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Production and separation of no-carrier-added $^{181-184}\text{Re}$ radioisotopes from proton irradiated tungsten target

<https://doi.org/10.1515/ract-2017-2869>

Received August 23, 2017; accepted March 16, 2018

Abstract: Cross sections for the production of $^{181-184}\text{Re}$ isotopes from proton irradiated thin $^{\text{nat}}\text{WO}_3$ target at 20.9, 17.9, 14.9, 11.9 and 8.8 MeV projectile energies have been measured. An acceptable agreement between experimentally obtained data and available literature reports was found. The produced no-carrier-added (NCA) Re radionuclides have been separated from bulk tungsten target by liquid-liquid extraction (LLX) using trioctyl amine (TOA) and di-(2-ethylhexyl)phosphoric acid (HDEHP) dissolved in cyclohexane. A clear separation has been observed at 0.1 M and 1 M TOA with 0.1 M HNO_3 concentration. However, by using HDEHP NCA Re radionuclides could not be separated from bulk W target without its contamination.

Keywords: No-carrier-added, $^{181-184}\text{Re}$, excitation function, radiochemical separation, liquid-liquid extraction.

1 Introduction

The neutron deficient radioisotopes of Re offer a wide spectrum of applicability in various fields of sciences. The radioisotopes such as ^{181}Re ($T_{1/2} = 19.9$ h), ^{182}Re ($T_{1/2} = 64$ h) may be utilised for preparation of vascular implants [1] while ^{183}Re ($T_{1/2} = 70.0$ d) and ^{184}Re ($T_{1/2} = 38$ d) may be used for geochemical tracer studies [2]. Studies on the extraction properties of no-carrier-added (NCA) rhenium radionuclides are also important for searching appropriate room for superheavy elements. Apparently, Re is the lower

homologue of bohrium (Bh) ($Z=107$). Bh isotopes are short-lived (half-lives in the order of seconds) and hence its chemical properties are simulated with the help of NCA rhenium radionuclides. The nuclear characteristics of these radioisotopes are given in Table 1.

Reports on the production of the neutron deficient $^{181-184}\text{Re}$ by both light and heavy ion induced reactions on different target materials are available [2–18]. Tárkányi et al. [3] measured the excitation functions of $^{181,182\text{m},182\text{g},183,184\text{g},186}\text{Re}$ by proton induced nuclear reactions on $^{\text{nat}}\text{W}$ target up to 34 MeV, while Zhang et al. [4] studied the production of the same radionuclides from natural tungsten target using proton beam in the energy range 7.5–22.5 MeV. Khandaker et al. [5] measured the cross sections of the same reactions covering a wider range of proton energy from 6.6 MeV to 40 MeV by the stacked-foil technique. At the same time, Lapi et al. [6] reported the cross sections for the same reactions upto 17.6 MeV.

To exploit the suitable nuclear characteristics of Re radionuclides, it is important to separate them post production from the bulk components. The commonly used procedures for separation of NCA Re radionuclides from their target are dry distillation, thermo-chromatography [19, 20], liquid-liquid extraction (LLX) [21–23], anion exchange controlled liquid-solid sorption [22, 24–26] and also adsorption on alumina column [22, 27, 28]. Kaka-vand et al. [21] separated ReO_4^- from WO_4^{2-} by LLX using methylethyl ketone (MEK) which could extract rhenate selectively from alkaline solution of irradiated sodium tungstate. Reports on the use of anion exchange resin such as DIAION and AG 1X8 for separation of $^{186,186\text{g}}\text{Re}$ from bulk W are also available [24–26].

This paper deals with the production of the less explored neutron deficient radioisotopes of Re, i. e. $^{181-184}\text{Re}$ from WO_3 target at different proton energies, the maximum being 20.9 MeV. Though there are several reports on the proton induced production of $^{181-184}\text{Re}$ radionuclides, but all of them have used relatively thicker targets and measured cross section using stacked foil technique, while this paper reports the excitation function measurement using thin targets irradiating a single sample at each incident particle energy, therefore avoiding the corrections due to loss of energy of the incident projectile, its energy spread,

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Table 1: Nuclear characteristics of the radioisotopes of rhenium produced (<https://www.nndc.bnl.gov/chart/chartNuc.jsp>).

Radioisotope	Half-life	Decay mode	Production route	$E_{\text{threshold}}$ (MeV)	Principal γ -ray energy in keV (intensity, %)
^{181}Re	19.9 h	$\epsilon(100)$	$^{183}\text{W}(p,3n)$	16.86	360.7 (20), 365.5 (56),
			$^{182}\text{W}(p,2n)$	10.64	639.3 (6.4)
^{182}Re	64 h	$\epsilon(100)$	$^{184}\text{W}(p,3n)$	17.3	130.8 (7.5), 1231 (14.9)
			$^{182}\text{W}(p,n)$	3.6	
			$^{183}\text{W}(p,2n)$	9.8	
			$^{184}\text{W}(p,2n)$	8.8	162.3 (23.3)
^{183}Re	70 d	$\epsilon(100)$	$^{183}\text{W}(p,n)$	1.3	
			$^{186}\text{W}(p,3n)$	15.3	792.0 (37.5), 903.2 (37.9)
^{184}Re	38 d	$\epsilon(100)$	$^{184}\text{W}(p,n)$	2.3	

etc. Radiochemical separation procedure of the produced NCA Re isotopes from bulk W was also developed by LLX method using both liquid anion exchanger, trioctyl amine (TOA) and liquid cation exchanger, di-(2-ethylhexyl)phosphoric acid (HDEHP).

2 Experimental

2.1 Reagents

The reagents TOA, cyclohexane (for HPLC), were purchased from MERCK, India. HDEHP, diethylenetriaminepentaacetic acid (DTPA) and ethylenediaminetetraacetic acid (EDTA) were procured from ICN Pharmaceuticals, UK, LobaChemie, India, and SRL, India, respectively. Single element W NIST, USA standard (3163) in $\text{HF} + \text{HNO}_3$ of concentration $9.99 \text{ mg/g} \pm 0.04 \text{ mg/g}$ was used. De-ionized water ($18.2 \text{ M}\Omega \text{ cm}$) obtained from Thermoscientific Barnstead NANOpure water purification system was used throughout the experiment.

2.2 Irradiation and data acquisition

Five $^{nat}\text{WO}_3$ ($936 \text{ }\mu\text{g}/\text{cm}^2$) targets were prepared by deposition on Al foil ($7.3 \text{ mg}/\text{cm}^2$) via evaporation technique at Tata Institute of Fundamental Research (TIFR) target

laboratory. The targets were irradiated with 8.8 to 20.9 MeV incident proton beam at BARC-TIFR Pelletron, Mumbai, India. We followed the standard protocols for determining the production cross section and yield of various radionuclides, especially the recommendations made in a review on nuclear data relevant to production of medical radioisotopes [29]. In a pelletron (contrary to cyclotron) the energy spread is only a few keV and in BARC-TIFR pelletron it is maximum 30 keV for proton beams. The initial proton energy was determined by the following equation,

$$B = \frac{720.76\sqrt{mE}}{q}$$

where, B = magnetic field strength (in Gauss), q = electrical charge on the particle, m = mass of particle, E = energy of the particle. The k factor of TIFR Pelletron is 720.76. Afterwards terminal voltage (V in MV) of the pelletron is maintained following the equation:

$$E = V(1 + q).$$

The exit energy of the projectile was calculated by the software SRIM [30]. The beam current was measured with the help of a Faraday cup placed at the rear end of the target coupled with a current integrator (Danfysik). The irradiation details are given in Table 2. To allow decay of all the short-lived radionuclides irradiated targets were set aside for 30 h. Before dissolution of the sample, a series of γ -ray spectra were taken in a p-type HPGe detector of

Table 2: Yield of the produced rhenium isotopes at EOB at experimental conditions.

Incident energy (MeV)	Exit energy (MeV)	Energy at centre of mass (MeV)	Irradiation time (h)	Integrated charge (μC)	Yield, kBq			
					^{181}Re	^{182}Re	^{183}Re	^{184}Re
21.0 ± 0.03	20.81 ± 0.03	20.9 ± 0.12	10.75	2887	142.3 ± 1.3	8.3 ± 0.2	1.1 ± 0.01	2.6 ± 0.03
18.0 ± 0.03	17.76 ± 0.03	17.9 ± 0.07	4.3	3369	105.1 ± 0.5	4.7 ± 0.15	1.6 ± 0.05	0.57 ± 0.01
15.0 ± 0.03	14.75 ± 0.03	14.9 ± 0.18	2.6	1800	53.6 ± 0.30	3.2 ± 0.13	0.5 ± 0.04	0.01 ± 0.02
12.0 ± 0.03	11.71 ± 0.03	11.9 ± 0.22	3.1	1800	9.2 ± 0.25	1.5 ± 0.07	0.2 ± 0.02	0.12 ± 0.03
9.0 ± 0.03	8.64 ± 0.03	8.8 ± 0.19	3.75	1800	0	0.44 ± 0.04	0.03 ± 0.01	0.18 ± 0.02

resolution 1.99 keV at 1.33 MeV in combination with a digital spectrum analyzer (DSA 1000, CANBERRA) and Genie 2K software (CANBERRA). The sample to detector distance was ~10 cm, hence correction for coincidence loss was not needed. The radionuclides were identified by their corresponding photo peak and decay data. The energy and efficiency calibration of the detector were performed using ^{152}Eu ($T_{1/2} = 13.53\text{a}$) standard source of known activity.

2.3 Dissolution of the foil and ICP-OES measurement

The WO_3 foils were digested with 3 mL aqua regia at 80 °C using CEM MARS Xpress microwave digester for 10 min at 400 watt. Since no radioisotopes of W with measurable γ -ray energies were produced, W was monitored by ICP-OES (ICAP duo 6500 from Thermo-Scientific). Radio-frequency power of 1150 watt, pump rate at 75 rpm, auxiliary gas flow at 1L/min, nebulizer gas flow at 0.6 L/min were the parameters used during the ICP-OES measurements. Prior to the measurement, the ICP-OES system was calibrated with NIST standards varying from 0.1 to 5 ppm W. Three emission wavelengths, viz. 207.9, 209.4, 209.8 nm, were selected to monitor bulk tungsten.

2.4 Radiochemical separation

Different concentrations of TOA and HDEHP were prepared by dissolving them in appropriate amount of cyclohexane. 0.1 mL of active stock solution was added to 3.4 mL of different acid concentrations and 3.5 mL TOA or HDEHP solution. The systems were shaken and settled for 10 min. Afterwards, 2.8 mL aqueous part was taken out and counted for 1000 s on a HPGe detector. The reference was prepared similarly by addition of 0.1 mL stock solution to 3.4 mL 1 M HNO_3 , and similarly 2.8 mL was taken out and counted for 1000 s. The back extraction of Re activity was carried out by alkaline DTPA, alkaline EDTA and 6 M HNO_3 solutions.

3 Results and discussion

3.1 Analysis of gamma ray spectrum

Analysis of the γ -ray spectra of each proton irradiated thin $^{nat}\text{WO}_3$ sample was carried out 30 h after the end of bombardment (EOB). The γ -ray spectrum revealed the

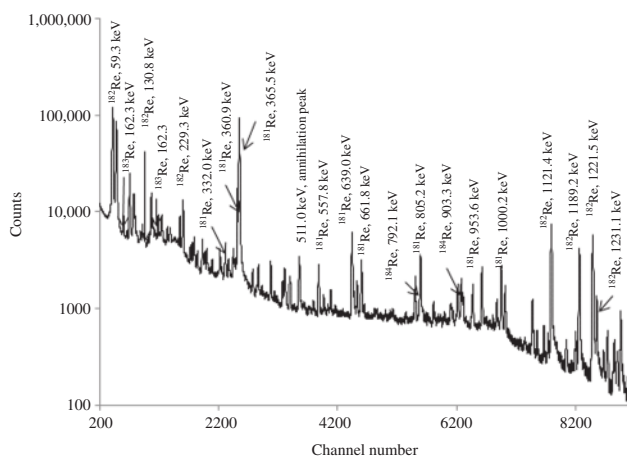


Figure 1: γ -ray spectrum of irradiated WO_3 taken 30 h after EOB.

Table 3: Measured cross sections of the $^{nat}\text{W}(p,xn)^{181-184}\text{Re}$ reactions at different energies.

Projectile energy (MeV)	Cross section, mb			
	^{181}Re	^{182}Re	^{183}Re	^{184}Re
20.9 ± 0.12	400 ± 28	66 ± 5	228 ± 17	283 ± 20
17.9 ± 0.07	332 ± 23	46 ± 3	420 ± 32	77 ± 5
14.9 ± 0.18	204 ± 14	38 ± 3	154 ± 12	2.2 ± 0.2
11.9 ± 0.22	30 ± 2	16 ± 1	56 ± 4	16.3 ± 1
8.8 ± 0.19	–	5 ± 0.4	8 ± 0.6	28 ± 2

production of $^{181-184}\text{Re}$ via $^{nat}\text{W}(p,xn)$ reactions (Figure 1). The γ -ray energies of each radionuclide given in Table 1 have been considered for the cross section measurements. The yield of each radionuclide at EOB and at experimental condition has been provided in Table 2.

3.2 Measurement of excitation functions

The five $^{nat}\text{WO}_3$ targets were irradiated sequentially at varying proton energies. The production cross sections of $^{181-184}\text{Re}$ isotopes were measured as a function of proton energy. The production cross section for each Re radioisotope was calculated (Table 3) from the activity at EOB, by using the following activation equation:

$$A = n\sigma(E)Ip(1 - e^{-\lambda t})$$

where, A = Activity (Bq) of a particular radionuclide at EOB
 $\sigma(E)$ = cross section of production of the radionuclide at incident energy E

Ip = Intensity of proton beam in particles/s

n = no. of atoms/cm²

λ = disintegration constant

t = Time of irradiation

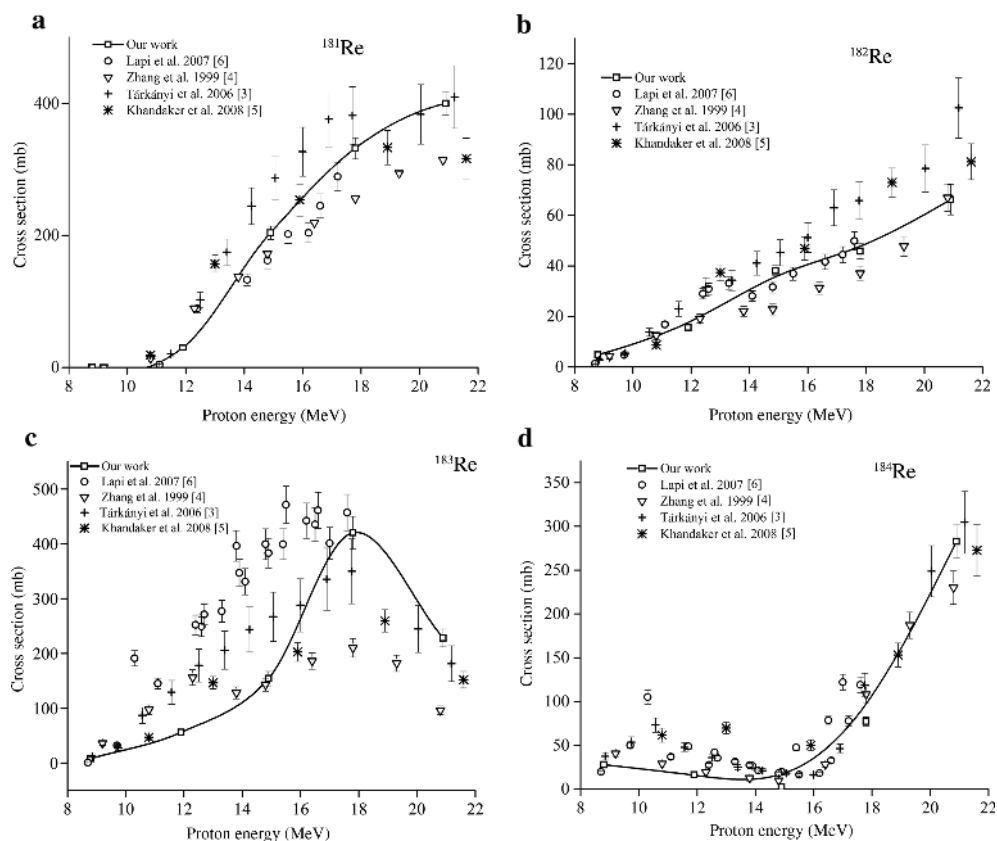


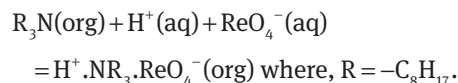
Figure 2: Comparison of present experimental cross section data for (a) ^{181}Re (b) ^{182}Re (c) ^{183}Re (d) ^{184}Re with different reported values, plotted as a function of proton energy. The curve gives eye-guides to our data.

While calculating the cross section data, various sources of errors such as efficiency calibration of HPGe detector, counting error, errors in target thickness, errors in beam current and beam energy, etc. were considered. Error due to counting statistics was different for different isotopes, ^{181}Re (~0.3%), ^{182}Re (~1.2%), ^{183}Re (~4.5%) and ^{184}Re (~1.7%). Error in determining the target thickness was ~5%. The combined uncertainties due to beam current, incident beam energy, etc. were ~10%. The total error related to the cross-sectional measurement was determined considering all the factors.

The experimentally obtained cross sections for each radioisotope at different projectile energies have been compared with previously reported results of Tárkányi et al. [3], Lapi et al. [6], Zhang et al. [4] and Khandaker et al. [5] (Figure 2). Although only five data points are reported in this work, it is observed that the experimental cross sections are well in accordance to the literature data despite the fact that the present experiment used 50–100 times thinner targets compared to other experiments. No definite clear shapes of the excitation functions can be discerned because several reactions contribute to the formation of each product.

3.3 Radiochemical separation of NCA Re and bulk W by LLX

LLX experiments with anion exchanger TOA with different concentrations of HNO_3 revealed the extraction of Re into the organic phase while W remained in the aqueous phase throughout the entire range of acid concentration. More than 90% extraction of NCA Re radionuclides was observed in single extraction without any contamination of bulk W until 0.5 M HNO_3 was used; after which a sharp decrease of Re extraction was also observed (Figure 3). The high extraction of Re in the organic phase may be explained by the fact that under oxidising conditions, Re predominantly exists in the form of the anionic perrhenate ion $[\text{ReO}_4]^-$ [31]. In presence of excess TOA, the reaction responsible for the extraction of Re is as follows [32]:



The reaction of the oxy-acid of Re (HReO_4) and TOA depends on the acid concentration in the aqueous phase. With lower acid concentrations, more loading of

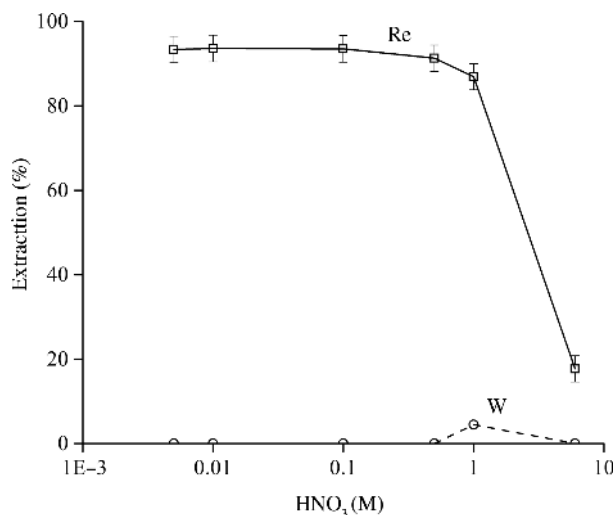


Figure 3: Extraction profile of NCA Re and bulk tungsten with 0.1 M TOA and varying HNO₃ concentration.

the compounds into the organic phase takes place [33]. Therefore in higher acid concentration the extraction of Re decreased sharply. The low or non-extractability of W may be due to the production of the neutral aqua complex H₂[WO₄] or cationic complexes [W(OH)₄(H₂O)₄]²⁺ which cannot be extracted by TOA.

To study the best extraction condition, TOA concentration was also varied keeping HNO₃ concentration fixed at 0.1 M (Figure 4). It was found that maximum separation was obtained when 1 M TOA or 0.1 M TOA were used in the organic phase and 0.1 M HNO₃ was used in the aqueous phase. The back extraction of NCA Re was also tried with different reagents like DTPA, EDTA or HNO₃,

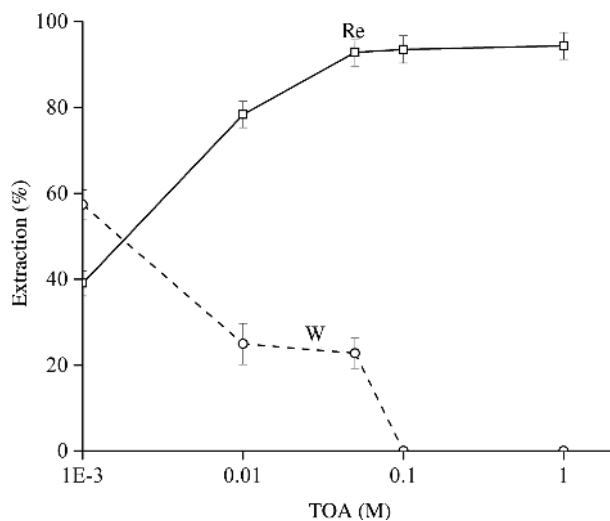
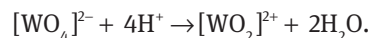
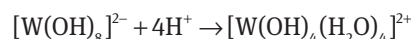


Figure 4: Extraction profile of NCA Re and bulk tungsten with 0.1 M HNO₃ and varying TOA concentration.

The back-extraction was rather difficult, with only 50% of the Re radionuclides being back-extracted in a single run. However, multiple back-extractions increased the overall isolation yield. The isolation yield was increased to 73% after two back-extractions.

The separation was also carried out with liquid cation exchanger HDEHP. LLX with 10% (v/v) HDEHP against varying concentrations of HNO₃ led to extraction of W into the organic phase while Re₃ remained in the aqueous phase throughout the entire HNO₃ concentration range (Figure 5). However, about 30% contamination from bulk W was always present in Re fraction. Therefore extraction by HDEHP would not be a good solution for separation of NCA Re radionuclides from proton irradiated tungsten target even after multiple extractions.

The monomeric tungstate ion WO₄²⁻ is stable above pH ≥ 6. In aqueous solutions, it exists as [WO₂(OH)₄]²⁻ or [W(OH)₈]²⁻. At lower pH values, tungstate ions condense into polytungstate species such as W₂O₇²⁻. The cationic tungstate species are in equilibrium with the anionic entities. On acidification, the concentration of the cationic species increases due to protonation of a portion of the hydroxo ligands of the anionic tungstates. An example of the protonation mechanism is shown below [34]:



The presence of [W(OH)₄(H₂O)₄]²⁺ species might be responsible for extraction of W in the HDEHP phase. In

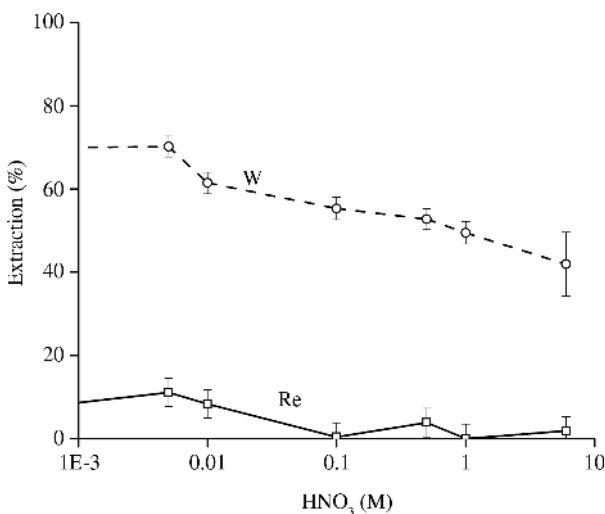


Figure 5: Extraction profile of NCA Re and bulk tungsten with 10% HDEHP and varying HNO₃ concentration.

Table 4: Optimum conditions of NCA Re nuclides from bulk W.

No	Separation condition	Extraction of W (%)	Extraction of NCA Re radionuclides (%)
1	0.1 M HNO ₃ + 0.1 M TOA	0	93.4
2	0.1 M HNO ₃ + 1 M TOA	0	94.3
3	10% HDEHP + 0.0001 M HNO ₃	70	5

Table 4 we have provided the details of separation at optimum conditions by both TOA and HDEHP.

4 Conclusion

A new data set of excitation function of ^{nat}W(p,xn) reactions have been obtained for four neutron deficient Re radioisotopes in the proton range 8.8–20.9 MeV. The data matches well with literature data. In addition, we have used simple analytical technique like LLX with both liquid anion and cation exchangers to separate NCA Re nuclides from bulk tungsten target. This paper exploits the strong affinity of rhenate ions towards the anionic exchanger TOA and establishes a clean and fast separation from bulk W. The work also clearly revealed that HDEHP was not effective in separating W from Re isotopes.

Acknowledgement: Authors are thankful to BARC-TIFR Pelletron staffs and TIFR target laboratory staffs for their cooperation during proton irradiation. We gratefully acknowledge the support through the research grants from SINP-DAE 12 Five year plan Trace, Ultratrace Analysis and Isotope Production (TULIP), Government of India. One of the authors, NN would like to thank University Grants Commission (UGC) for providing the necessary fellowship.

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