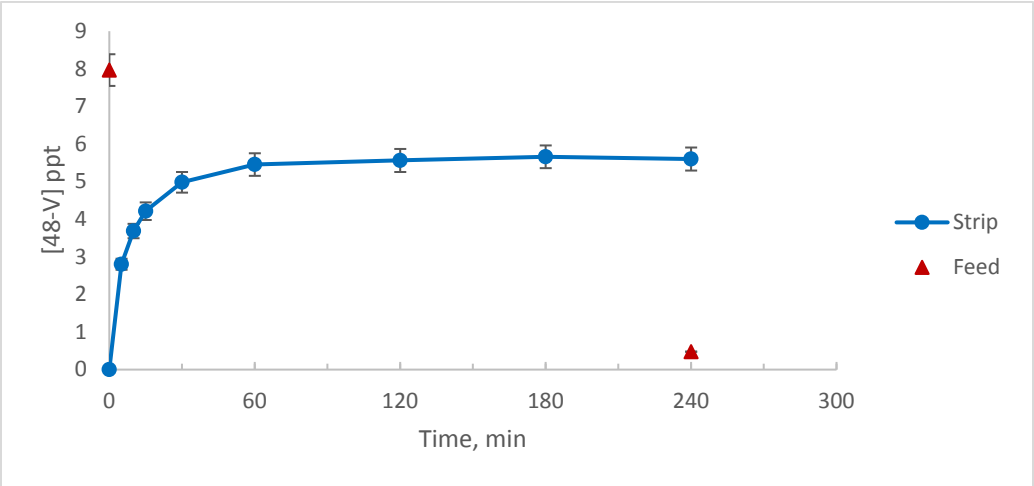


Figure 5

Characteristics	Descriptions
Model	Liqui-Cel 2.5 x 8 Extra Flow
Module diameter (mm)	66.6
Module length (mm)	281.8
Inside diameter of hollow fiber (μm)	240
Outside diameter of hollow fiber (μm)	300
Effective length of hollow fiber (cm)	15
Number of hollow fibers	35,000
Average pore size (μm)	0.03
Surface Porosity (%)	25
Effective surface area (cm^2)	1.4×10^4
Area per unit volume (cm^2/cm^3)	29.3
Tortuosity factor	2.6
Operating temperature (C)	5 – 70
Recommended flow rate mL/ min (gpm)	900 – 11350 mL/min (0.5-3)
Membrane material	X50 polypropylene membrane
Max. pressure Pa (psi)	827371 (120)

Table 1

Estimated beam dump yield, 1 week ^{58}Ni primary beam			Tested
Isotope	Particles	ppt	ppt

⁴⁸ V	1.92E+17	2	8
¹¹ B	5.00E+16	0.1	100
³² P	7.46E+16	0.6	100
⁴⁷ Ti	2.71E+17	3	10
⁵¹ Cr	2.88E+17	3.5	10
⁵³ Mn	3.59E+17	4.5	10
⁵⁸ Ni	6.59E+19	910	10000

Table 2