TRISKEM
Expertise in Separation Chemistry

EXTRACTION CHROMATOGRAPHY

Technical Documentation - **All Resins**

Extraction chromatographic resins

Specialty ion exchange resins

Analytical grade ion exchange resins

Sample preparation

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TRISKEM INTERNATIONAL

TrisKem International develops, manufactures and commercializes highly selective resins for the separation of a large variety of elements in numerous domains.

Triskem Resins are used for:

- the analysis of precious samples (environmental monitoring, radiation protection, bioassay)
- the recuperation of high value elements (production of radio metals for medical use and recuperation of their respective target materials and recovery of critical metals)
- the high precision determination of isotope ratios (dating/geochronology/provenancing)
- the removal of particular elements before effluent release
- quality control of final and intermediary products in numerous industries including radiopharmacy

We also commercialize a complementary **range of accessories** such as vacuum box systems and products used for sample preparation like LSC vials, filters and discs for alpha spectrometry.

Our technical support is carried out by a **worldwide recognized scientific team** consisting of radiochemistry PhDs who are amongst others members of the French nuclear equipment standardization agency (BNEN), ISO workgroups, and external IAEA experts and lecturers.

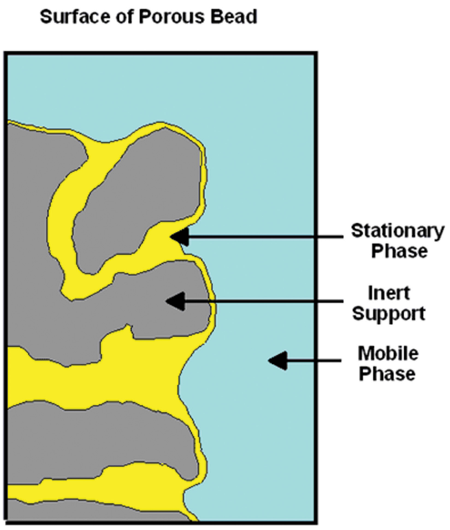
TrisKem International products have now become **the standard technology** in the field of radiochemistry, geochemistry and radiopharmacy with methods used by accredited laboratories (Orano, BNFL, CEA, NNL,...) and regulatory organizations (IRSN, IAEA, BRGM,...) and many other customers working on radionuclide and element separation and purification.

EXTRACTION CHROMATOGRAPHY

Extraction chromatography (EXC) is a technique that is ideally suited to the separation of radionuclides from a wide range of sample types. This technique combines the selectivity of liquid-liquid extraction with the ease of operation of column chromatography.

Principles

The figure hereunder is a simplified depiction of a portion of an extraction chromatographic resin bead showing the three major components of an EXC system:the inert support, the stationary phase, and the mobile phase. Liquid extractants, either single compounds or mixtures, are used as the stationary phase. Diluents can also be used to help to solubilize the extractant and to increase the hydrophobicity of the stationary phase.



Resin Bead

The mobile phase is usually an acid solution, e.g., nitric or hydrochloric acid, although complexants, such as oxalic or hydrofluoric acids, are frequently used to enhance selectivities or the stripping of strongly retained metal ions from columns.

The relationship between the distribution ratio (D) as measured in a solvent extraction (SX) system and the number of free column volumes to peak maximum, k', for the corresponding EXC system is shown in the following equation.

$$(1) \quad k = D \times \frac{V_s}{V_m}$$

Where v_s *and* v_m *are the volumes of stationary and mobile phases, respectively.*

It is important to note the concentration of extractant is usually much higher in EXC systems where diluents may be absent altogether than in conventional SX systems. Therefore, D and k' are not usually measured directly for EXC systems, but calculated from the weight distribution ratio, D_w, which is easily measured.

The weight distribution ratio, D_w, is obtained by measuring the amount of a given metal ion taken up by a measured weight of resin from a given volume of aqueous solution. In the studies described in this report, radioactive isotopes were used to measure the uptake of metal ions. The weight distribution ratio is then calculated using the following equation :

$$(2) \quad D_w = \frac{A_0 - A_s}{A_s} \times \frac{ml}{g}$$

Where $A_0 - A_s$ *= activity sorbed on a known weight of resin (g), and* A_s *= the activity in a known volume (mL) of solution.*

The volume distribution ratio D is calculated from the volume of stationary phase per gram divided into D_w. The volume of stationary phase is obtained from its weight percent sorbed on the inert support and its density (the density of the stationary phase is obtained from an independent measurement.) The number of free column volumes to peak maximum, k', is then calculated from equation (1). The quantities, v_s and v_m, are measured from the weight of resin required to fill a column to a known volume and the density of the EXC resin.

Details for the calculation of D and k' and the independent measurement of v_s and v_m can be found in Table 1 gives the ratio of v_s/v_m and the factors to convert from D_w to k' for five EXC resins.

Converting D_w to k'

Resin	V_s / V_m	To convert D_w to k' divide by
TEVA	0.23	1.9
UTEVA	0.25	1.7
TRU	0.22	1.8
Actinide	0.20	1.9
SR	0.22	2.0

Achievement of Separation

To achieve separation in EXC, band spreading must be sufficiently small to avoid early breakthrough and to avoid excessive cross-contamination of the constituents that one is trying to separate. Even if the extractants comprising the stationary phase exhibit very high selectivity for one of the ions, poor column efficiency, as manifested in excessive band spreading, can result in essentially no practical separation.

Column efficiency is generally expressed in terms of the height equivalent to a theoretical plate. Plate height is a complex function of a number of physical and chemical factors.

In EXC systems, column efficiency is determined primarily by flow phenomena, diffusion in the stationary phase and extraction kinetics.

The relative importance of each of these three factors to column efficiency depends on the specific chemical system, the particle size and porosity of the support, the extractant loading and mobile phase velocity and the operating temperature.

Achievement of Selectivity

The EXC resins listed cover a wide range of selectivities and enable one to perform a number of novel highly efficient separations of selected fission products and individual actinides or groups of actinides. The following pages show the acid dependencies for the uptake, as measured by k', of selected actinides and non-actinides on numerous resins of varying selectivity. Table 2 summarizes the important properties of some of these resins.

Properties of EXC Resins

Particle size (µm) 100-150, 50-100, 20-50
Bed density (g/mL) 0.33 to 0.39
Free column value (% bed volume) 65 to 69

Working capacity*	(mg/mL of Bed)
TEVA	15 (Pu)
UTEVA	32 (U)
TRU	4.5 (Am)
SR	6.5 (Sr), 12 (Pb)
LN	11 (Nd)
Actinide	14 (Am), 8,6 (Nd)

*50% of maximum capacity
By E. Philip Horwitz

APPLICATIONS

Environmental monitoring and Bioassay

TrisKem's extraction chromatographic resins allow the separation and determination of radionuclides (e.g. actinides and fission products) from bioassay samples and environmental matrices e.g. **soil, sediment, vegetation and seawater samples.** Their use allows a rapid and precise determination of radionuclides in emergency and routine situations, as well as, combined with suitable sample preparation methods, to obtain very low detection limits through analysis of large volume samples.

Radiopharmacy

TrisKem manufactures selective resins for **Lanthanides, Sr, Cu, Zr, Ac, Ge, Sc, Ga** and many other radionuclides for medical purposes. We develop rapid, highly specific separation techniques for:

- Separation of radionuclides from irradiated targets
- Quality control of radionuclides for medical use
- Post-generator purification
- Radioprotection and Radioanalysis

Decommissioning

The high selectivity of our resins not only allows the analysis of standard alpha and beta emitters (actinides, Sr-89/90, Ni-63, Tc-99,...) from high matrix samples including concrete, spent resin, but also the determination of so called difficult-to-measure (DTM) radionuclides such as **Sn-121m/Sn-126, Zr-93,...**

We are constantly working on developing new methods according to your needs. Methods for the following radionuclides are currently under development: **Se-79, Sb-125, Cs-135, Nb-93m/Nb-94, Ag-108m, Pd-107, ...**

Geochemistry and Metals Separation

TrisKem's expertise in separations and it's variety of different resins with varying selectivities provides a number of opportunities to solve analytical problems through sample preparation. With our broad offer of products and long experience, TrisKem can help you meet lower detection limits with less uncertainty. The problems encountered are frequently centered on removing matrix that interferes with the instrumental measurement. ICP-MS is an example. Isobaric interferences often have to be removed in order to allow accurate determination of your analytes. Selectivity for the analyte of interest is important in these cases. Extraction chromatography is already widely used in various applications including **geochronology, isotope ratio determination and provenancing.**

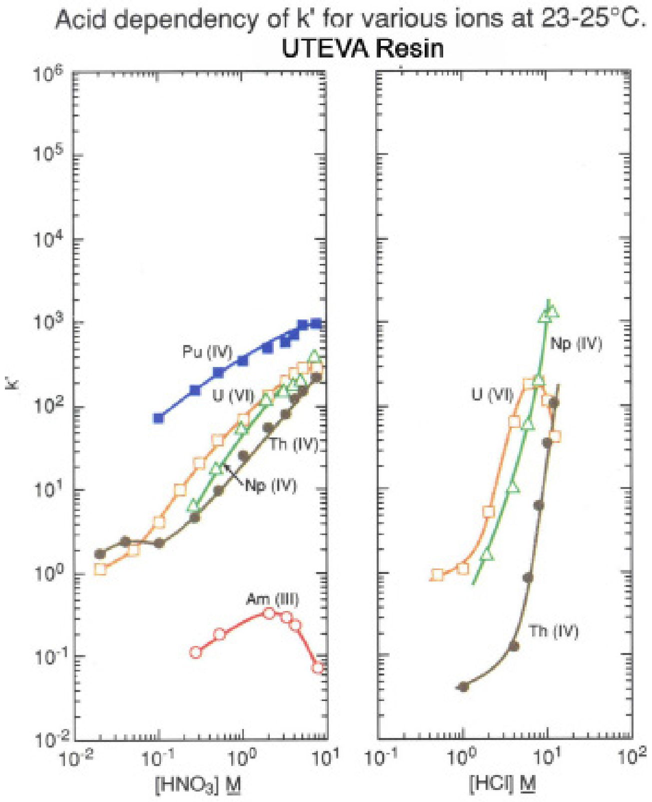
UTEVA Resin

UTEVA Resin (Uranium und TEtraValents Actinides), is used for the separation of Uranium and tetravalent actinides like Np, Th and Pu. The extractant coated on the inert support is the DP[PP] (Dipentyl pentylphosphonate) which shows an affinity for U(VI), Th(IV), Np(IV) and Pu(IV) over commonly occurring constituents such as Al, Fe, alkali and alkaline earth ions.

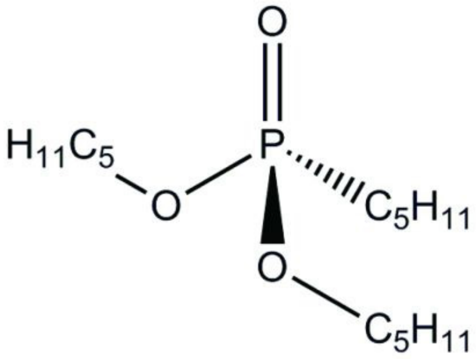
It's robustness against interference from common matrix elements greatly facilitates e.g. the analysis of U in small soil samples by alpha spectrometry, the completely digested sample can be directly separated on UTEVA resin after dissolution in 3M HNO_3/1M $Al(NO_3)_3$.

Depending on the radionuclides to be measured, UTEVA Resin may be used alone or in combination with other resins. Combined with TRU Resin, UTEVA Resin is commonly used to sequentially separate U, Pu and Am.

UTEVA Resin is also frequently used for the separation of Zr from various matrices.



k' values, UTEVA Resin, various elements, HNO_3 and HCl.

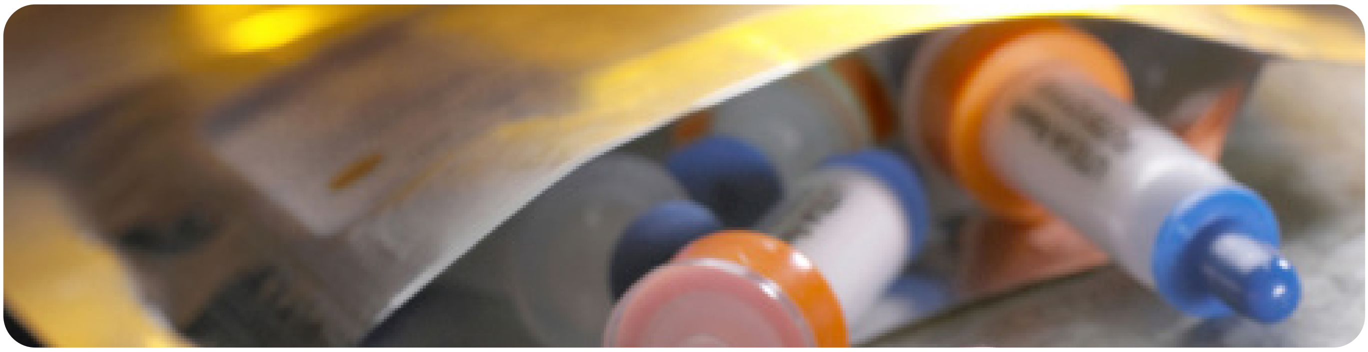


Dipentyl pentylphosphonate (DP[PP]) also called Diamyl amylphosphonate (DAAP)

Main applications

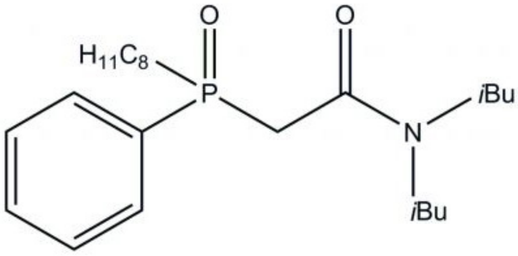
- U in soil, water and urine samples
- Actinides in soil, water and urine samples
- Separation of Zr

Ready-to-use methods available at: www.triskem.com



TRU Resin

The stationary phase of TRU (TRans Uranium elements) Resin consists of a mixture of a carbamoylmethylphosphine oxide (CMPO) and tri-n-butyl phosphate (TBP).



CMPO

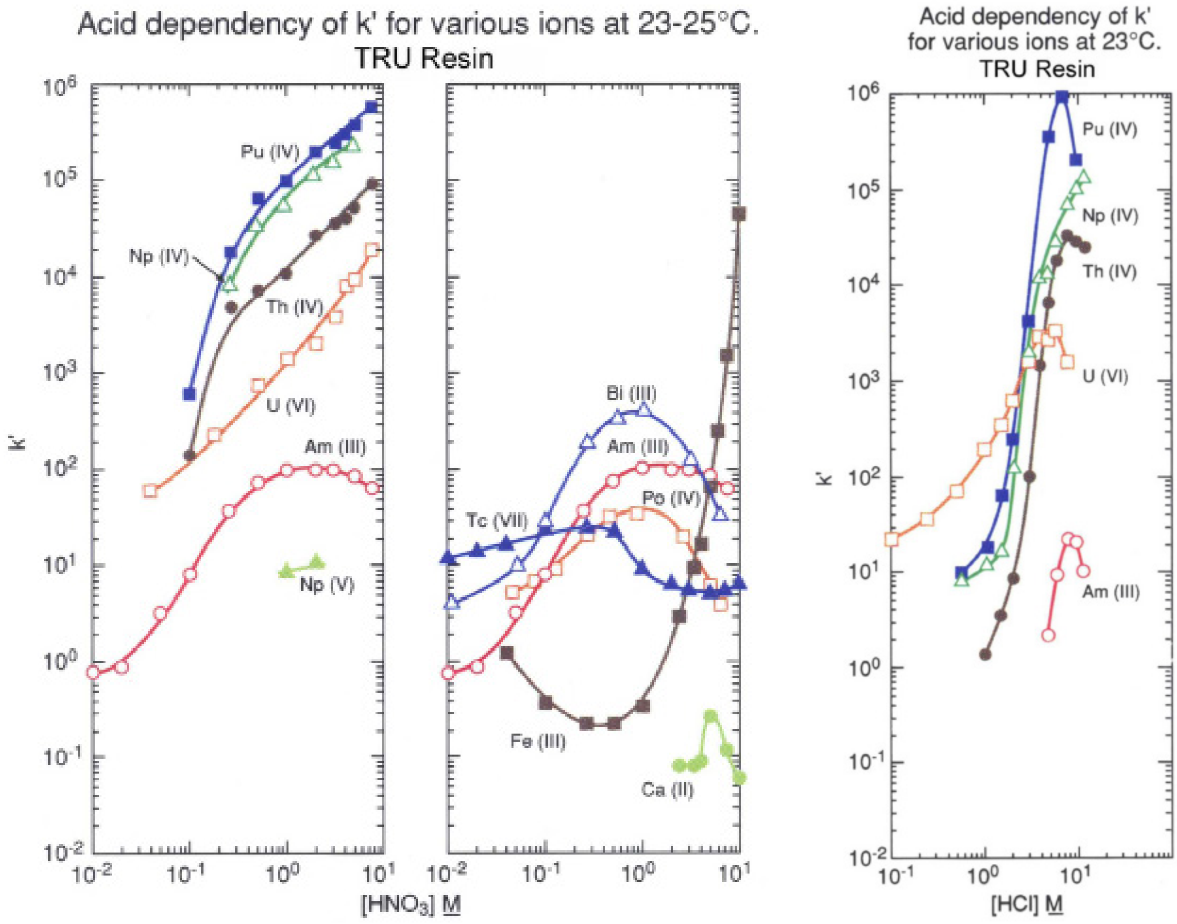
TRU Resin is used for the extraction and separation of tetra- and hexavalent actinides and, other than TEVA and UTEVA, also Am(III). Similar to UTEVA and TEVA Resins, TRU Resin allows by adjusting acid and acid concentration separating the actinides from matrix elements and each other.

Fe(III) shows no affinity for the resin in the range of 0.05 - 2M HNO_3. Above 2M HNO_3 Fe(III) affinity is increasing with the HNO_3 concentration. TRU Resin properties towards Fe can be used for the separation and measurement of Fe-55.

The TRU Resin has further been used for the concentration of lanthanide groups and the removal of matrix elements upfront to their separation on LN Resin. Burnett et al. used the TRU resin in combination with anion exchange for the purification of Pa.

Main applications

- Actinides in soil, water and urine samples
- Fe-55 in water samples



k' values of different elements in HNO_3 and HCl media on TRU Resin

Ready-to-use methods available on our website: www.triskem.com

TEVA Resin

TEVA Resin is mainly used to separate TEtraValent Actinides and technetium. The extractant that gives it its selectivity to the TEVA Resin is Aliquat® 336 a quaternary ammonium salt. k' values of various radionuclides in HNO_3 and HCl are presented in figure hereafter. Differences in k' values between HNO_3 and HCl media may be used to separate Th, Np and Pu from the other actinides.

Pu(IV), Np(IV) and Th(IV) show maximum retention in 2-4M HNO_3. In this acidity range, Am(III) and U(VI) are not fixed.

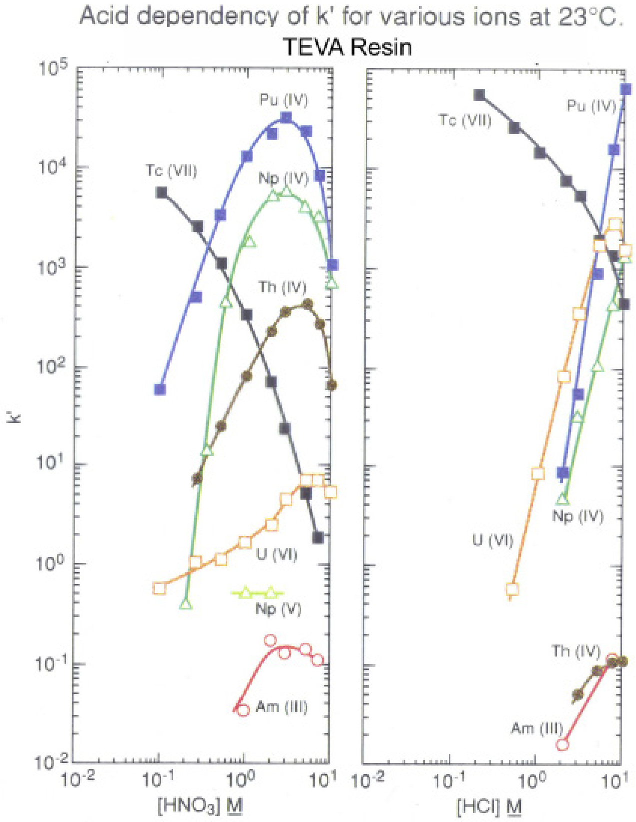
k' differences between HNO_3 and HCl media may be used to separate Th from the other actinides. When the sample is loaded on the resin from 4M HNO_3, Pu(IV), Th(IV) and Np(IV) are retained. Th may then be eluted with 6M HCl while Pu(IV) and Np(IV) remain on the resin in these conditions.

Pu might be stripped out of the resin with 4M HNO_3 or 8M HCl, after having reduced its oxidation state from +IV to +III.

Under specific conditions, Am may be fixed on the TEVA Resin and separated from light lanthanides. In 1-2M NH_4SCN – 0.1M HCO_2H, Am is fixed on the resin while La and Eu are eluted. Am is then stripped from of the resin with 0.25M HCl.

Further to actinide separations TEVA Resin is also used to separate Tc and Re from various sample matrixes.

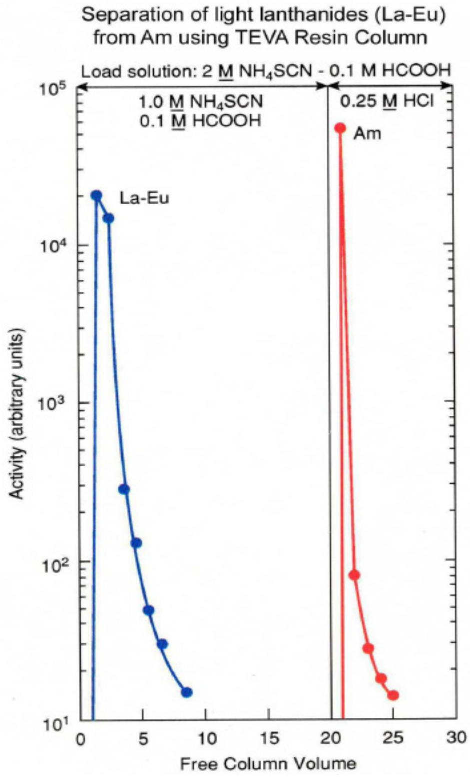
TEVA Resin is also available in form of discs allowing for the rapid analysis of Tc-99 in aqueous samples of elevated volume.



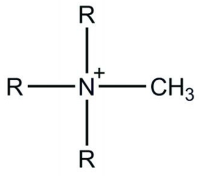
k' values, TEVA Resin, various elements, HNO_3 and HCl

Main applications

- Actinides in soil, water and urine samples
- Tc-99 in water and urine samples
- Am/LN separation



Separation of La-Eu and Am on TEVA resin



Quaternary ammonium salt Aliquat® 336,
R = octyl or decyl

Ready-to-use methods available at: www.triskem.com

SR Resin

SR Resin is based on the principle of ionic recognition. The stationary phase consists of a dicyclohexano 18-crown-6 derivative dissolved in octanol.

The data in the following figures shows that the uptake of Sr is greater than the uptake of other alkali and alkaline earth ions and most other cations tested.

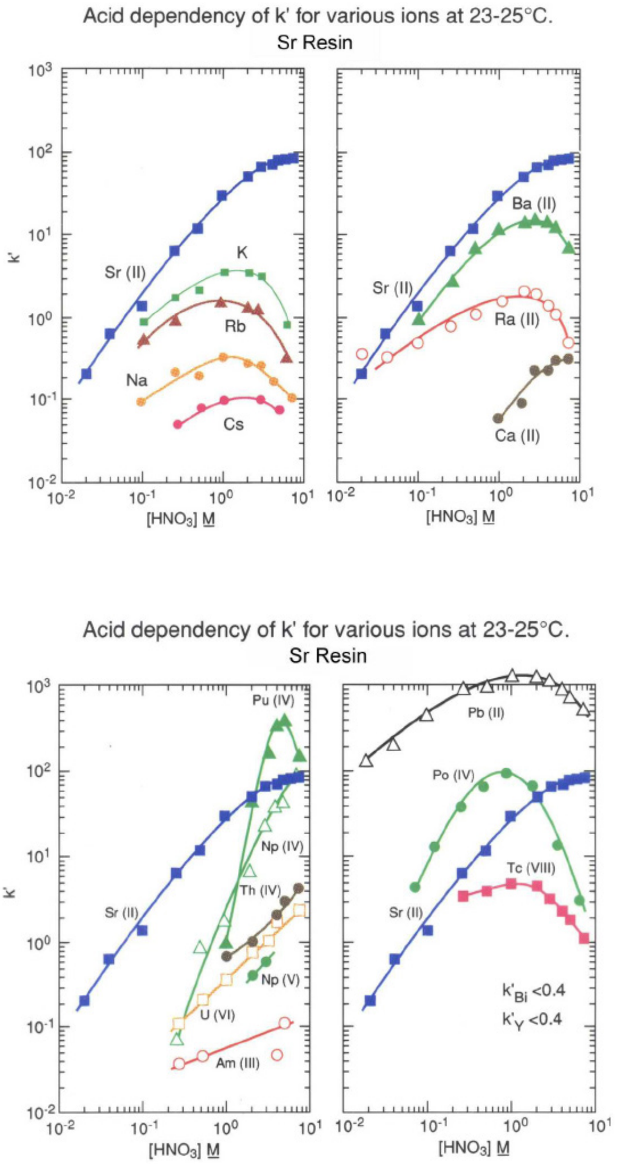
The selectivity for Sr over Ca is particularly noteworthy because Ca is a major constituent in many samples. Pb is even stronger retained on the SR Resin, allowing for a facile elution of Sr from the resin using dilute HNO_3, leaving Pb retained.

Potassium shows very small affinity for the resin. However, if its concentration is larger than 0.01M, which is often the case in soil, vegetables and other environmental matrices, it affects the strontium uptake. In these cases, it is advisable to perform an earth alkaline oxalate precipitation to eliminate potassium, prior to the load.

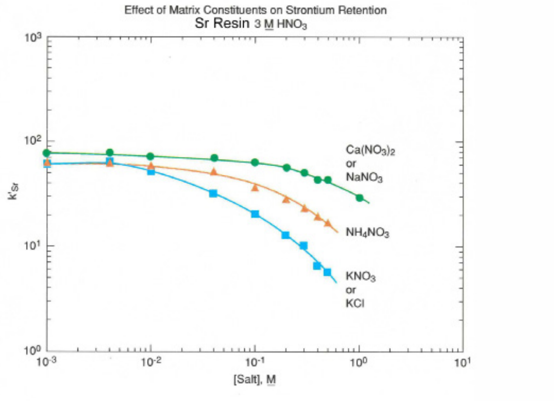
The high selectivity of the SR Resin for Pb makes its elution from the resin more complicated, which is why the PB Resin has been developed.

SR Resin typically finds application in the determination of Sr-89/90 and Pb-210/Po-210 in biological and environmental samples, as well as in the determination of Sr and Pb isotopic ratios by mass spectrometry and in radionuclide production.

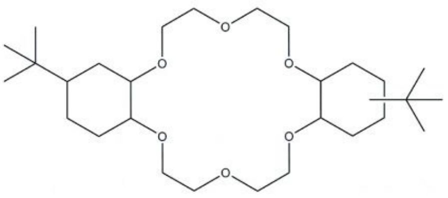
Ready-to-use methods available at: www.triskem.com



Acid dependencies of k' values of selected metal ions by SR Resin



Interference of different elements on strontium retention



4,4'(5')-di-t-butylcyclohexano-18-crown-6

Main applications

- Sr-89/90 in soil, food, water and environmental samples
- Sr and Pb isotope ratios
- Pb-210 in soil samples
- Pb-210 and Po-210 in food, water and environmental samples

PB Resin

Lead Resin (PB Resin) is composed of the same crown-ether used in the SR Resin, isodecanol is used as diluent though.

The isodecanol, having a longer carbonated chain than n-octanol, is used to facilitate lead elution from the resin.

The PB Resin has a very similar selectivity than the SR Resin, but k' values are in general lower, allowing for simpler Pb elution. PB Resin is typically used for the determination of Pb-210 in water samples.

Lead affinity (k'_{Pb}) for PB Resin over the range 0.01 to 10 M HNO_3 is varying from ~20 to 800. In HCl media, k'_{Pb} for Pb Resin varies from 20 to 100. The maximum retention is observed between 0.05 to 2 M HCl. Below and above these HCl concentrations, Pb affinity for the resin is rapidly decreasing.

Monovalent and divalent ions show similar k' values as those observed on SR Resin.

Sodium and calcium do not interfere with the lead uptake on the PB Resin for concentrations from 0.01 to 1 M.

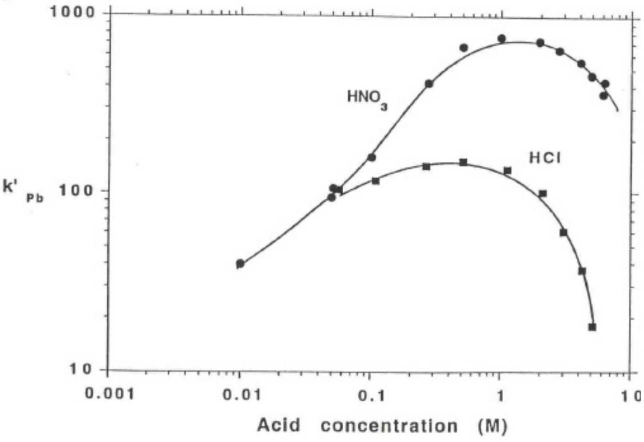
The presence of potassium up to 1M still allows a lead uptake with k'_{Pb}~80.

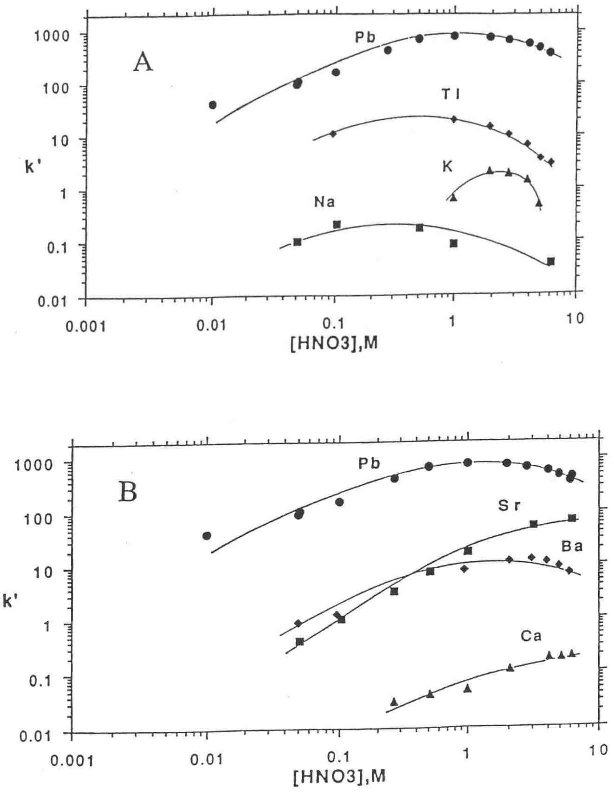
Elution of lead can be performed with different solutions as 0.1M or 0.05M ammonium citrate, dilute citric acid, 0.1M ammonium tartrate or even water.

Main applications

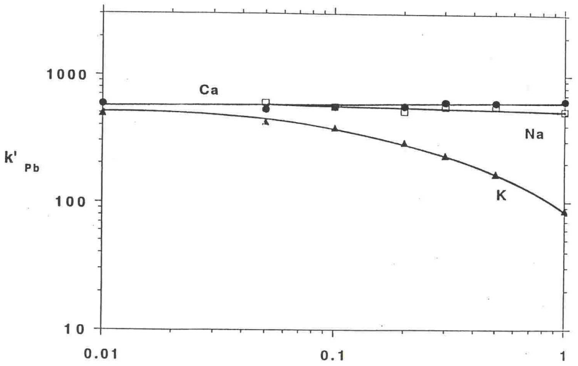
- Pb-210 in water samples

Ready-to-use methods PBW01 Lead-210 and Polonium-210 in Water PBS01 Lead in Soil... available at: www.triskem.com





Acid dependencies of k' values
of selected metal ions by PB Resin



Influence of interfering ions on k'_{Pb} on PB Resin
in HNO_3 media

DGA Resins

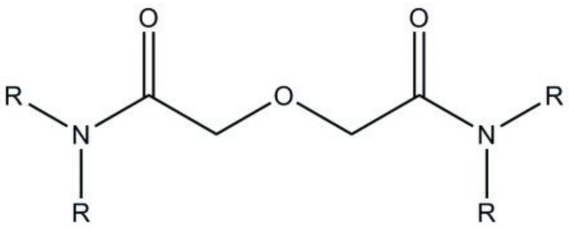
DGA Resin is an extraction chromatographic resin based on either N,N,N',N'-tetra-n-octyldiglycolamide (DGA, Normal resin, abbreviated DN Resin, TO-DGA in literature) or N,N,N',N'-tetrakis-2-ethylhexyl-diglycolamide (DGA, Branched Resin, abbreviated DB Resin, TEH-DGA in literature) extractant.

The following figure compares the k' values for Am(III) obtained on a number of extraction chromatographic resins. As it can be seen the DGAs Resins show very high k' at high acid concentrations, whereas it can easily be eluted with dilute acid solutions. It compares k' values of selected actinides on normal and branched DGA Resins.

This combined with DGAs stability against interfering elements like Al(III), Fe(III) or Ti(IV) makes it an interesting tool for the etermination of Am in environmental samples, for example in sequential separation methods for the determination of actinides in large soil or food samples.

Horwitz et al. showed that cations that are forming very large and soft chloro-anions (e.g. $FeCl_4^-$) strongly increase Am retention on DGA. This effect can be used to concentrate Am (together with other actinides) from elevated volumes of soil sample (100g) leachates while most matrix elements pass the DGA Resins without being retained.

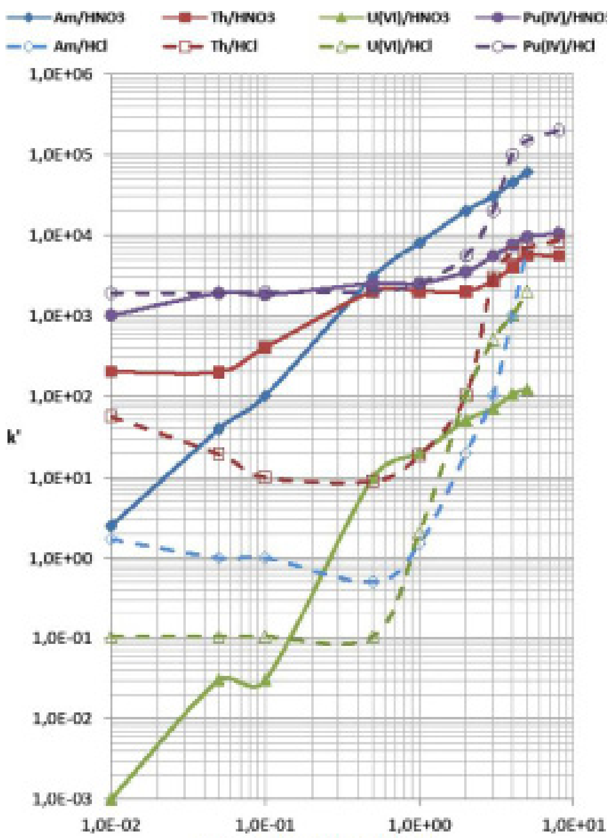
It also was shown that DGA can successfully be used for actinium separation, in environmental applications e.g. in the determination of Ra-228 as well as in the field of radiopharmaceuticals, for example in the separation of Ac-225 for the preparation of Bi-213.



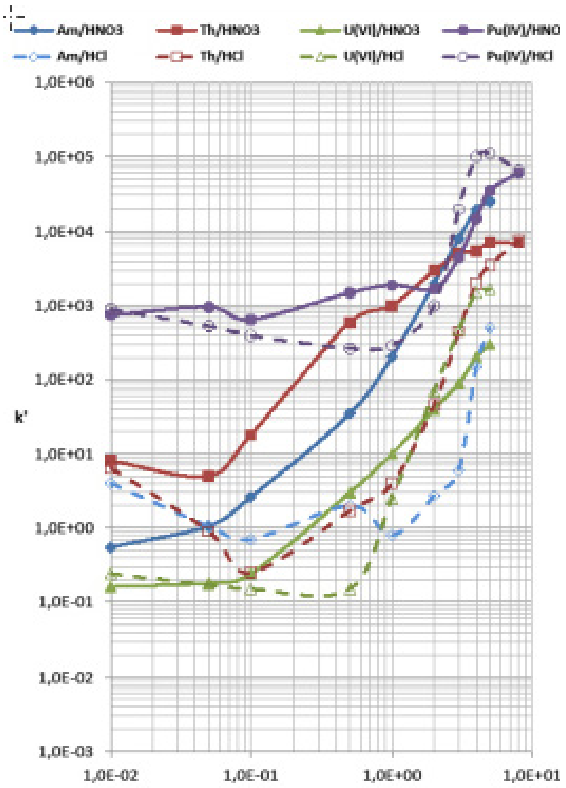
DGA resin extractant, R = octyl or ethylhexyl

Main applications

- Preconcentration of actinides from large soil and environmental samples
- Determination of Am in large soil and food samples
- Separation of Ac-225
- Determination of Ra-228

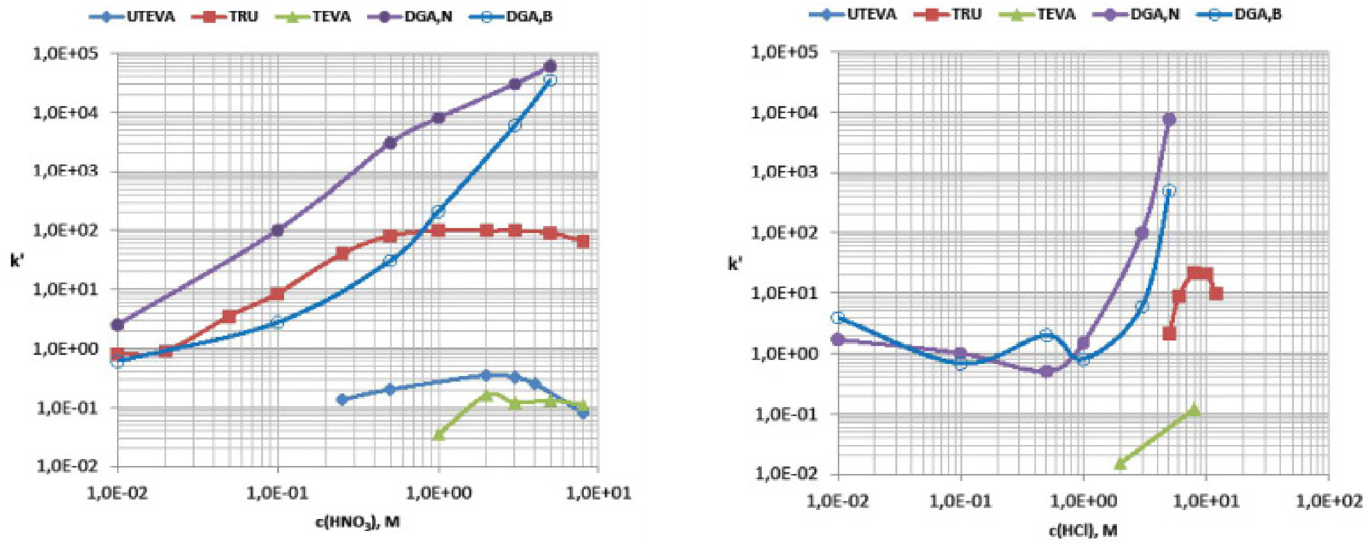


c(H+), M - DGA,N (TODGA)

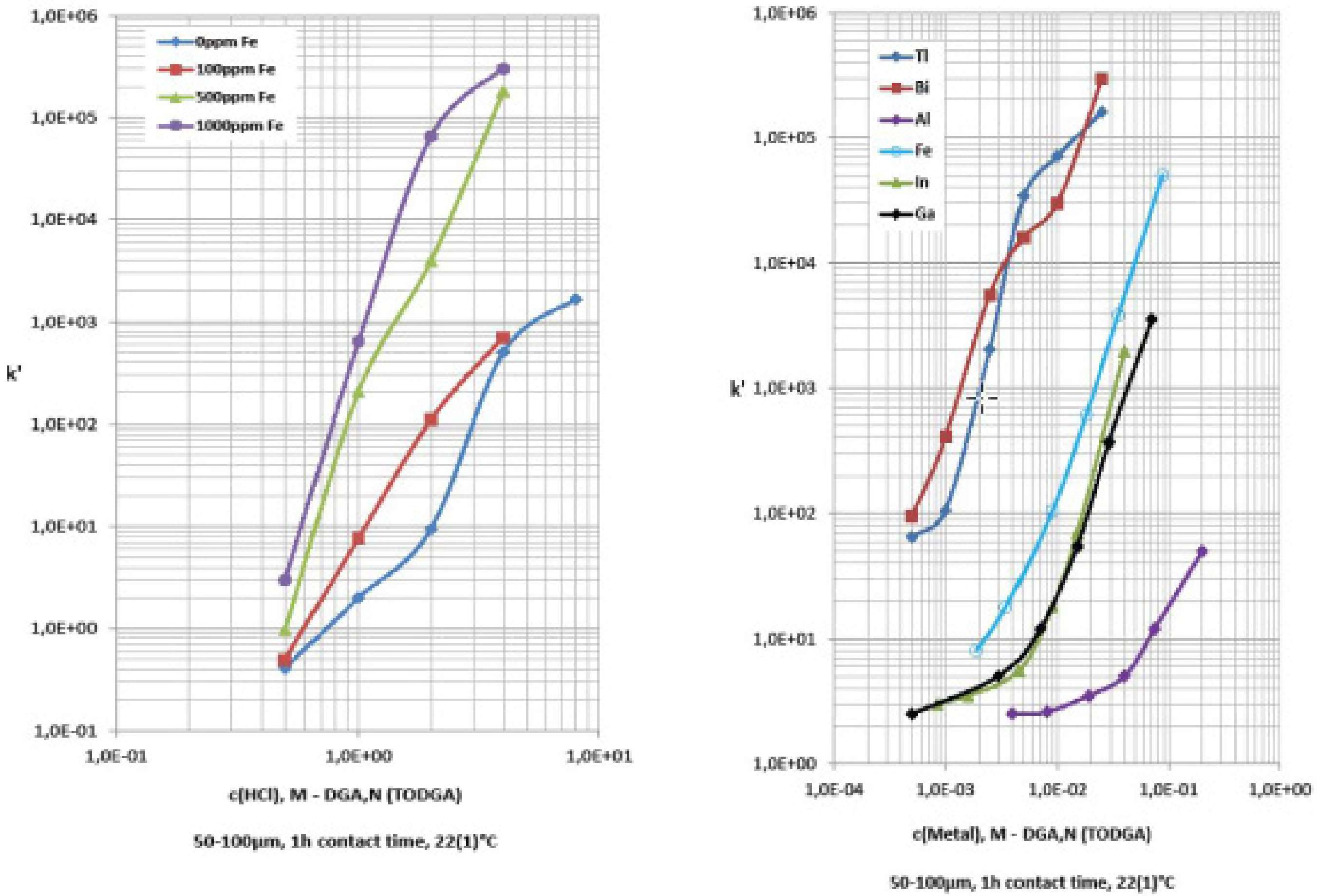


c(H+), M - DGA,B (TEH-DGA)

Comparison of k' values of different actinides on DGA Resins



Comparison of Am k' values on different extraction chromatographic resins

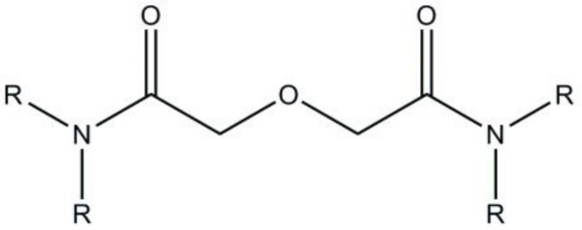


Am k' values for varying metal and HCl concentrations on Normal DGA

DGA Sheets

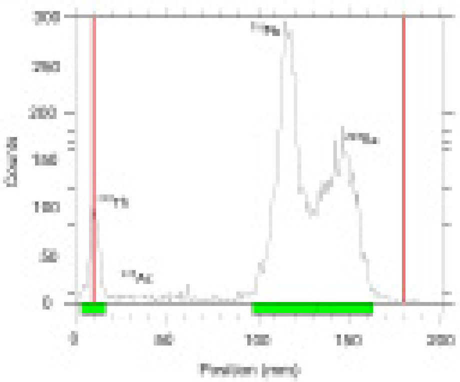
A DGA impregnated chromatographic paper

Radionuclide separation and radionuclidic purity determination was never easier. Separation of generator isotopes and radionuclide mixtures on DGA impregnated chromatographic paper, which has been developed at the CVUT, including mixtures like Ac-227, Th-227, Ra-223, Sr-90/Y-90, Ge-68/Ga-68, Mo-99/Tc-99m, Pb-212 and Ac-225/Bi-213 is now possible using one separation material, just by changing the composition of the mobile phase (diluted mineral acids like 1M HNO_3 or HCl). The chromatographic paper is available impregnated with DGA with variable active compound loading (0.1-20%) and supports (Whatman paper = DGA Sheets, iTLC support = DGA iSheets).

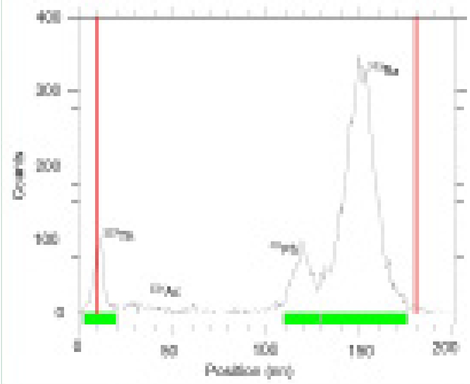


The method is easy to validate and TLC scanners or alternatively, after cutting the paper, common radiometers may be used to determine radionuclidic purity of a generator eluate or a purified radionuclide product.

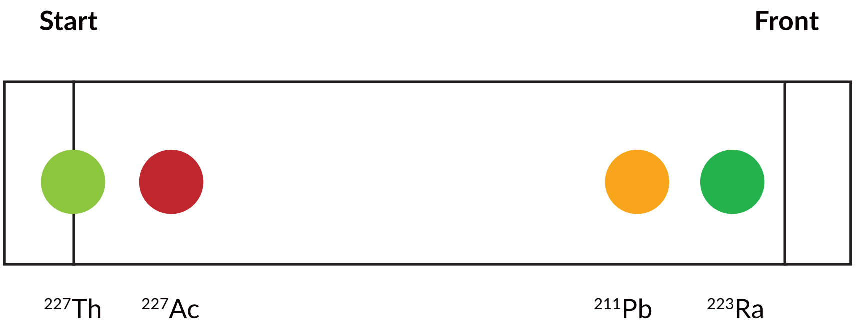
Proposed standard sheet dimensions are 5 x 20cm, 10 x 10cm and 20 x 20cm. Other selectivities/extractants, formats and custom dimensions are also available upon request.



Radiochromatogram measured immediately after separation



Radiochromatogram measured one hour after separation



Scheme of a chromatographic separation of a mixture of Ac- 227 and its descendents.
Th-227 remains on start, Ac-227 has a retention factor R_f of ca. 0.2, Pb-212 of ca. 0.7 and Ra-223 of ca. 0.9.

All data provided by J. Kozempel and M. Vlk, CVUT

DGA application notes available on our website: www.triskem.com /tki-methods.php

LN Resin Series

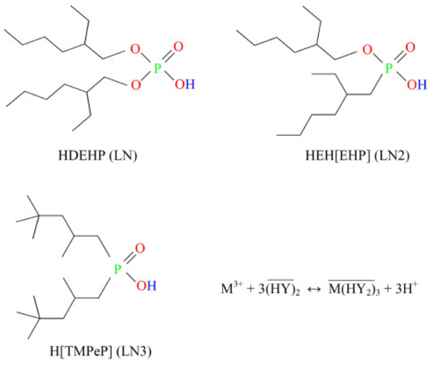
The LN Resins contain dialkyl phosphoric (LN), phosphonic (LN2) or phosphinic (LN3) acids, the acidity of the impregnated extractants is diminishing in the order LN>LN2>LN3. This difference in acidity is having an impact on the retention behavior of the resins.

LN Resin finds application in methods for the determination of Ra-226 and Ra-228 in environmental samples and the separation of lanthanides. In the latter case it is frequently employed in combination with the RE Resin which is used to concentrate the lanthanides, and to eliminate part of the matrix.

Besides the LN Resin, TrisKem also provides LN2 Resin, which can be used for the separation of heavy lanthanides (e.g. separation of Lu-177 from irradiated Yb-176) and LN3 which is used in Lanthanide separation and the separation of Zn and Co.

The figure on the right compares the relative selectivity of the resins for lanthanide elements (normalized to k'_{La} /LN2=1)

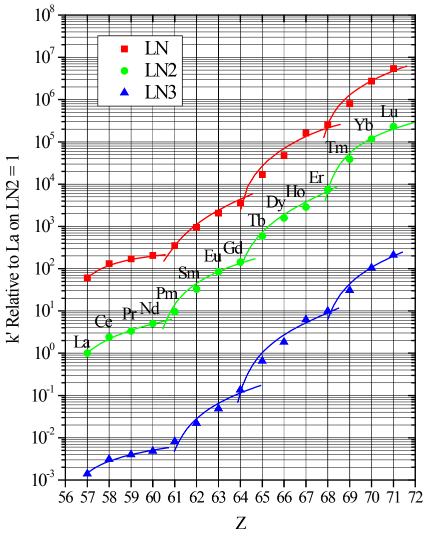
The figure hereufter summarizes the k' values of selected cations on all three resins.



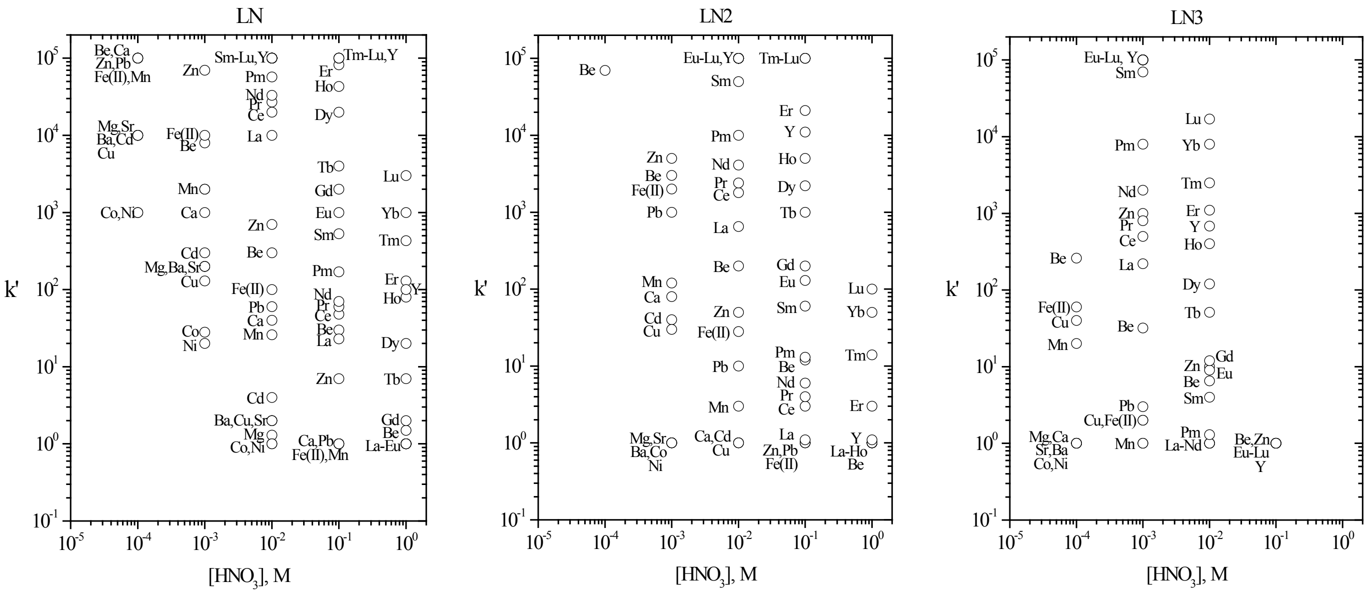
Extractants used in LN series Resins

Main applications

- Separation of lanthanides
- Ra-226 and Ra-228 in water samples



Relative k' values of lanthanides normalized to La retention on LN2 Resin (k'_{La} /LN2=1)



Capacity factor k' of various cations in HNO_3 on LN / LN2 / LN3 Resins

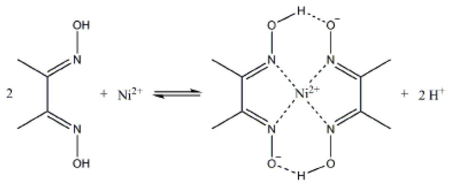
NI Resin

Nickel (NI) Resin consists of dimethylglyoxime (abbreviated DMG) coated on an inert support, and is dedicated to the separation of nickel from the other elements.

Unlike other extraction chromatographic resins, it is based on an on-column precipitation of nickel with dimethylglyoxime at pH 8-9.

The pink Ni(DMG)$_2$ complex formed during precipitation is very stable and insoluble under these conditions, and is thus retained on the column.

The sample loading is performed in presence of ammonium citrate in order to keep elements such as Fe in solution. After rinsing with pH 8-9 ammonium citrate solution in order to remove matrix elements and interferents Ni can be eluted from the column using 3M HNO$_3$. The following table shows typically obtained decontamination factors.



Precipitation of nickel cations with 2 molecules of dimethylglyoxime, Ni(DMG)$_2$.

Decontamination factors obtained on NI Resin for different radionuclides:

Radionuclides	Decontamination factors (D_f)
Cr-51	3.5E+02
Mn-54	8E+03
Fe-55	4E+0.2
Co-58	1E+03
Co-60	1.1E+03
Nb-95	1.3E+02
Cs-134	2.8E+03
Cs-137	3E+03

Main applications

- Ni-63 and Ni-59 separation in water, environmental and decommissionning samples

RE Resin

The RE (Rare Earth) Resin, like the TRU Resin, is composed of CMPO dissolved in TBP and impregnated onto an inert support. In case of the RE resin the proportion of CMPO used is higher than for the TRU Resin, with the aim of increasing its affinity for the REEs.

The resin is mostly used for the separation of group Rare Earth Elements (REE), especially of the heavy REEs, mainly with respect to matrix elements. It is often used with the LN Resin which is used for the actual separation of the lanthanide elements.
RE Resin is further used for the separation and purification of Y isotopes, e.g. of Y-90 for medical use.

Main applications

- RE in soil, water and environmental samples
- Y separation

CL Resin

The CL Resin is based on an extraction system that is selective for platinum, palladium, gold, mercury and silver and is mainly used for the separation of chloride and iodide, especially in the context of Cl-36 and I-129 analysis.

The selectivity for halides is introduced by loading the resin with Ag^+ allowing good selectivity for anions, especially halides, forming sparely soluble or insoluble Ag complexes. Since the resin retains Ag^+ over a wide range of pH values it also allows loading chloride and iodide from various conditions (slightly alkaline to strongly acidic), ideally under reducing conditions to assure their presence as halides.

After loading and rinsing of the resin (removal of matrix elements and interferents), chloride can be easily eluted from the resin using dilute SCN^- solutions whereas iodide remains fixed. Iodide can then be eluted from the resin using a moderately concentrated S^{2-} solution.

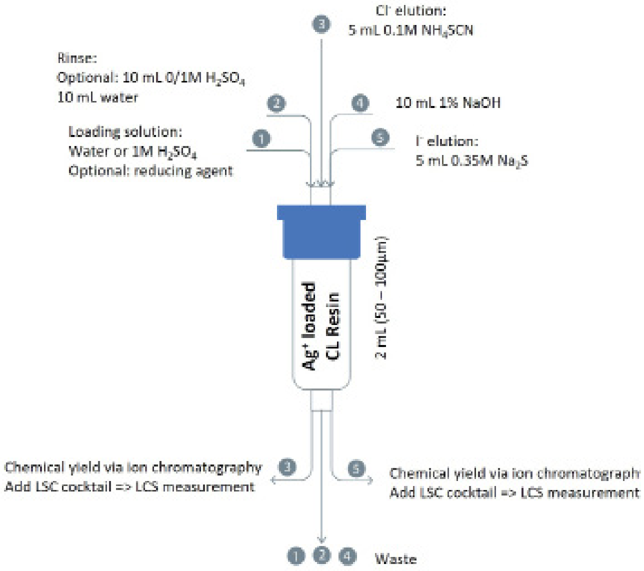
This allowed Zulauf et al. developing a simple scheme for the separation of chloride and iodide. The small elution volumes (5mL) used for elution allow for direct measurement of the obtained fractions by liquid scintillation counting.

Warwick et al. developed a method for the analysis of decommissioning samples (e.g. spent resin) based on the thermal decomposition of the sample to be analysed e.g. using a Raddec 'Pyrolyser'. Volatilized chlorine species are transported by a stream of moistened air into a bubbler containing a 6mM Na_2CO_3 solution where they are trapped. The authors modified the separation procedure so that the bubbler solution could then directly be loaded onto a Ag^+ loaded CL Resin column.

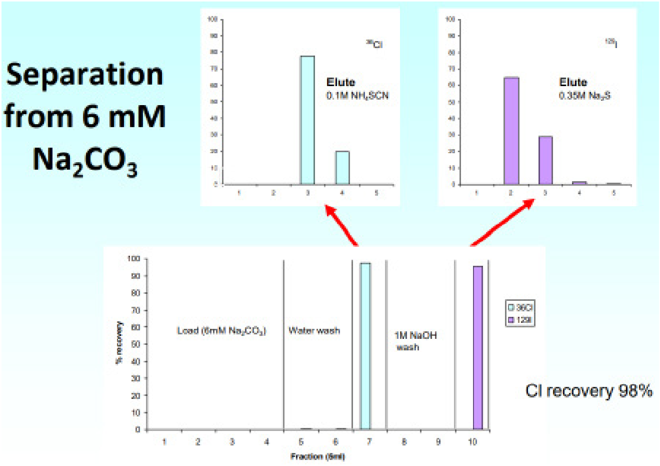
It was observed that, since the sample was not loaded from a highly acidic sample solution, an additional rinsing step consisting of 5mL 0.1M H_2SO_4 was necessary in order to improve C-14 decontamination.

Nottoli et al. employed the CL Resin for the determination of I-129 in spent resins by AMS. The authors first mineralized the resin via microwave digestion or by oxygen bomb combustion. Iodine was then purified on CL Resin using a modified purification method. Samples were prepared for AMS measurement by oxidation of the sulphide to sulphate with H_2O_2, removal of the sulphate by precipitation with Ba followed by centrifugation, and finally a AgI precipitation.

With respect to its high selectivity for noble metals the CL Resin is currently being evaluated for use in Ag and Pd separation and determination.



Scheme of chloride / iodide separation



Chloride / iodide separation according to Warwick et al.

Main applications

- Cl-36 and I-129 in water, environmental and decommissionning samples

CU Resin

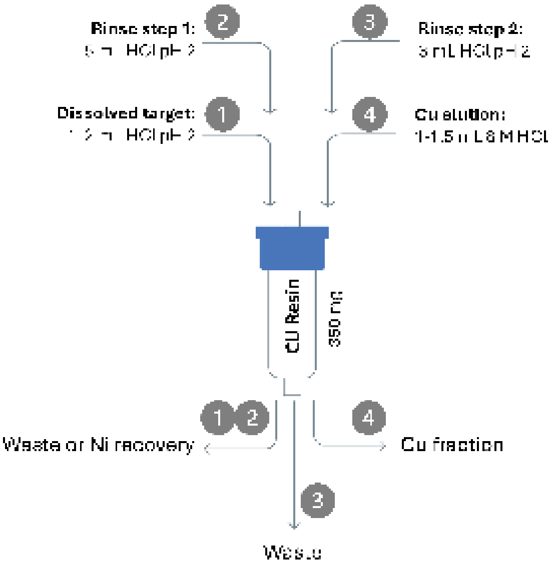
The CU Resin is used for the separation of Cu and is based on a Cu selective extraction system. The extraction behaviour of the CU Resin towards a number of elements in three different acids at varying acid concentrations is presented in the following figures.

Overall the resin shows high selectivity for Cu over all tested cations including Ni and Zn. Cu uptake is generally high at pH values greater than 2 while it can be easily eluted with mineral acids of elevated concentrations.

Further the resin shows high robustness against interference by elevated amounts of Zn and Ni, even at 1g of Ni or Zn per g of CU Resin employed the D_w(Cu) remains greater than 1000.

A simple and fast method for the separation of Cu from irradiated targets was developed by Dirks. et al. allowing to obtain highly pure Cu in a very small volume (1 – 1.5mL) in less then 10min using a vacuum assisted separation setup.

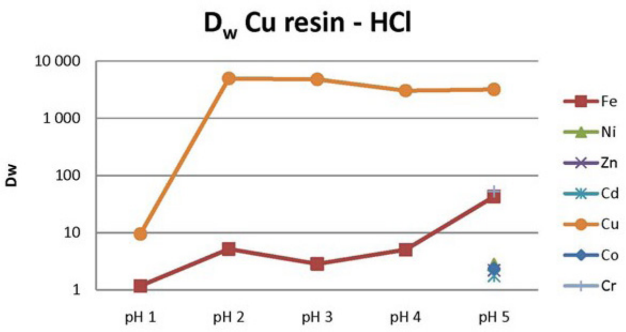
It further could be shown that Cu can be concentrated and separated from other high-matrix samples such as sea water.



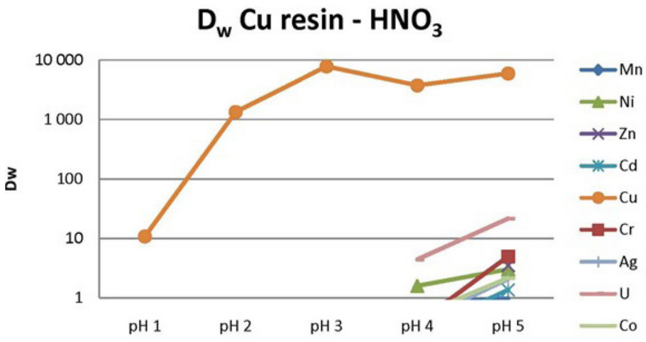
Optimized Cu separation method

Main applications

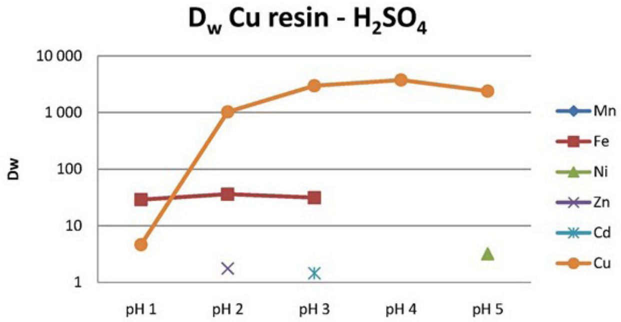
- Separation of Cu-64/67 from irradiated targets
- Concentration and separation of Cu from environmental matrices
- Purification of target materials



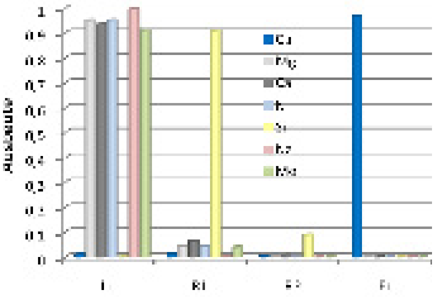
D_W of Cu and selected elements on CU Resin in HCl in varying pH values



D_W of Cu and selected elements on CU Resin in HNO_3 in varying pH values



D_W of Cu and selected elements on CU Resin in H_2SO_4 in varying pH values



L1 = 10 mL Cu spiked sea water sample

R1 = 5 mL HCl pH 2

R2 = 3 mL HCl pH 2

E1=1mL HCl 8 M

Elution study Cu spiked sea water sample

CU iSheets

CU iSheets are comprised of iTLC-SG TLC (Thin Layer Chromatography) paper (Agilent) impregnated with the same Cu selective extractant that is also employed in the CU Resin.

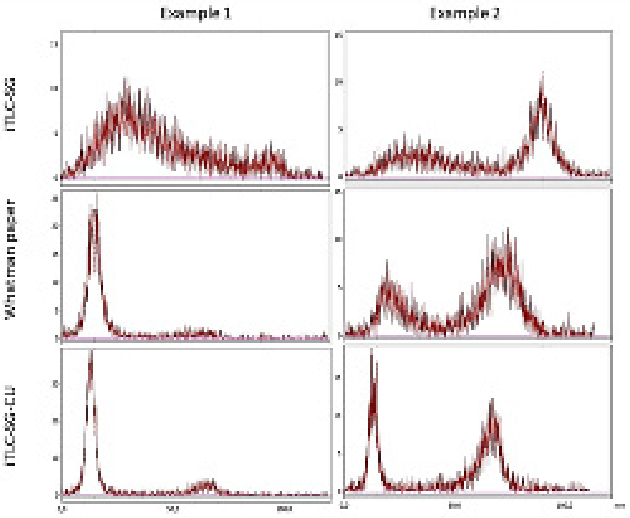
TLC papers are frequently used in the quality control (determination of radiochemical purity) of labeled compounds for use in radiopharmacy. In some cases, for example when analyzing Cu-labelled peptides, artefacts can form during such a TLC test when using silica gel based supports. These artefacts can then interfere with the analysis of TLC scans by creating or distorting peaks. While switching to non-silica based TLC supports (e.g. Whatman paper) generally leads to an improvement, this comes with the disadvantage of significantly longer development times and broad peaks.

Svedjehed and Gagnon could show that using CU iSheets a significantly better resolution with short development times can be obtained for Cu labelled peptides.

The authors produced ^{61}Cu via the ^{nat}Ni(d,x)^{61}Cu reaction using a GE PETtrace solid target system and purified the [^{61}Cu]CuCl$_2$ using a TBP/TK201 Resin based method as described bt Gagnon et al.

Aliquots of [^{61}Cu]CuCl$_2$ were then incubated (90°C; 30min; pH 4.4 [0.3M acetate buffer]) with varying low concentrations of NOTA-octreotide trifluoroacetate or NODAGA-RGD trifluoroacetate. In all cases the ligand concentration was kept below excess to ensure incomplete labelling and thus presence of non-labeled ^{61}Cu.

5µL of the respective ^{61}Cu-labelled peptide were then spotted with origin at 1cm onto strips of 10cm length of: a.) iTLC-SG, b.) Whatman paper (both non-impregnated) and c.) CU iSheets, and developed to at least 7cm in 1:1 MeOH/1M ammonium acetate. The following fig. shows the result of TLC scans of [^{61}Cu]Cu-NOTA-octreotide with elevated levels of free ^{61}Cu (example 1) and [^{61}Cu]Cu-NOTA-octreotide scans at comparable levels of labelled and unlabelled ^{61}Cu (example 2). The authors reported that similar results were obtained in case of [^{61}Cu]Cu-NODAGA-RGD.



TLC scans of [^{61}Cu]Cu-NOTA-octreotide spotted on:
top: iTLC-SG; middle: Whatman paper; bottom: CU iSheets.
Example 1 notes elevated levels of unlabelled ^{61}Cu, while example 2 notes comparable levels of labelled to unlabelled ^{61}Cu (data provided by Svedjehed et al.)

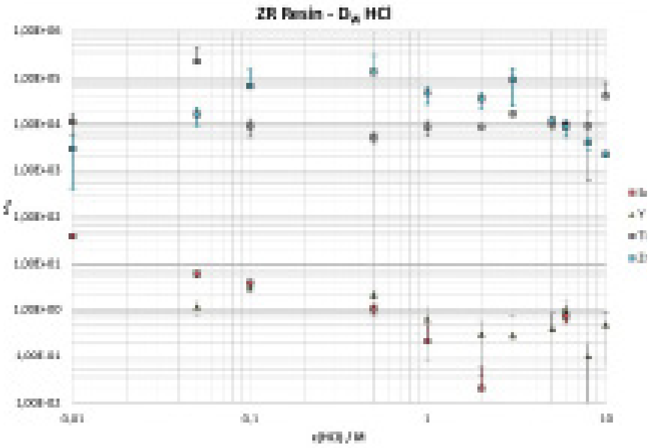
The scans show that in this case using non-impregnated iTLC-SG and Whatman papers results in wide, non-uniform peaks of low resolution. TLC scans using CU iSheets show significantly sharper, separated peaks with the added benefit of short development time compared to the Whatman paper (<10min instead of ~25-30min for the Whatman paper). The authors report even greater differences when decreasing the amount of peptide.

Main applications

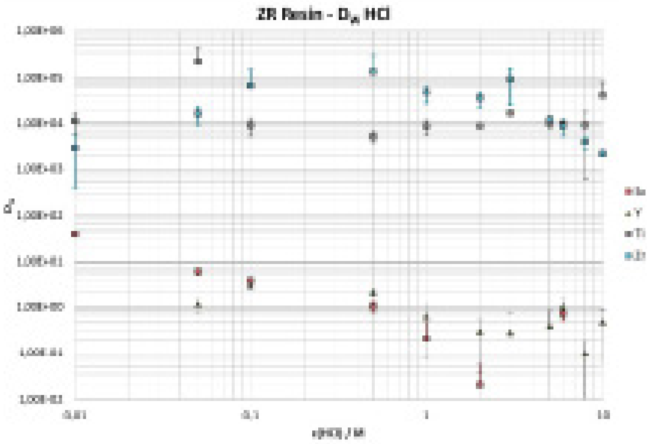
- Quality control of Cu labelled compounds via TLC

ZR Resin

The ZR Resin is based on the hydroxamate functionality frequently used for the separation of zirconium, especially from Y target materials, for later use in radiopharmaceutical applications.
Dirks et al. characterized the resin with respect to its selectivity for selected elements in HNO_3, HCl and oxalic acid; results are summarized in the following figures.



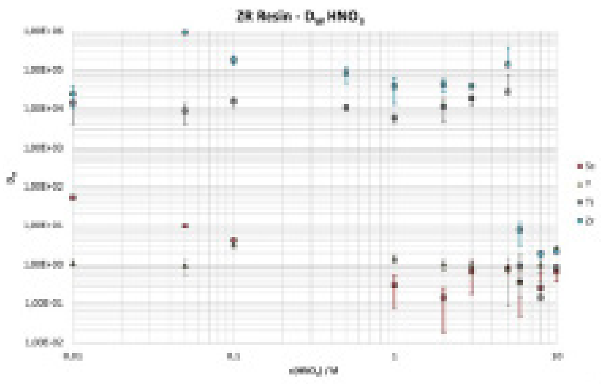
D_W values, ZR Resin, HCl, various elements



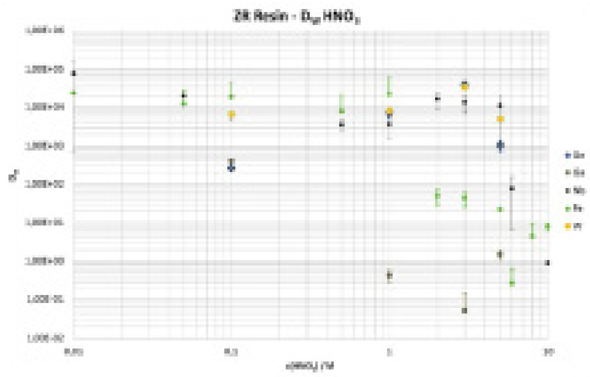
D_W values, ZR Resin, HCl, various elements

The ZR Resin shows high selectivity for Zr, Ti and Nb over a wide range of HCl concentrations (0.01M – 10M), Fe(III) is strongly retained at low and elevated HCl concentrations, retention is weak from 1 – 6M HCl. As expected, the resin shows very little selectivity for Sc and Y, a separation e.g. of Zr from Y and of Ti from Sc is thus feasible.

The resin further shows quite interesting selectivity with respect to Ga and Ge.
Ga is very well retained at low HCl concentrations (≤0.1M) as well as at high concentrations (≥5M HCl), while Zn e.g. is not retained at all. At HCl concentrations in-between, particularly at 1-2M HCl it is not retained. Ge on the other hand is very well retained at HCl concentrations >0.1M.

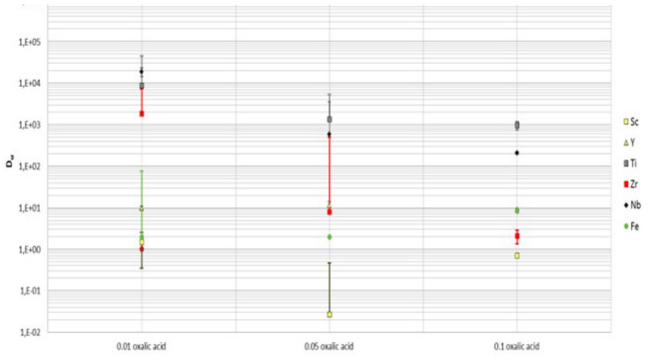


D_W values, ZR Resin, HNO_3, various elements



D_W values, ZR Resin, HNO_3, various elements

Especially at 2M HCl the selectivity for Ge over Ga is very high.
The resin shows rather similar selectivity in HNO_3. Zr, Ti and Nb are well retained up to 5M HNO_3, Fe(III) is well retained up to 1M HNO_3. At higher HNO_3 concentrations the nitric acid starts decomposing the extractant, as indicated by a colour change of the resin from white to brown; accordingly, the resin shows no significant selectivity towards the tested cations under these conditions. As in HCl, Y and Sc show no significant retention on the ZR Resin in HNO_3.



D_W values, ZR Resin, HNO_3, various elements

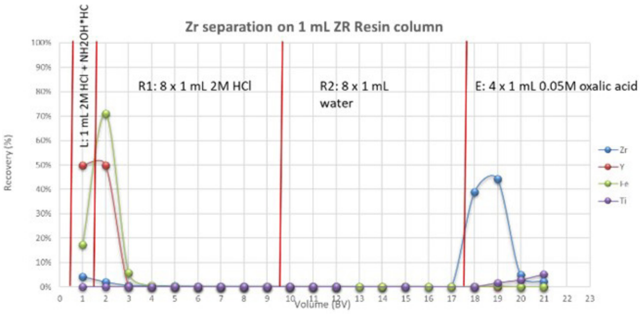
It should be noted that the ZR Resin also shows high selectivity for Ge over Ga (and Ni/Co) at elevated HNO_3 concentrations.

Oxalates are very strong complexing agents for Zr, accordingly they are very frequently used for the elution of Zr.

It could be shown that oxalic acid concentrations above 0.05M lower the D_w value of Zr on the ZR Resin strongly; they are thus suitable eluting agents for Zr. It was further observed that Nb shows rather elevated D_w values even at 0.05M oxalic acid, indicating that Zr and Nb may be separated by adjusting the oxalic acid concentration accordingly.

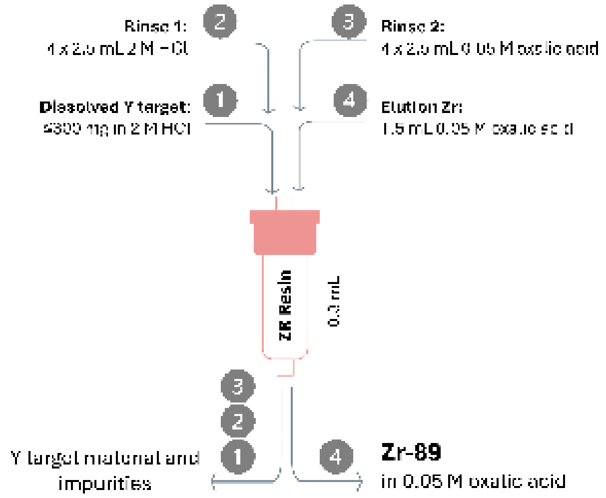
Based on obtained D_w values several elution studies were performed with main focus on the use of the resin in the context of radionuclide production for radiopharmaceutical use.

As indicated by the D_w values, the ZR Resin will retain Zr over a range of HCl concentrations. The rinsing conditions were kept close to the conditions suggested by Holland et al.: after loading the resin it is first rinsed with 4 x 2.5mL 2M HCl, followed by an additional rinse with 4 x 2.5mL water. Zr is finally eluted using 0.05M oxalic acid or higher.



Elution study ZR Resin, 100mg, various elements, fractions analysed by ICP-MS

Under the given conditions a very clean separation of Zr from Y, Ti and Fe was obtained. Y and Fe are removed during the loading and rinsing of the ZR Resin, while Ti remains retained on the resin. Zr can be recovered quantitatively in ~2 bed volumes (BV) of 0.05M oxalic acid. High chemical yields could be obtained even in presence of up to 300mg stable Y (using 100mg ZR Resin).



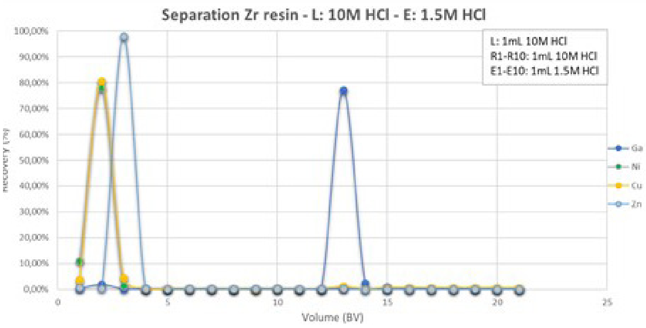
Suggested method for the separation of Zr from Y targets (≤ 300mg) using the ZR Resin.

The ZR Resin is currently also being tested for use in radioanalytical applications such as the quantification of Zr-93, as well as, in combination with the TK400 Resin, the separation of Fe/Nb/Mo, e.g. in decommissioning samples.

Another increasingly important use of the ZR Resin is the purification of Ga-68 from irradiated Zn targets. The production of Ga-68 via the irradiation of Zn-68 (as liquid or solid targets) on a cyclotron is indeed increasingly finding use as an alternative to Ge-68 generator produced Ga-68, as it allows frequent production of high activities.

As shown in the D_w value graphs the ZR Resin retains Ga very strongly in dilute HCl and HNO_3, as well as in HCl of elevated concentration (≥ 5M HCl). Zn on the other hand is not retained under any of these conditions. This selectivity allows its use for the separation of Ga-68 (and Ga-67) from irradiated Zn targets - liquid targets (typically dilute HNO_3) as well as solid targets (typically dissolved in HCl of high concentration).
HCl between 1M and 2M on the other hand is very suitable for Ga elution as its retention is particularly low under these conditions.

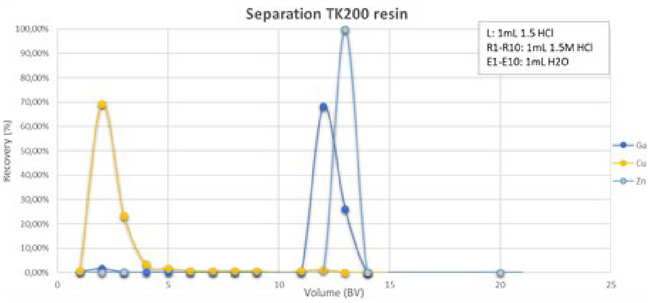
The following graph shows a typical example of such a separation. While Zn, and other typical impurities such as Cu and Ni, are not retained on the ZR Resin (in this example from high HCl) Ga is very well retained. A clean Ga fraction is then obtained by elution of Ga e.g. with 1.5M HCl.



Elution study, Ga separation on ZR Resin, various elements, fractions analysed by ICP-MS

This Ga-68 containing 1.5M HCl solution is too acidic for direct use in labelling or injection. Instead of converting the solution to more suitable conditions e.g. via evaporation and redissolution it is possible to use another resin for this step, the TK200 Resin. More information on the TK200 Resin selectivity may be found in its product sheet, in this context the most important fact is that it retains Ga well in the range of 1 – 2M HCl, while it allows for Ga elution in dilute HCl or water, making it very suitable for this required conversion.

It should be noted though that while other impurities like Cu are very well removed during load and rinse on the TK200, remaining traces of Zn are only partially removed, accordingly a clean separation on the ZR Resin upfront is very important, as can also be seen in the following elution study.



Elution study, Ga conversion on TK200, various elements, fractions analysed by ICP-MS

Several publications describe the use of the combination of ZR Resin and TK200 for the separation of Ga-68 from liquid Zn targets. The publication by Rodnick et al. is particularly interesting as it describes the use of a modified rinse on the TK200 cartridge (2M NaCl/0.13M HCl) instead of the usual rinse with HCl. This allows for recovering the final Ga fraction at a better-defined HCl concentration during elution. The following scheme shows the separation method they developed.

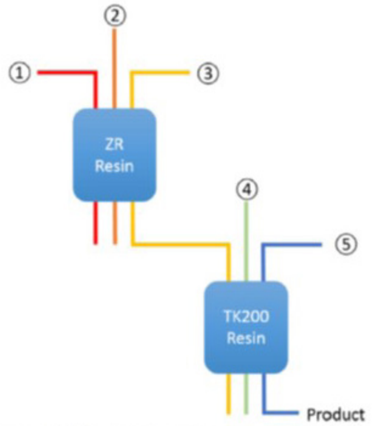


Fig. 4 Two-column approach for ^{68}Ga chemical separation

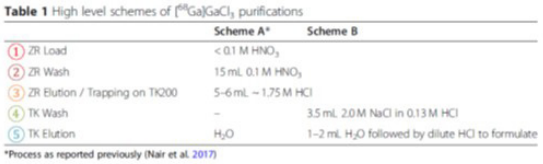


Table 1 High level schemes of [^{68}Ga]GaCl$_3$ purifications

	Scheme A*	Scheme B
① ZR Load	< 0.1 M HNO$_3$	
② ZR Wash	15 mL 0.1 M HNO$_3$	
③ ZR Elution / Trapping on TK200	5–6 mL ~ 1.75 M HCl	
④ TK Wash	–	3.5 mL 2.0 M NaCl in 0.13 M HCl
⑤ TK Elution	H$_2$O	1–2 mL H$_2$O followed by dilute HCl to formulate

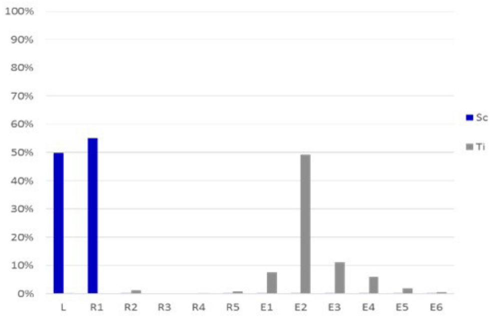
*Process as reported previously (Nair et al. 2017)

Scheme of Ga-68 separation from liquid Zn targets using ZR Resin and TK200 Resin, taken from Rodnick et al.

Compared to liquid targets the irradiation of solid Zn targets allows for obtaining higher Ga-68 activities per production run.

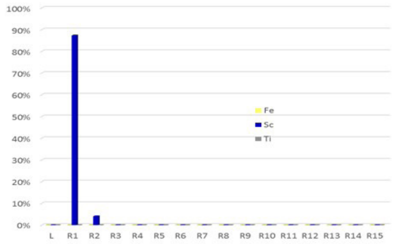
Thisgaard et al. describe the production of 194GBq Ga-68 (at end of purification) as [^{68}Ga]GaCl$_3$ of high purity, and it's subsequent successful use for the labelling of PSMA-11 and DOATATE. The authors used a three-resin method for the separation. An additional LN Resin cartridge is used between the ZR Resin and the TK200 Resin to further remove potentially present impurities, particularly Fe.

The ZR Resin further shows very interesting selectivity for Ti, especially with respect to Sc.
The graph below shows an example of a Ti/Sc separation performed on the ZR Resin, while Sc is not retained from 10M HCl Ti is fixed very well. 0.1M citric acid may then be used to elute Ti from the resin. However, the elution requires up to 10 bed volumes (BV) of the eluent or more. Beside citric acid, hydrogen peroxide or oxalic acid of elevated concentration may also be employed.



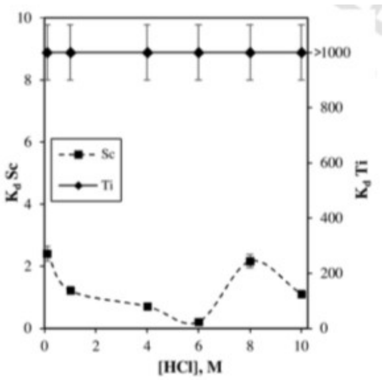
Ti/Sc separation on ZR Resin (0.3mL), fractions analysed by ICP-MS

As Ti is retained over a very wide range of HCl concentrations, including dilute HCl, its potential for use as support for a Ti/Sc generator was also evaluated initially. In order to do so a 100mg ZR Resin column (0.3mL) was loaded with a small volume of a solution containing Ti and Sc. The column was then rinsed five times with 1mL 0.01M HCl, followed by 10 rinses with 5mL 0.01M HCl. Sc is easily removed in a small volume of dilute hydrochloric acid whereas Ti remains retained throughout the experiment, the general selectivity of a generator is thus given.



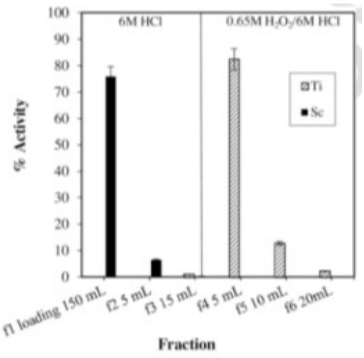
Elution study on 100mg ZR Resin, Ti and Sc, load from 0.01M HCl, repeated elutions, fractions analysed by ICP-MS

Radchenko et al. examined the system in greater detail and using real, irradiated samples. They confirmed the ZR Resins selectivity for Ti over Sc, shown by the K$_d$ values the author obtained.



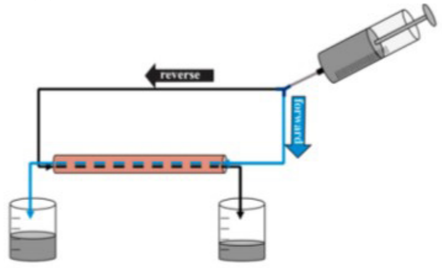
K_d values for Sc and Ti in HCl on ZR Resin, taken from Radchenko et al.

Based on these values they developed a separation method for the purification of Ti, to be more precise Ti-44, from irradiated Sc targets of elevated size (4g). The graph below shows the elution profile they obtained.
As may be seen a clean Ti separation from Sc was obtained. The Ti-44 was obtained as an HCl/H_2O_2 solution.



Ti/Sc elution profile on ZR Resin, irradiated 4g Sc target, taken from Radchenko et al.

The authors used the purified Ti-44 solution for the preparation of two types of Ti-44/Sc-44 generators, one direct flow generator and, the preferred option according to the authors, one ‘forward/reverse flow’ generator.



Schematic of a forward/reverse flow radionuclide generator, taken from Radchenko et al.

Especially the ‘forward/reverse flow’ generator showed very promising results with stable very low Ti breakthrough and high Sc elution yields. The obtained Sc-44 was successfully used to perform DOTA labelling with high yields, further indicating its high purity.

Malinconico et al. also used the ZR Resin to produce Ti-45 from irradiated Sc-45 targets.

Besides for the purification of Ga-68 from Zn targets the ZR Resin may actually also be used for the separation of Ge-68 from irradiated GaNi or GaCo targets. As discussed before, while Ga is very well retained at low mineral acid concentrations (typically ≤0.1M) and at high HCl concentrations, it is not retained at medium high HCl and HNO_3 concentrations, and medium to high H_2SO_4 concentrations. Ge on the other hand is very well retained at elevated mineral acid concentrations. The D_w values show that especially between 1M – 3M HCl and HNO_3 the Ge retention is significantly higher than Ga retention.
It could be shown that the same is true for e.g. 5.5M H_2SO_4. The ZR Resin further shows no selectivity for Ni or Co under these conditions.

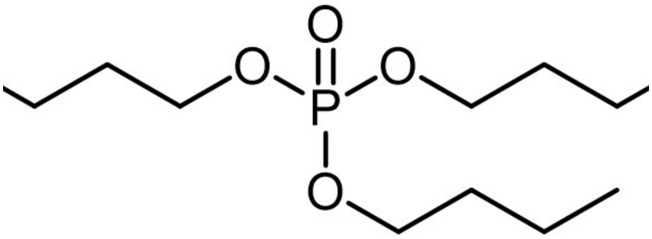
While the selectivity for Ge over Ga is very high in HCl its use for Ge separations is often avoided due to the high volatility of $GeCl_4$. In H_2SO_4 on the other hand Ge is not volatile, it further allows an efficient dissolution of typically employed target materials. Accordingly, a method for the separation of Ge-68 from multi gram irradiated GaNi or GaCo targets is currently being optimized.

The method is based on two subsequent purification steps on ZR Resin. First the dissolved target is adjusted to 5.5M H_2SO_4 and then loaded onto a 2mL ZR Resin cartridge. After rinsing with 5.5M H_2SO_4 and purge with air for acid removal Ge is eluted with dilute citric acid. The Ge fraction is again adjusted to 5M H_2SO_4 and further purified on a 1mL ZR Resin cartridge. Ge is once more recovered in dilute citric acid. In order to obtain the final product in dilute HCl (typically 0.05M HCl) the Ge is converted from citric acid to dilute HCl by adjusting it to 9M HCl or higher, loading e.g onto a Guard Resin cartridge, followed by elution with water or dilute acid. Further optimisation of the method is currently on-going.

Main applications

- Separation of zirconium
- Separation of gallium
- Separation of germanium
- Separation of titanium

TBP Resin



TriButylPhosphate (TBP)

The TBP Resin is comprised of an inert support impregnated with Tributylphosphate (TBP). TBP is a widely used extractant, it finds for exemple application in the Purex process, the reprocessing of U and Pu from spent fuel. Other applications include, amongst others, the separation of yttrium for analytical purpose.

The following figure shows the D_w values of selected actinides on the TBP Resin in HNO_3 and HCl.

Vajda et al. also evaluated the influence of Fe(III) and three anionic interferences (oxalic, sulfuric and phosphoric acid) on the U and Pu uptake.

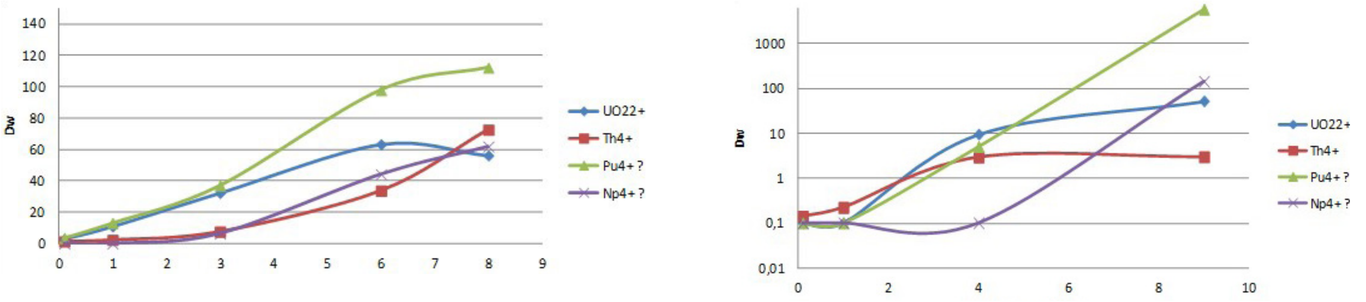
In 8M HNO_3 a Fe concentration of 0.1M is not interfering with the uptake of U(VI) and Pu(IV), the same is true for Np(IV) and Th(IV). Unlike this, the same Fe concentration very strongly interferes with the retention of U and Pu in 9M HCl.

Oxalates show no interference with the U uptake even at concentrations of up to 0.5M. Sulfate, and especially phosphate, interfere significantly when present in elevated amounts. In general the actinides show an increasing retention at increasing acid concentrations.

However, of all actinides only Pu shows D_w values higher than 100 in HNO_3, whereas the other actinides peak at D_w values between 60 and 80. Pu is even stronger retained at high HCl concentrations (D_W > 1000 on 9M HCl), retention of the other actinides, with exception of Np in 9M HCl (D_W=140) is rather low, especially Th(IV) shows very little affinity to the TBP Resin.

Nora Vajda et al. could show that the TBP Resin can be used for the separation of Pu from other actinides and developed a method for its use for the determination of Pu in water samples.

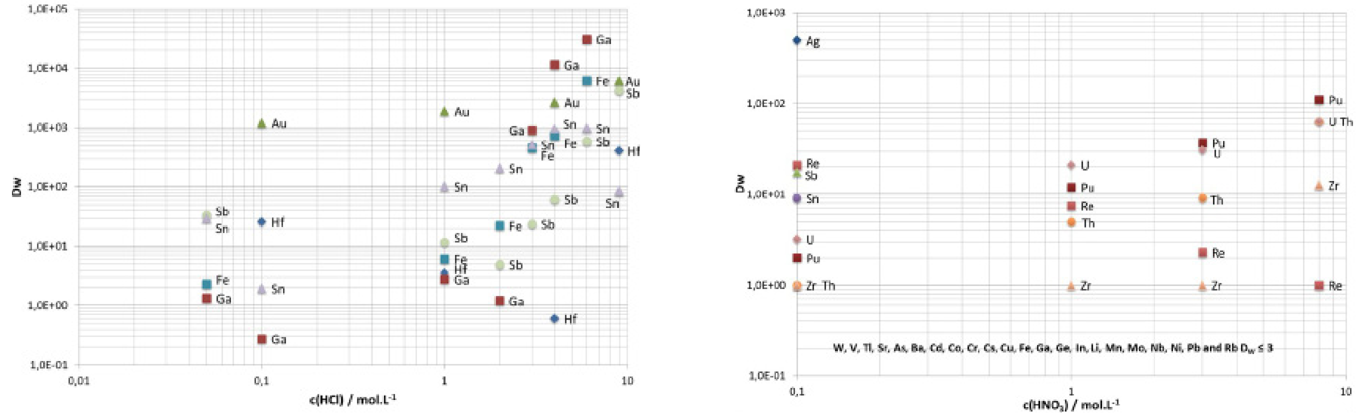
The Pu(IV) extraction from 9M HCl is very robust, although the tested anions do interfere with its uptake when present in concentrations ≥ 0.1M, the D_w values of Pu still remain greater than 500.



HNO_3 concentration M

HCl concentration M

D_W values of selected actinides in HNO_3 and HCl media on TBP Resin



D_W values of selected elements on TBP Resin in HCl and HNO_3 .

The TBP Resin has further been characterized with respect to the uptake of various elements in HNO_3 and HCl, the results are summarized in the two figures.

Beside Pu(IV) and Np(IV) several other elements such as Au, Hf, Fe, Sn and Ga also show high affinity for the TBP resin in HCl.

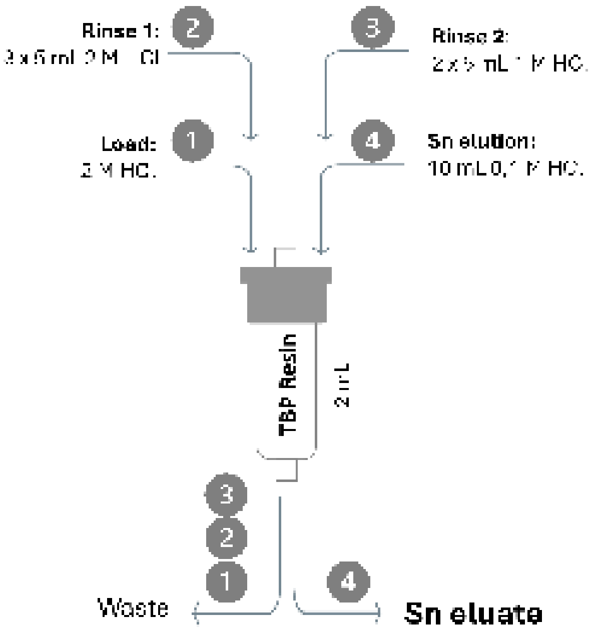
While Au remains retained under all tested conditions, making its elution rather difficult in HCl, the other elements only show high D_w values at elevated acid concentrations, and low D_w values at lower concentrations.

In 1M HCl for example only Sn shows elevated D_w values whereas Fe, Ga, Sb... show very little affinity to the resin, allowing for its separation from these elements. Sn can then be eluted e.g. with 0.1M HCl.

The TBP Resin generally shows very good selectivity for Sn over Te (Te-126 is an isobaric interferences for the mass spectrometric determination of Sn-126, a long-lived beta emitter frequently determined in decommissioning and radioactive waste samples) and Cd which is frequently used as target material for the production of Sn-117m, a conversion electron emitter used in nuclear medicine. The resin also shows interesting selectivity for Sb, however its oxidation state needs to be carefully controlled.

In HNO_3 of all tested elements only the actinides (at elevated HNO_3 concentrations), and Ag (at low HNO_3 concentrations), are retained on the resin. Based on the obtained data, Dirks et al. developed a procedure for the separation of Sn from various elements, the figure on the right shows a scheme of the suggested method using a 2mL TBP column.

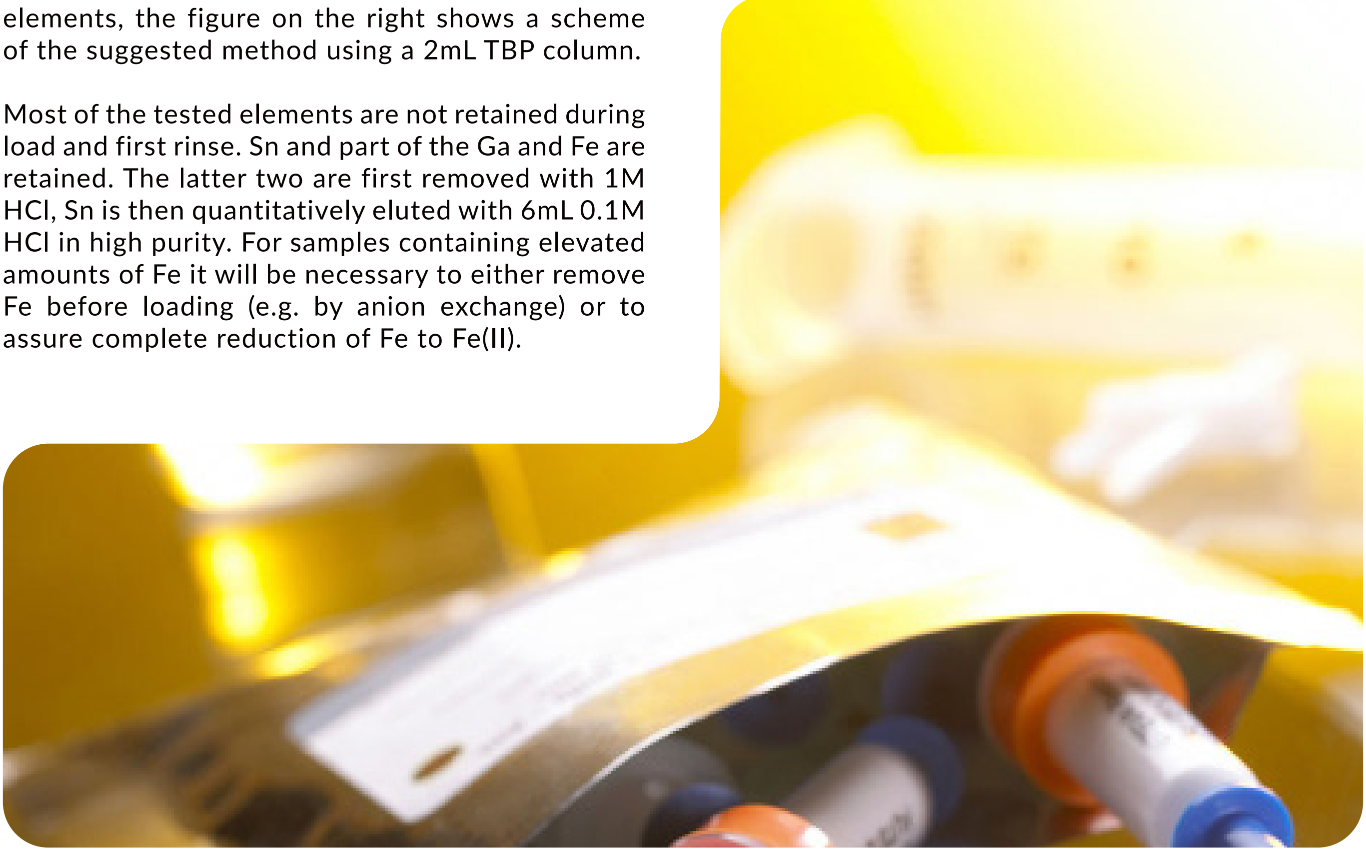
Most of the tested elements are not retained during load and first rinse. Sn and part of the Ga and Fe are retained. The latter two are first removed with 1M HCl, Sn is then quantitatively eluted with 6mL 0.1M HCl in high purity. For samples containing elevated amounts of Fe it will be necessary to either remove Fe before loading (e.g. by anion exchange) or to assure complete reduction of Fe to Fe(II).



Separation scheme Sn separation on TBP Resin.

Main applications

- Separation of tin
- Separation of zirconium
- Separation of scandium
- Separation of actinides



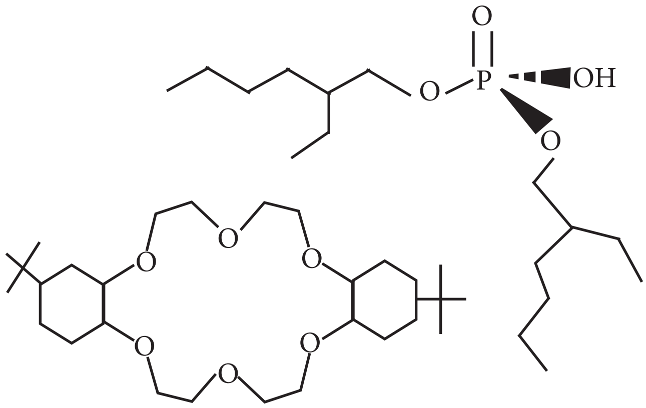
TK100 Resin & TK101 Resin

Crown-ether based extraction chromatographic resins such as the SR and PB Resins are frequently used for the separation and determination of Sr-90 and Pb-210 in aqueous samples via liquid scintillation (LSC) or gas proportional counting (GPC).

These resins only show significant Sr and Pb retention at moderate to high acid concentrations; thus not allowing for direct loading of the analytes from filtered raw or acidified water samples.

TK100 Resin

The TK100 Resin consists of a crown-ether with high selectivity for Sr and Pb and HDEHP a liquid cation exchanger .



4,4'(5')-di-t-butylcyclohexano-18-crown-6

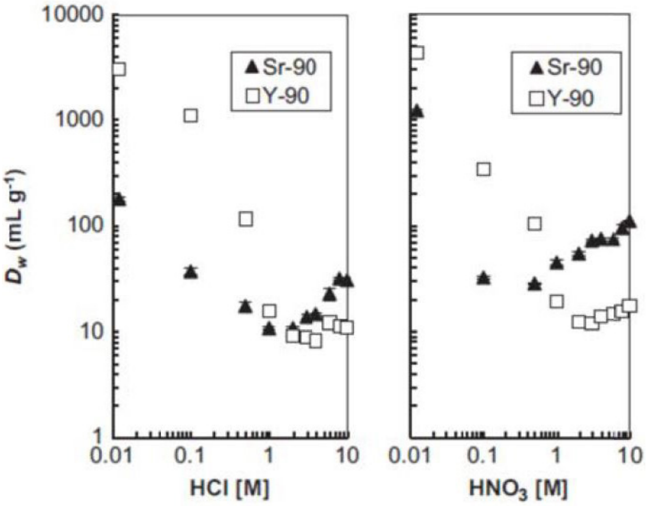
Di(2-ethylhexyl) orthophosphoric acid (HDEHP)

Jake Surman from Lancaster University characterized the resin with respect to D_w values of Sr at different pH values ≥ pH 2, as well as at HNO_3 and HCl concentrations higher than 0.01M. It could be shown that the resin shows high D_w values for Sr at pH values up to 8 (D_W >> 100).

As shown in the following figure, the Sr uptake at pH 2 is high, especially in HNO_3. The latter fact is of importance as water samples are often acidified to pH 2 using HNO_3 directly after sampling in order to stabilize them for storage before analysis.

At elevated HNO_3 concentrations the resin shows an increase of the Sr D_w values to about 100 at 8 – 10M HNO_3. The TK100 Resin is thus behaving very similar to the SR Resin under these conditions. This makes the additional use of pre-concentration steps such as ion exchange or co-precipitation necessary.

In order to simplify the Sr-90 and Pb-210 separation, two new extraction chromatographic resins with an extended uptake pH range were developed, allowing for direct loading of water samples (pH 2 - 8) and the subsequent purification of the analytes on the same column.



D_W values Sr and Y on TK100 Resin, varying HNO_3 and HCl concentrations

As Sr elution with water or dilute nitric acid is not possible, a number of other eluting agents were tested, 2M HCl, 3M HCl and 0.1M EDTA were found to be most suitable of all tested.

Several other elements also show an affinity to the TK100 Resin at pH 7, making separation chemistry necessary in order to obtain a clean Sr fraction.

The influence of several typical matrix elements on its uptake onto the TK100 Resin at pH 7 was tested. Even though high salt contents indeed interfere with Sr uptake, even at NaCl concentrations of 500mM, K concentrations of 400mg/L, Mg concentrations of 1300mg/L and Ca concentrations of 500mg/L the D_w values for Sr remain greater than 100.

Surman et al. could further show through elution studies that the resin allows for the separation of Sr from a number of elements such as Ca, K, Mg.

Even if the limiting concentrations will be lower for combinations of the interferences, the resin seems well suited for surface and especially drinking waters.

Surman et al. could further show through elution studies that the resin allows for the separation of Sr from a number of elements such as Ca, K, Mg, Ba, Co, Am, Cs and Pb. It should be noted that Pb is not eluting under the chosen conditions.

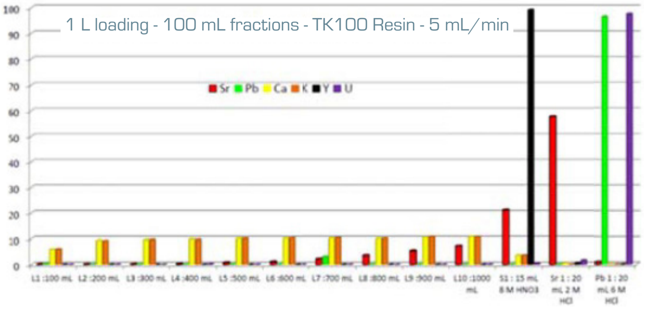
Additional studies performed by Dirks et al. with 1L samples at pH 7 loaded onto a 2mL TK100 column in aliquots of 100mL at a flow rate of 5mL/min showed that K and Ca break through during load without being retained.

Unfortunately Sr breakthrough starts occurring after a loading volume of approx. 600mL indicating that the maximum sample volume to be loaded onto the 2mL column for Sr analysis is ~500mL.

Dirks et al. could indeed show that Sr can be separated with high yields from 250mL (95.2% +/- 2.5%, N=3) – 500 mL (88.2 +/- 4.3, N=3) water samples with high yields even at sample loading flow rates of 5 – 10mL/min. Y, Pb and U on the other hand are very well retained, even when a sample of 1L is loaded. Y can be quantitatively removed using 8M HNO_3. Pb and U remain retained even after the Sr elution step and can be eluted using 6M HCl e.g. for Pb-210 determination via α/β discrimination LSC.

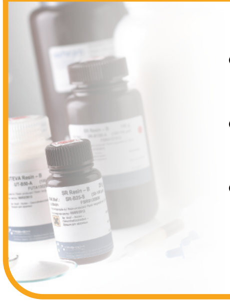
Van Es et al. could show that TK100 allows retaining Ra from up to 1L groundwater.

Work on the separation of Pb and U on TK100 are ongoing, however, another resin called TK101 allows for facile separation of Pb from other elements.



Elution study, various elements, 1L sample, 100mL aliquots, TK100 Resin

Main applications

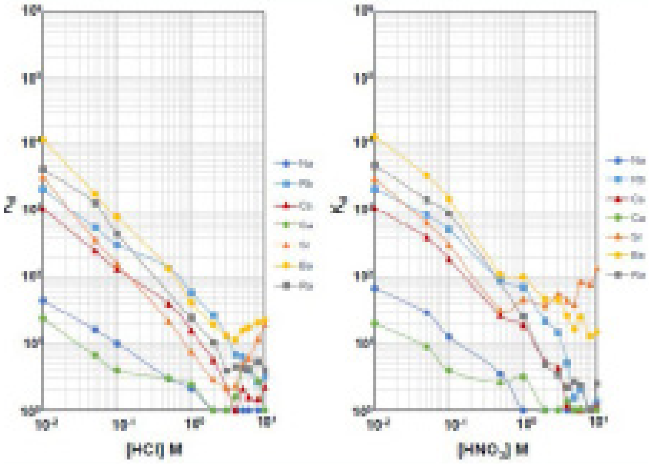


- Direct separation of Ra from water samples
- Direct separation of Pb from water samples
- Direct separation of Sr from water samples

TK101 Resin

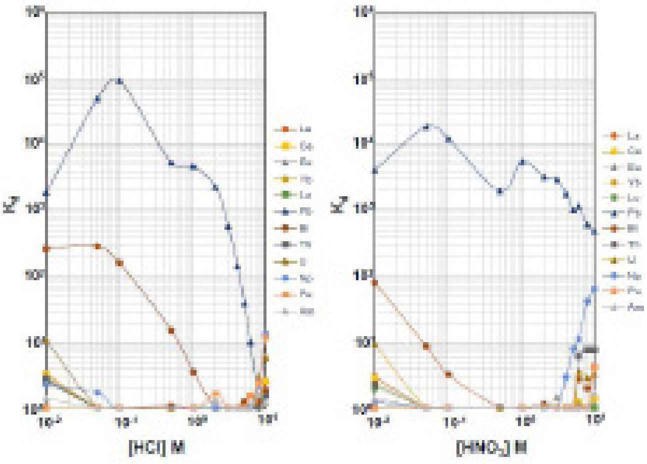
Dietz et al. showed that the mechanism of Sr extraction by crown-ethers in ionic liquids strongly depends on the chain-length of the ionic liquid. Long chained ionic-liquids favor liquid-liquid extraction mechanism with high Sr uptake at high nitric acid concentrations whereas short chained ionic liquids introduce a cation exchange mechanism at low acid concentrations, leading to high Sr retention at elevated pH values. This mechanism further also favors the retention of other divalent cations like Pb, Ba and Ra.

Sr retention then decreases with increasing acid concentrations to a minimum at about 1M HNO_3. At higher HNO_3 concentrations the D_w values increase as expected when the liquid-liquid extraction mechanism becomes predominant.
The following graphs show the selectivity of the TK101 Resin.



D_W values of selected elements on TK101 in HNO_3.
Data courtesy of Ben Russel (NPL)

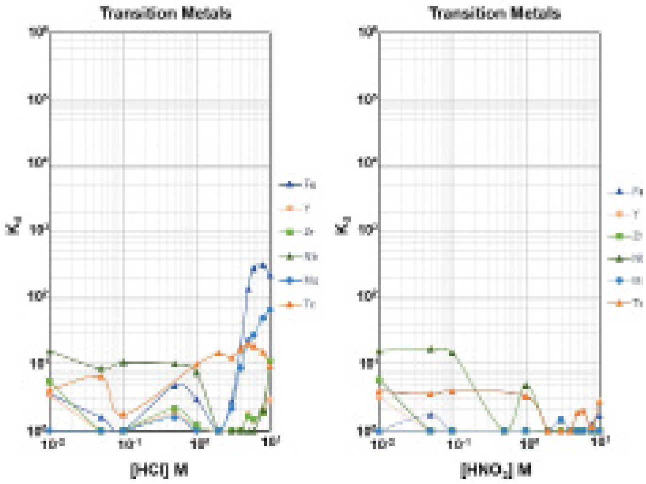
As expected, while SR, PB and TK102 Resins show no selectivity for Sr, Ba, Ra at vey low acid concentrations TK101 does. In dilute HNO_3 as well as in HCl Ba, Ra and Sr are very well retained from ≤0.01M to approx. 0.05M. Their retention decreases significantly as the acid concentration increases though. Sr retention in HNO_3 is an exception as it shows increasing retention at HNO_3 concentrations >3M. It should be noted that, while Ra and Ba behave similar at lower acid concentrations, they do show differences at higher acid concentrations, notably at 3M HNO_3, where the K_d Ba is higher than K_d Ra, thus allowing for a Ra/Ba separation.



D_W values of selected elements on TK101 in HNO_3
Data courtesy of Ben Russel (NPL)

Pb is very well retained over the whole HNO_3 concentration range. It is also very well retained from HCl up until approx. 3M HCl. Its retention then decreases very sharply as the HCl increases, making HCl of high concentration suitable for Pb elution.

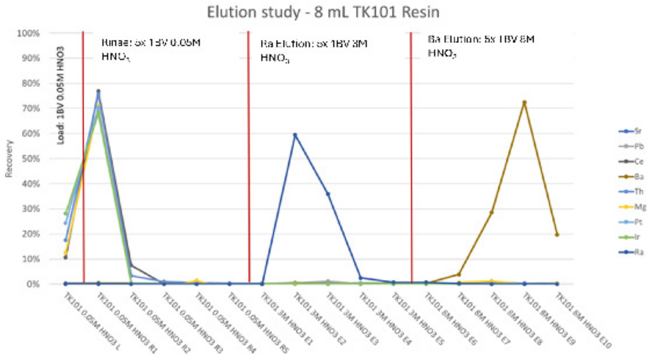
Out of the elements shown in the graph above only Bi is also retained, particularly from dilute HCl at elevated acid concentrations. It can be very easily removed though, especially using HNO_3. Lanthanides and actinides are not retained on TK101 not in HCl neither in HNO_3.



D_W values of selected elements on TK101 in HCl and HNO_3
Data courtesy of Ben Russel (NPL)

Out of the tested transition metals only Nb shows some retention at high HCl, all other elements tested show very low K_d values.

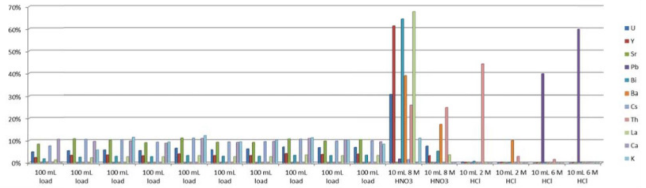
The graph below shows the result of an elution study performed with amongst others Th, Ce, Pt, Ir, Ba, Ra and Pb.



Elution study of selected elements on TK101
Ra data courtesy of Nora Vajda (RadAnal)

Other elements tested than Ba, Ra and Pb are removed during load and rinse with dilute acid (here 0.05M HNO_3). Ra is then eluted with 3M HNO_3, while Ba and Pb remain retained. Ba is eluted with 8M HNO_3. Pb is still retained under these conditions and may then be eluted with 6 – 8M HCl or dilute citrate/citric acid.

An elution study performed by Dirks et al. using the TK101 Resin showed that indeed a clean Pb fraction can be obtained applying a similar separation scheme as employed for the TK100 Resin. High Pb yields are obtained even when loading 1L samples or more at flow rates of 5 – 10mL/min.



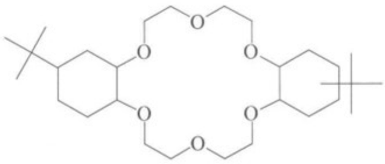
Elution study, 1L sample, 100mL aliquots, TK101 Resin

Main applications

- Direct separation of Pb from water samples
- Ra separation

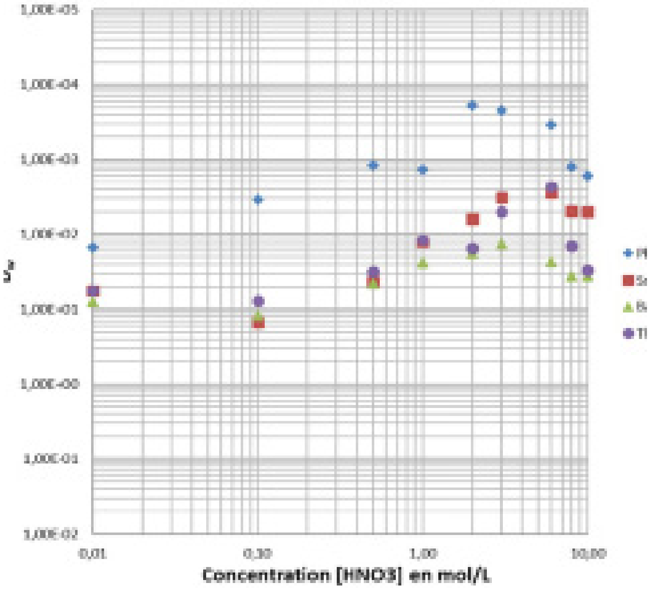
TK102 Resin

The TK102 Resin is based on the same crown-ether that is also used in the SR and PB Resin.



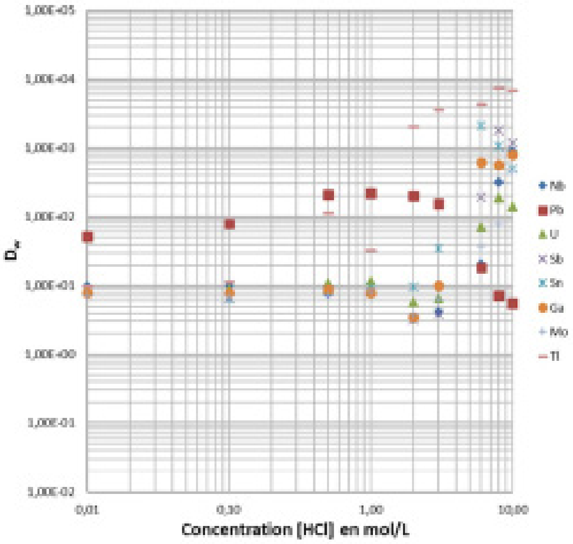
4,4'(5')-di-t-butylcyclohexano-18-crown-6

Other than these resins the TK102 Resin contains a long-chained fluorinated alcohol as diluent. The resin further contains a larger amount of the crown-ether compared e.g. to the SR Resin. Further the organic phase is impregnated onto an inert support containing aromatic groups for increased stability against radiolysis.
The resin was originally optimized for the separation of Ba and Ra, how ever it also shows very interesting properties with respect to Sr and Pb separation.
The following two graphs show the selectivity of the TK102 Resin for a range of elements in HNO_3 and HCl. The third graph shows the influence of increasing amounts of Na, K and Ca on the Sr retention in 3M HNO_3.
All D_w shown in these graphs were obtained through ICP-MS measurements.



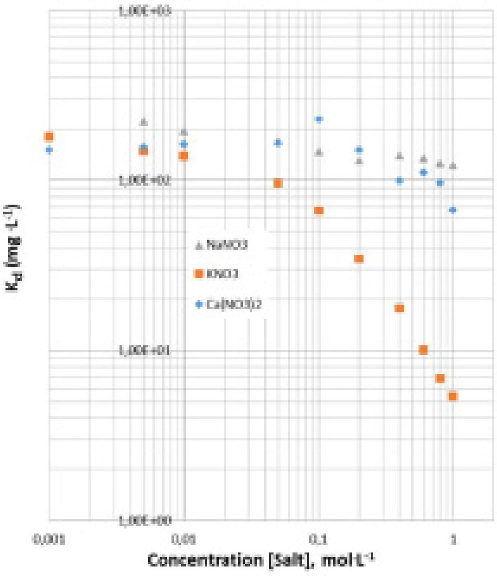
D_W values of selected elements on TK102 in HNO_3

Pb is very well retained over the whole HNO_3 concentration range. Sr is well retained at elevated HNO_3 concentrations (3 – 10M HNO_3), showing higher Sr D_w values than the SR Resin under these conditions. The same is true for Ba at 3M HNO_3, TK102 shows stronger Ba retention than SR Resin Further it is notable that Tl is strongly retained from 3 – 6M HNO_3.



D_W values of selected elements on TK102 in HCl

As expected, Pb is well retained over a wide HCl concentration range, from dilute HCl up to 2 – 3M HCl. Pb D_w values drop strongly for higher HCl concentrations (≥ 6M HCl), allowing for its elution under these conditions.
The TK102 Resin retains, to a certain extent similar to the TK400 Resin, a number of elements at very elevated HCl concentrations, including Tl, Sb, Sn, Ga and Nb.



D_W values Sr on TK102 in 3M HNO_3
and in presence of increasing amounts of Na, K and Ca

Na shows very little influence on the Sr retention on the TK102 Resin, even at concentrations up to 1M D_w values for Sr remain high.

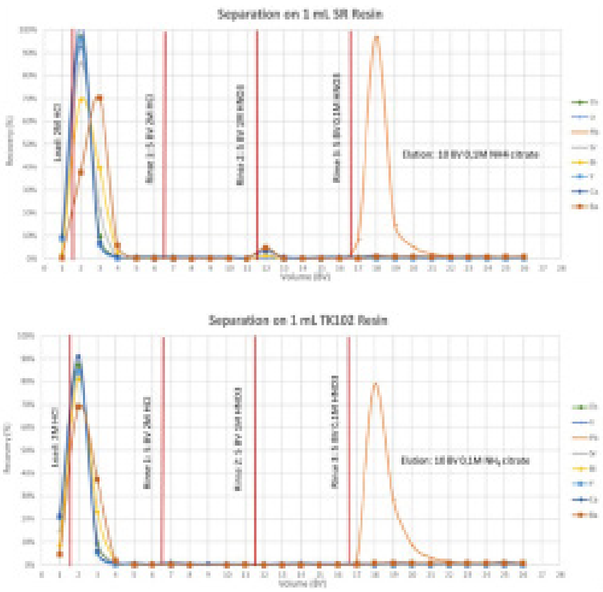
Ca is showing a higher impact, nevertheless even at concentrations up to 0.5M Sr shows elevated D_w values.
As expected, K is interfering with the Sr retention very strongly, even concentrations ≥0.05M will lead to a significant decrease in Sr retention.

Just like for the SR Resin, performing a co-precipitation (e.g. with calcium phosphate) to remove K before the actual separation on TK102 Resin is crucial.

The following figures are showing three comparative elution studies on TK102 Resin and SR Resin.

The first example is a typical Pb separation based on loading from 2M HCl, Po removal with dilute HNO_3 and finally Pb elution with citrate.

Both resins are showing very similar elution profiles, TK102 Resin might require a slightly larger elution volume for Pb though. Nevertheless, typically employed elution volumes (e.g. 10mL) should assure quantitative elution of Pb also from TK102.


Separation on 1 mL SR Resin

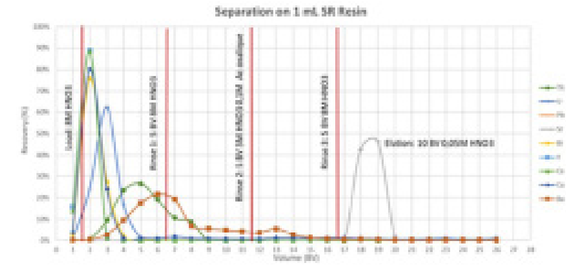
Separation on 1 mL TK102 Resin

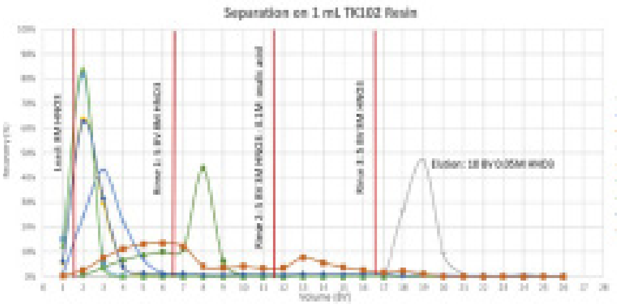
Comparative elution studies, SR and TK102 Resin, Pb separation

The second example is a typical Sr separation based on loading from 3M HNO_3, rinsing with 8M HNO_3 and 3M HNO_3/0.1M oxalic acid, and finally Sr elution in 0.05M HNO_3.

Again, both resins are showing similar elution profiles. One distinct difference being Th, for the TK102 Resin 3M HNO_3/0.1M oxalic acid rinse is required to remove most of the Th while on SR Resin the majority is already removed with 8M HNO_3.

Like for the Pb separation Sr elution from TK102 seems to require slightly larger volumes, but here too typically employed elution volumes (10 – 15 mL) seem to assure quantitative Sr elution.


Separation on 1 mL SR Resin

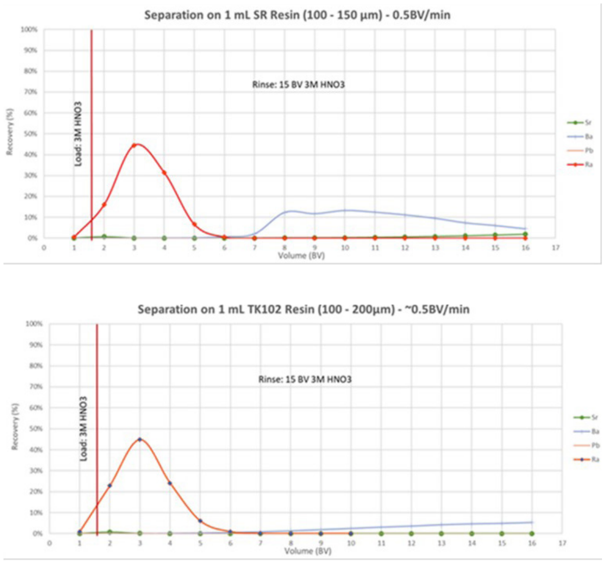

Separation on 1 mL TK102 Resin

Comparative elution studies, SR and TK102 Resin, Sr separation

The third example shows a comparative Ba/Ra separation elution study. TK102 and SR Resin were both loaded from 3M HNO_3, then both resins were rinsed with several bed volumes (BV) of 3M HNO_3.

For both resins Ra is eluted quickly during load and first rinsing steps, while Ba remains retained.

On the SR Resin Ba starts to significantly break through after 6 BV, on the TK102 Resin the Ba retention is distinctively stronger, it starts to very slowly elute after about 8 - 9BV.


Separation on 1 mL SR Resin (100 - 150 µm) - 0.5BV/min

Separation on 1 mL TK102 Resin (100 - 200µm) - ~0.5BV/min

Comparative elution studies, SR and TK102 Resin, Ba/Ra separation

Further the TK102 Resin shows high dynamic capacity for Sr (>40 mg/g) and Pb (>90 mg/g).

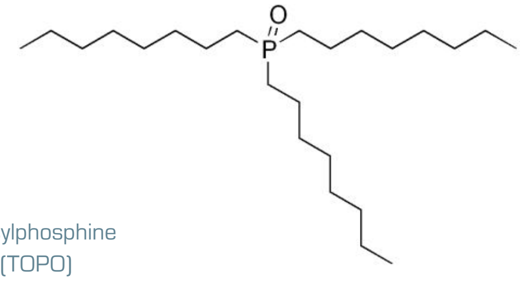
Due to the higher hydrophobicity of the diluent employed in the TK102 Resin it also shows significantly less bleeding of organic material, measured as Non-Purgeable Organic Carbon (NPOC), than the SR Resin.

Main applications

- Ra/Ba separation
- Sr separation
- Pb separation

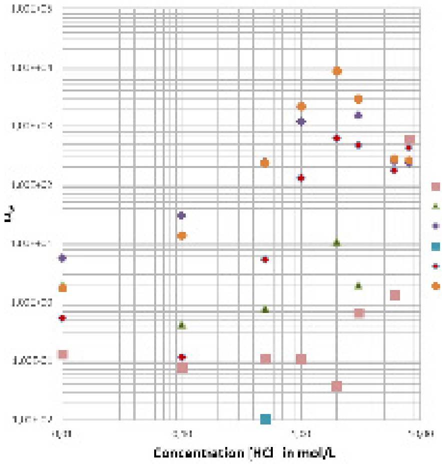
TK200 Resin

The TK200 Resin is based on TriOctylPhosphine Oxide (TOPO) an extractant widely used in the extraction of metal ions.

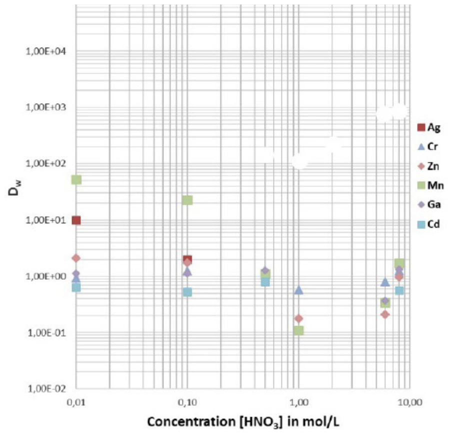


Trioctylphosphine oxide (TOPO)

Some examples of D_w values determined in HNO_3 and HCl using ICP-MS are shown below.



D_W values of selected elements on TK200 Resin in HCl



D_W values of selected elements on TK200 Resin in HNO_3

D_W values for a wide range of additional elements may be found in the corresponding product sheet.

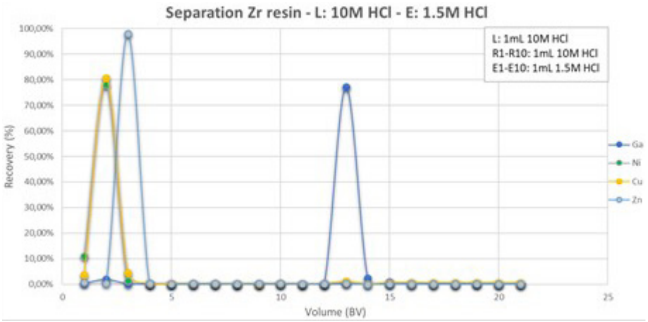
Cd, Zn and Ga are very well retained at HCl concentrations >1M. This is especially interesting with respect to Ga separation chemistry as Ga is not retained at 1 – 2M HCl on most resins.

In HNO_3 of the elements shown here only Ag is retained whereas e.g. Ga and Zn are not.

A typical example of the use of the TK200 Resin is the separation of Gallium isotopes (especially Ga-68) from irradiated Zn targets for medical use in combination with the ZR Resin.

ZR Resin is very well suited for the separation of Ga from Zn matrices, under low acid conditions (e.g. 0.1M HNO_3 often employed for liquid targets) as well as at high acid concentrations (e.g. 10M HCl) conditions often used for the dissolution of solid Zn targets.

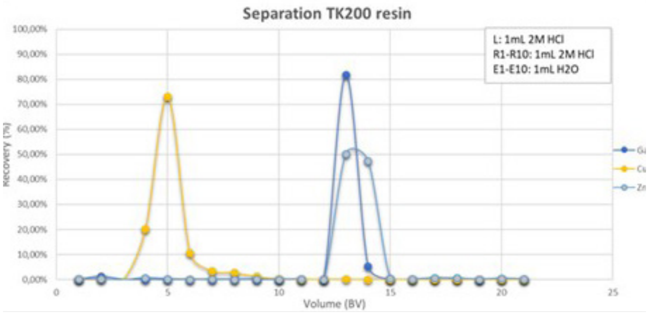
An elution study showing the separation of Ga from Zn and potential impurities on ZR Resin is hereafter:



Ga/Zn separation on ZR Resin - load from 10M HCl

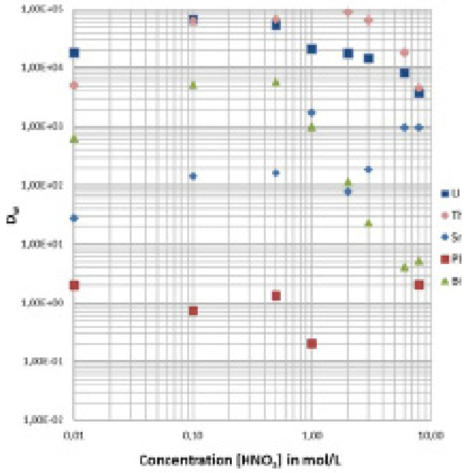
Ga is eluted from the ZR Resin in a small volume (1 – 2 column volumes) of 1.5M HCl, conditions too acidic for direct use in labelling reactions.

The TK200 Resin on the other hand allows for Ga extraction at 1.5M HCl, followed by Ga elution using aqueous solutions.



Ga elution from TK200 Resin with water following load from 1.5M HCl

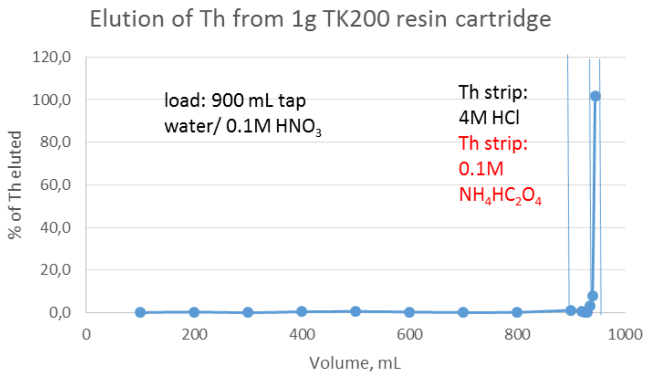
It should be noted though that only very little additional Ga/Zn separation is taking place on the TK200 Resin.



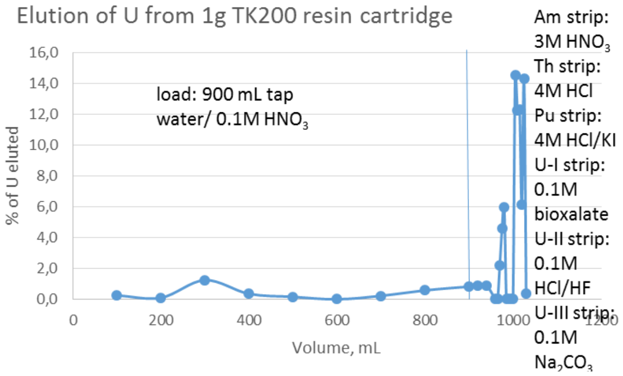
D_W values of selected elements on TK200 Resin in HNO_3

Another typical application of the TK200 Resin is the determination of actinides such as U, Th and Pu in water samples.

As shown in the figure above, U and Th are very well retained over the whole HNO_3 concentration range, including 0.01M.

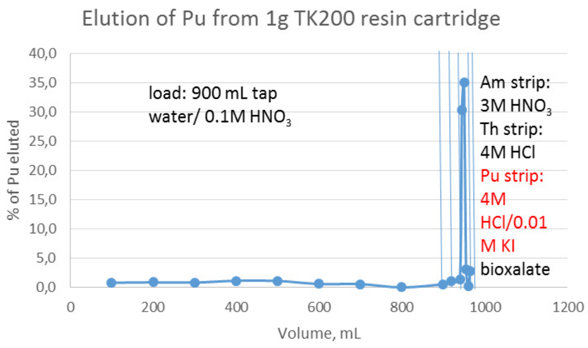


Elution study Th retention and elution on TK200 Resin (data courtesy of Nora Vajda)



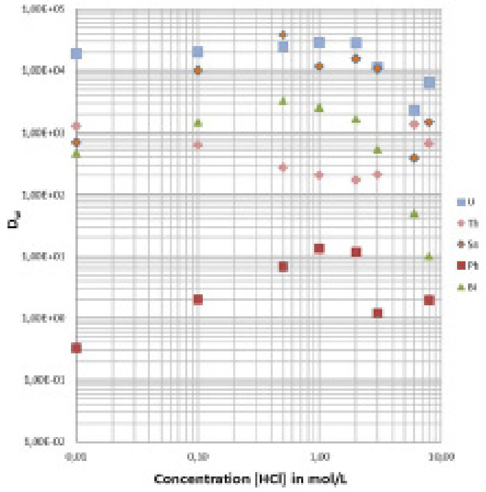
Elution study U retention and elution on TK200 Resin (data courtesy of Nora Vajda)

This allows for using the TK200 Resin for the preconcentration of actinides from acidified water samples, and their subsequent separation on the same column.



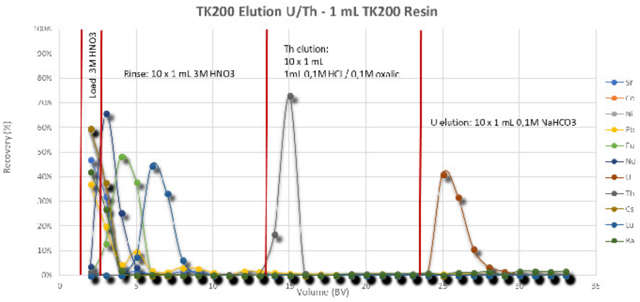
Elution study Pu retention and elution on TK200 Resin (data courtesy of Nora Vajda)

U and Th are also very well retained from HCl, Th even stronger than on TEVA Resin.



D_W values of selected elements on TK200 Resin in HCl

By employing oxalic acid of suitable concentration a clean U/Th separation may be obtained as shown here:



Elution study U/Th separation on TK200 Resin

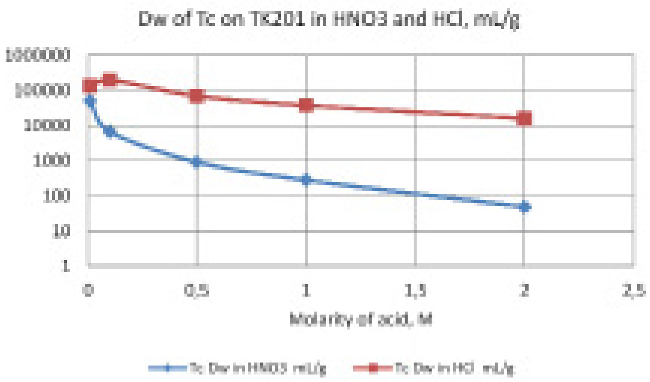
Main applications

- Ga separation for radiopharmaceutical applications (in combination with ZR Resin)
- U, Pu, Th concentration and separation

TK201 Resin

The TK201 Resin is based on a tertiary amine, it further contains a small amount of a long-chained alcohol (radical scavenger) to increase its radiolysis stability. The TK201 Resin rather acts as a weaker ion pair binding agent compared to the TEVA Resin, accordingly it is generally possible to elute under softer conditions.
Its main application is the separation of anionic species such as Tc(VII) or Re(VII).

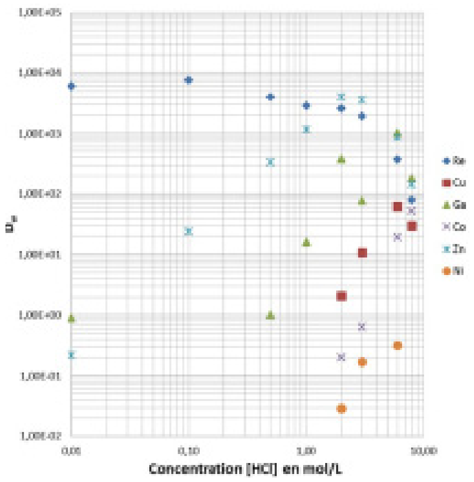
The following graph shows the D_w values for Tc in HNO_3 and HCl.



D_W values of Tc on TK201 Resin in HCl and HNO_3, obtained by LSC, data provided by N. Vajda (RadAnal)

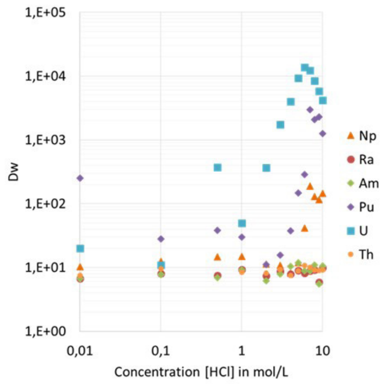
Tc(VII) is very well retained at low acid concentrations. Its retention is generally significantly higher in HCl than in HNO_3, even at elevated HCl concentration such 2M it remains very strongly retained. In HNO_3 on the other hand its retention is rather low at concentrations above 2M.

The following graphs show the selectivity of the TK201 Resin for a wide range of elements in HCl and HNO_3. All D_w shown in these graphs were obtained through ICP-MS measurements.



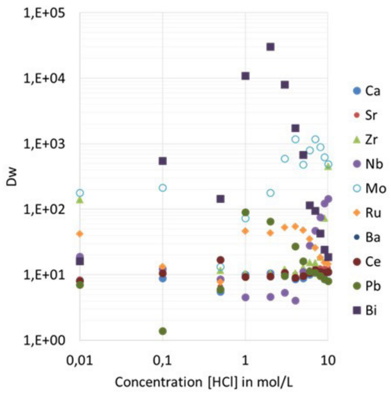
D_W values of selected elements on TK201 Resin in HCl

As expected, the TK201 Resin shows very high retention of Re(VII) in HCl even at rather elevated acid concentrations. Further Zn, Ga and Cu are retained, especially the latter allows for its use in radiopharmaceutical applications.



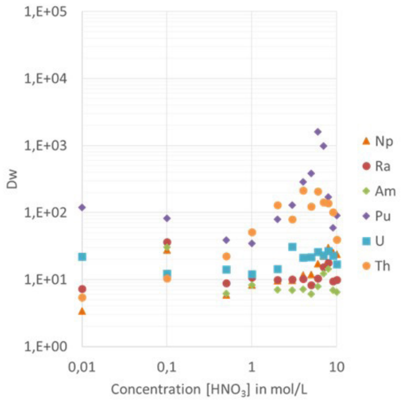
D_W values of selected elements on TK201 Resin in HCl, data provided by Russell et al. (NPL)

The TK201 Resin also shows strong retention of U and Pu at elevated HCl concentrations, both might subsequently be eluted in dilute acid.



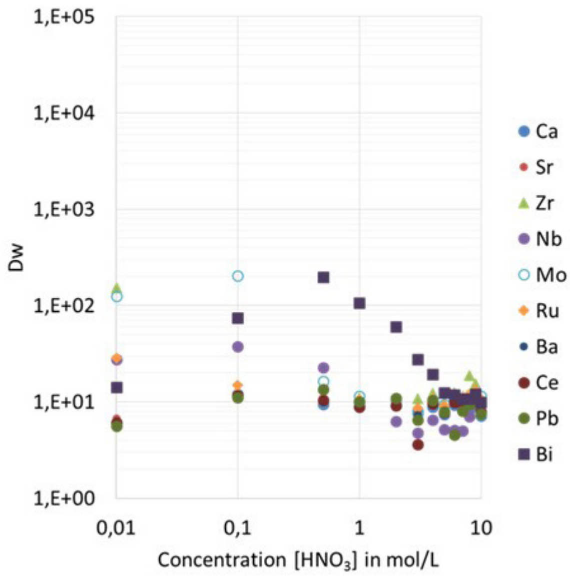
D_W values of selected elements on TK201 Resin in HCl, data provided by Russell et al. (NPL)

The TK201 Resin further strongly retains Bi and Mo at elevated HCl concentrations, while other elements tested show no or only very low retention (Ru, Nb).



D_W values of selected elements on TK201 Resin in HNO_3, data provided by Russell et al. (NPL)

The TK201 Resin generally shows rather limited selectivity in HNO_3, similar to Tc(VII) Re is well retained at low HNO_3 concentrations (0.01 – 0.1M HNO_3). At elevated HNO_3 concentrations Pu is well retained and Th fairly well, other actinides are not retained under these conditions.

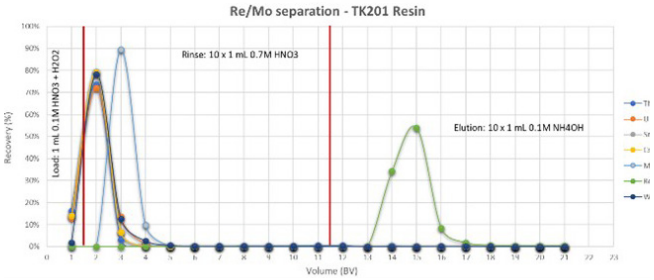


D_W values of selected elements on TK201 Resin in HNO_3, data provided by Russell et al. (NPL)

Out of the other elements tested only Bi (at about 0.5M HNO_3) and Mo (at low HNO_3 concentrations) are retained. It is important to note that Mo is not retained at HNO_3 concentrations above 0.5M while Tc and Re are well retained (as shown in the first figure), allowing for their clean separation.

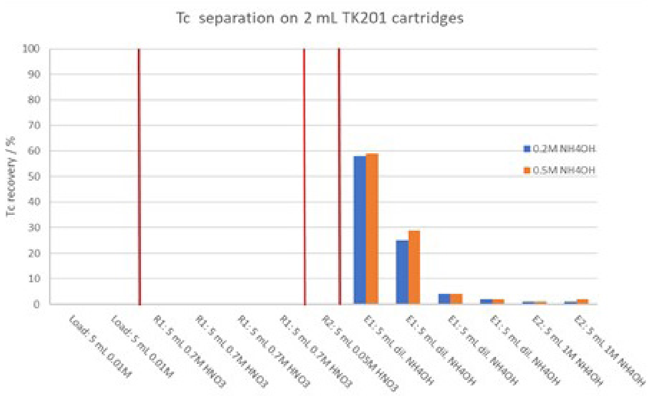
It could further be shown by Vajda et al. that D_w values for Tc(VII) are very low in dilute NH_4OH: in 0.1M NH_4OH Tc(VII) shows a D_w of only ~2, accordingly it is easily eluted by ≥ 0.1M NH_4OH.

Additional elution studies indicated that an efficient Mo separation from Re is possible using 0.7M HNO_3 for Mo removal and dilute NH_4OH for Re elution.



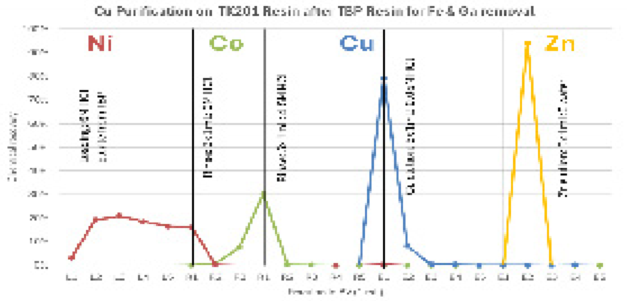
Elution study, Re separation from various elements (incl. Mo and W).

Vajda et al. could confirm that Tc is, like Re, not eluted in 0.7M HNO_3, validating that Re is a good surrogate for Tc and thus also allowing an efficient Mo/Tc separation. Most suitable conditions for Tc elution were found to be NH_4OH greater or equal to 0.2M.



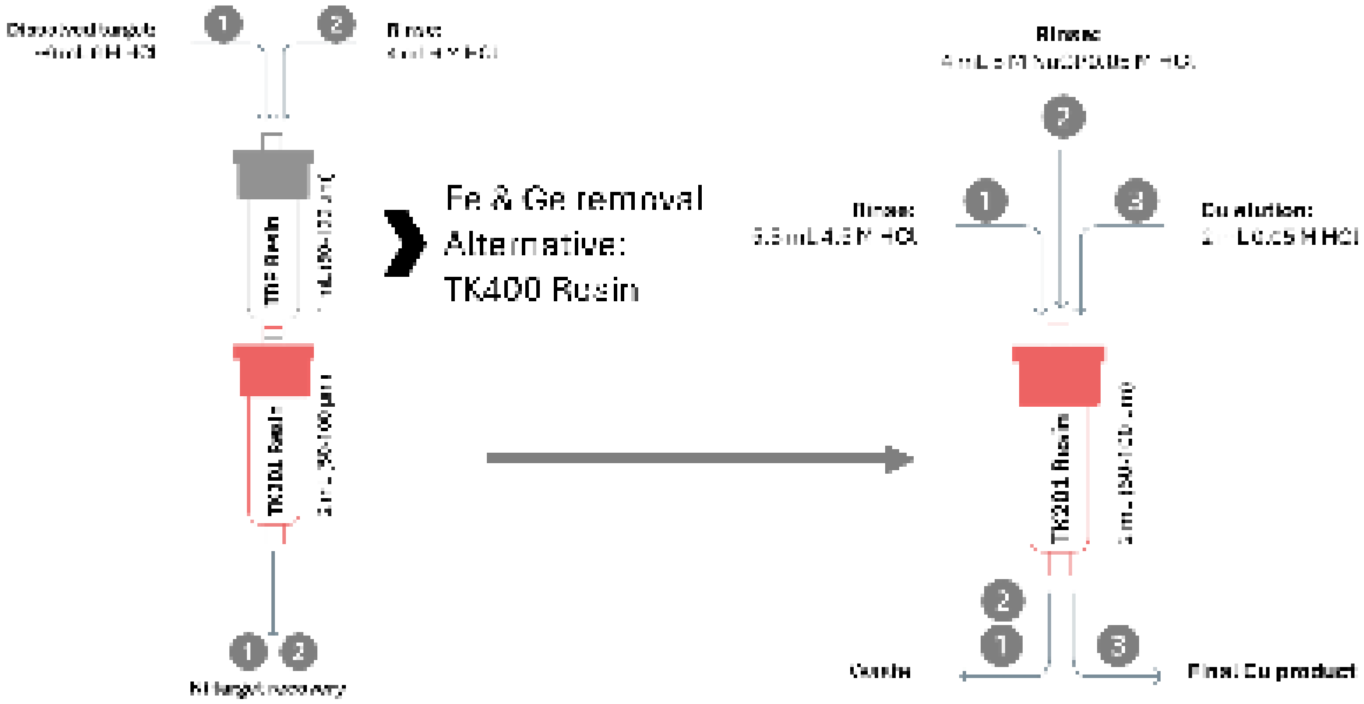
Elution study, Tc separation on 2mL TK201 cartridges, data provided by N. Vajda (RadAnal)

One of the main applications of the TK201 Resin is the separation of Cu isotopes (e.g. Cu-64) from solid Ni targets. Other than the CU Resin the TK201 Resin allows for Cu retention from high HCl (e.g. 6M), while letting Ni pass for subsequent recycling. Other potential impurities (e.g. Co) may be removed through rinses with 4 – 5M HCl. Cu may then be eluted in dilute HCl leaving Zn on the column. The latter can then be eluted with water or dilute HNO_3. This can be used e.g. in Zn isotope ratio determination as shown by Retzmann et al.



Elution study, Cu separation on 1mL TK201 cartridges

In order to remove Fe and Ga impurities potentially present the dissolved Ni target (6M HCl) may first be loaded through a small TBP (or TK400) cartridge which will retain both elements while letting Ni, Cu and Zn pass onto TK201 for further purification. Cu may then be eluted from TK201 e.g. in 0.05M HCl. This could be demonstrated i.e. by Svedjehed et al. The rinse with 5M NaCl/0.05M HCl is particularly noteworthy as it allows obtaining the final product in dilute HCl solution of defined concentration.



Cu separation using TBP and TK201 Resins according to Svedjehed et al.

TK201 may also be used to convert the Cu fraction eluted from the CU Resin (e.g. for the separation of Cu isotopes from Zn targets) from a highly acidic solution (e.g. 6 - 8M HCl) to conditions more suitable for labeling (e.g. dilute HCl). TK201 will retain Cu e.g. from 6M HCl and can then be eluted with dilute HCl as shown e.g. by Kawabata et al. This will also ensure further Zn removal.

Main applications

- Separation of technetium
- Separation of rhenium
- Separation of Cu isotopes
- (incl. combined with CU Resin)

TK211/2/3 Resins

The TK211, TK212 and TK213 Resins are based on different mixtures of organophosphoric, organophosphonic and organophosphinic acids. It could be shown that under certain conditions and for certain lanthanide pairs, such mixtures can show increased selectivity compared to the respective pure compounds.

The organic phase further contains a small amount of a long-chained alcohol that will act as radical scavenger to increase the radiolysis stability of the resin.
The inert support onto which the organic phase is impregnated contains aromatic groups which will also contribute to the increase of the radiolysis stability of the resins.

The inert support further shows an elevated capacity for the extractants. Accordingly, this allows the TK211/2/3 Resins to have a higher extractant load compared to e.g. the LN Resin series.

The TK211/2/3 Resins show, like the LN Resins differences in their respective acidities. TK211 is the most acidic resin, accordingly it will extract lanthanides, and other elements, at higher acid concentrations than e.g. TK212 and TK213. TK212 on the other hand is more acidic than TK213 (order of acidity: TK211 > TK212 > TK213).

The selectivity and retention of the lanthanides is generally very similar in HNO_3 and HCl on all three resins, accordingly both acids may be employed for the separation of lanthanides.

This difference in the relative acidity of the resins can be exploited to facilitate otherwise more complex lanthanide separations.
This will particularly be the case for the separation of very small amounts of one lanthanide from a large excess of its neighbouring lanthanide.

Typical examples are the production of nca Lu-177 (separation from irradiated Yb-176 targets) and nca Tb-161 (separation from irradiated Gd-160 targets).

By performing a first separation on a 'less acidic resin' such as TK212 followed by direct elution of the lanthanide fraction to be further purified onto a more acidic resin such as TK211 for further purification ("sequential separation") it is possible to eliminate intermediary steps such as the use of TK221 (or DGA) Resin to convert the lanthanide fraction from higher acid concentration to low acid concentration, leading to lower Lu/Yb losses.

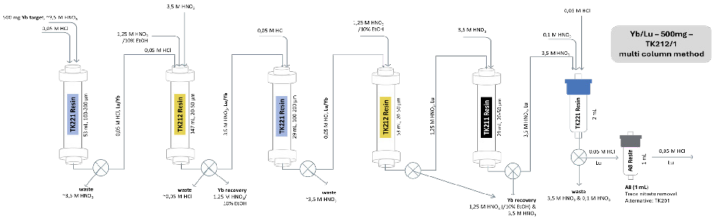
In an ideal case even a fully sequential three column separation might be possible (TK213 => TK212 => TK211).

Two examples of the use of such sequential separation steps will be shown in the following.
The production of nca Lu-177 is rapidly gaining importance due to its increased use in nuclear medicine. Reliable, preferably easy to automize methods that allow for its separation from irradiated Yb-176 targets of elevated size (≥500mg) are thus of increasing importance.

Horwitz et al. describe a method based on three LN2/DGA cycles for the separation of nca Lu-177 from 300mg Yb-176 targets. While this method gives good yields (~73%) in a short separation time (~4h) the fact that an elevated number of columns are required complicates its automatization. Further it has only been tested for up to 300mg of target material.

By introducing a sequential separation step this method can be partially simplified.

It could be shown that the method described in the following figure allows for separating Lu from up to 500mg of Yb with elevated Lu recovery (~85%) and a very low amount of residual Yb in the final Lu fraction.

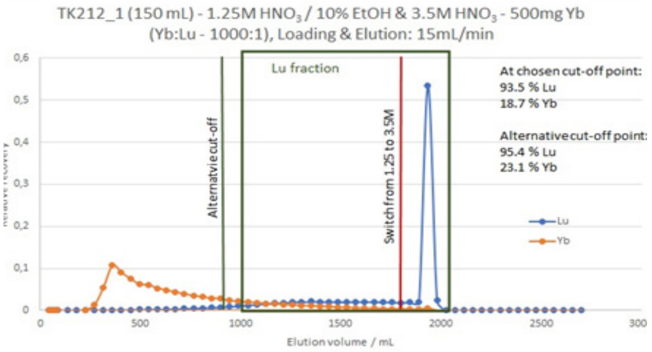


Scheme of a method for the separation of Lu from 500mg Yb using TK212, TK221 and TK211

The increased Lu recovery is, other than the use of TK212 instead of LN2, also due to the adjustment of the eluting agent used for the chromatographic separation of Lu and Yb on the first TK212 column from 1.3M HNO_3, as suggested by Horwitz et al., to 1.25M HNO_3 / 10% EtOH.

It should be noted that adding EtOH only showed an improvement for the 1.25M HNO_3 but not for the 3.5M HNO_3. Further, mixing 3.5M HNO_3 with EtOH should be strictly avoided for safety purposes.

The following figures show typical chromatograms obtained during the separation of Lu from 500mg of Yb (initial Lu:Yb ratio: 1:1000). All experiments were performed using stable elements, fractions of defined sizes were collected, diluted, and analysed off-line by ICP-MS. Relative recoveries were calculated for Lu and Yb and plotted against the elution volume.



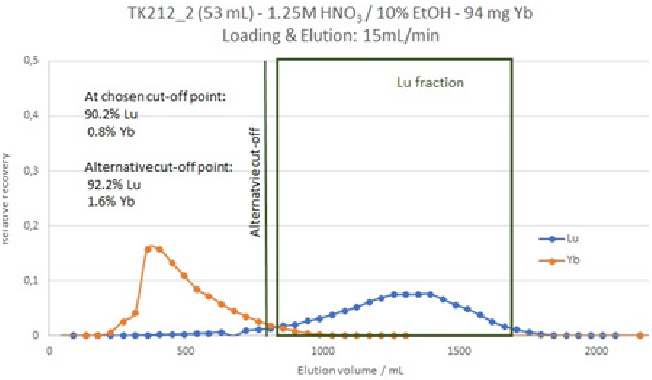
Example of a Lu separation from 500mg Yb on a TK212 column (2.5 x 30cm, 147mL) using 1.25M HNO_3 / 10% EtOH and 3.5M HNO_3

It should be noted that the switch to 3.5M HNO_3 in the given example was made at a rather late stage of the separation. In the final version of the process it should take place earlier, ideally triggered via radiation detection, close to the chosen cut-off point (left end of the green frame).

Indeed, the moment of the switch will have, especially on the first column, a considerable influence on the Lu recovery and Yb carry-over. This is mainly due to the significant tailing introduced by the macro-amount of Yb.

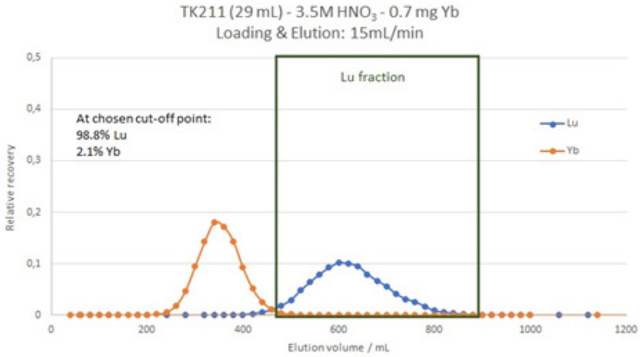
The fractions comprised in the green frame ("Lu fractions") were combined and passed through a 5-10g TK221 cartridge for conversion to ≤0.05M HCl. The Lu fraction thus obtained in dilute HCl was then loaded onto the next TK212 column (1.5 x 30cm, 53mL).
Due to the lower of amount of Yb present on the column the tailing of the Yb and Lu elution is less pronounced than on the first TK212 column.

Although this would be possible, in this example the Lu containing fractions (green frame) are not eluted in HNO_3 of elevated concentration (as described in the Horwitz method), passed through a TK221 (or DGA) cartridge and eluted in dilute HCl for another load onto TK212.



Example of a Lu separation fraction, obtained from a first TK212 separation, from 94 mg Yb, on a second TK212 column (1.5 x 30cm, 53mL) using 1.25M HNO_3 / 10% EtOH

Instead the combined Lu fractions are directly loaded onto a TK211 column (1.1 x 30cm, 29mL) for the final purification of the Lu.



Example of a Lu separation fraction, obtained from the second TK212 separation, from <1 mg Yb, on a TK211 column (1.1 x 30cm, 29mL) using 3.5M HNO_3

Lu is finally obtained following separation/elution e.g. with 3.5M HNO_3.

As final step the obtained Lu fractions (as indicated in the green frame) were combined and loaded onto a 2 mL TK221 cartridge, any last potentially present impurities are removed through consecutive rinses with 3.5M HNO_3 and 0.1M HNO_3. Lu is then finally eluted using ≤0.05M HCl.

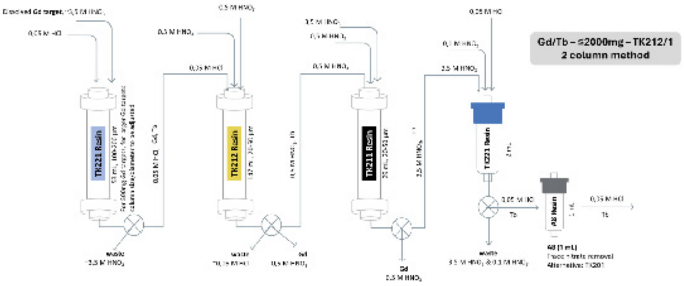
Last traces of nitrates that might still be present will be removed via a 1mL anion exchange resin cartridge (A8 Resin, alternatively TK201 may be used).

A further upscale of this separation process is currently being finalized.

Another radiolanthanide increasingly finding use is Terbium. As Tb isotopes may be used for PET imaging (Tb-152), SPECT imaging (Tb-155), alpha therapy (Tb-149) and beta therapy (Tb-161) it is also referred to as the 'swiss army knife' of nuclear medicine.
Particularly the interest in Tb-161 is currently increasing significantly, accordingly methods for the separation of Tb from irradiated Gd targets of elevated size are needed.

The upscale, of methods for the separation of Tb from 500-2000mg Gd is currently on-going. The next figure shows a scheme of a suggested separation process.

As may be seen the separation is more straightforward compared to the separation of Lu from Yb targets.



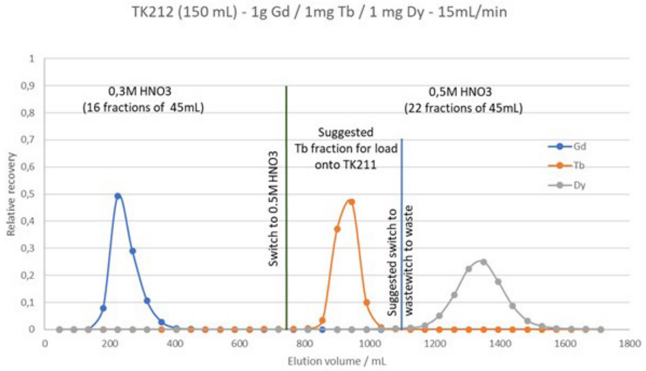
Scheme of a method currently under optimisation for the separation of Tb from 500-2000mg Gd using TK221, TK212 and TK211

The following two figures show typically obtained chromatograms (stable Gd, Tb and Dy, with an original ratio of 1000:1:1).

Like for the Lu separation the separations were performed using stable elements, fractions of defined volumes were taken and analysed by ICP-MS.

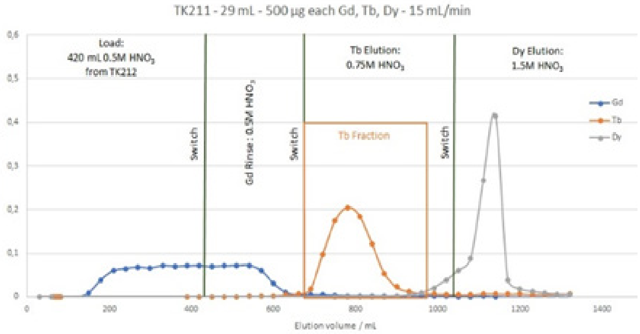
A first separation is performed on a TK212 column. This step allows for an initial separation of Tb from Gd and Dy.
The obtained Tb fractions (indicated by the orange frame) were then combined and directly loaded onto a TK211 column for final purification of the Tb.
As may be seen in the next figure under the chosen conditions most of the Gd is breaking through during the load, any Gd remaining on the columns is rinsed of with 0.5M HNO_3.

It could be shown for the Lu separation process that the addition of small amounts of EtOH (10% v/v) improves the separation, this is currently also being tested for the Tb separation.



Example of a Tb separation from 1000mg Gd on a TK212 column (2.5 x 30cm, 147 mL) using 0.3M HNO_3 and 0.5M HNO_3

Increasing the concentration of the mineral acid (in this example to 0.75M HNO_3) will lead to the elution of Tb, leaving potentially remaining traces of Dy on the columns. In case the presence of Dy can be ruled out, or deemed irrelevant, this elution can be performed at higher acid concentrations (e.g. 3.5M HNO_3), thus lowering the elution volume.



Example of a Tb separation from 500µg Gd on a TK211 column (1.1 x 30cm, 147 mL) using 0.5M HNO_3 and 0.75M HNO_3

As a final step the Tb will be concentrated on a 2mL TK221 cartridge, any last potentially present impurities are removed through consecutive rinsed with 0.75M HNO_3 and 0.1M HNO_3. Tb is the finally eluted using ≤0.05M HCl.

Last traces of nitrates that might still be present will be removed via a 1mL anion exchange resin cartridge (A8 Resin, alternatively TK201 can be used).

The indicated method is currently undergoing further optimisation and upscale.

Prepacked TK211/212/213 columns of various sizes (e.g. 380mL, 147mL, 53mL and 29mL) are available (PP and PEEK).

Main applications

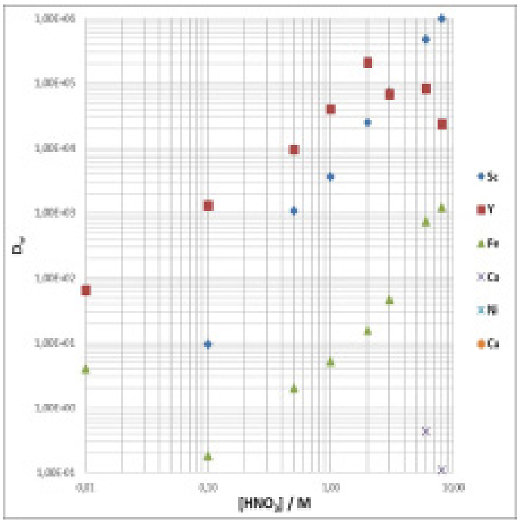
- Lanthanide separation e.g. nca Lu-177 and nca Tb-161

TK221 Resin

The TK221 Resin is based on a mixture of a diglocylamide and a phosphine oxide. It further contains a small amount of a long-chained alcohol and the organic phase is impregnated onto an inert support containing aromatic groups for increased stability against radiolysis.

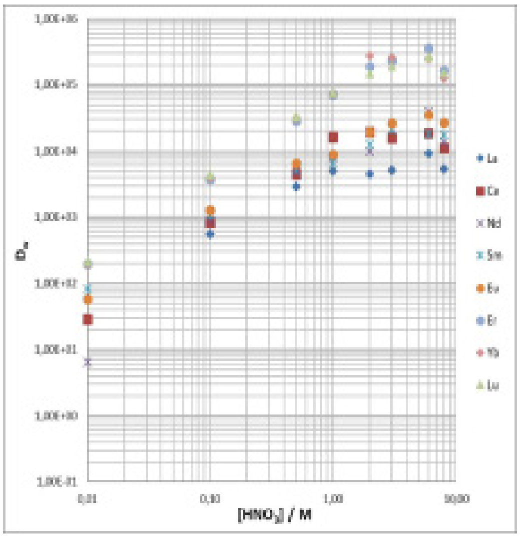
The following graphs show the selectivity of the TK221 Resin for a wide range of elements in HNO_3 and HCl.

Out of the tested elements only Ca is weakly retained on the TK221 Resin in HNO_3. Other alkaline, earth-alkaline elements and Al are not retained.



D_W values of selected elements on TK221 in HNO_3

Y and Sc are very strongly retained from HNO_3 of elevated concentration. Fe(III) is also well retained at HNO_3 concentration ≥ 3M HNO_3.

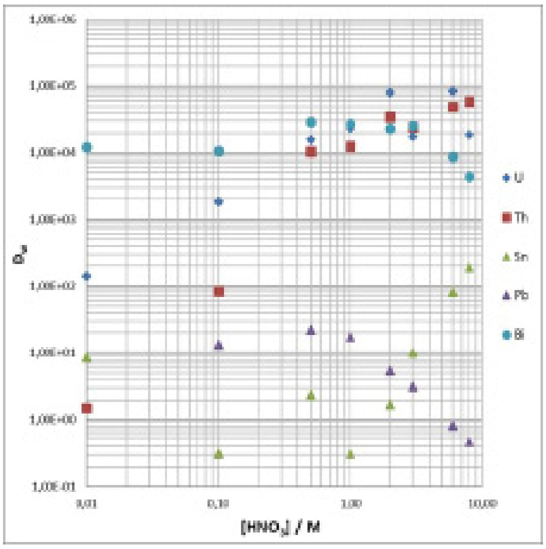


D_W values of selected elements on TK221 in HNO_3

A wide range of transition metals such as Zn, Ga, Co, Ni and Cu are not retained from nitric acid.

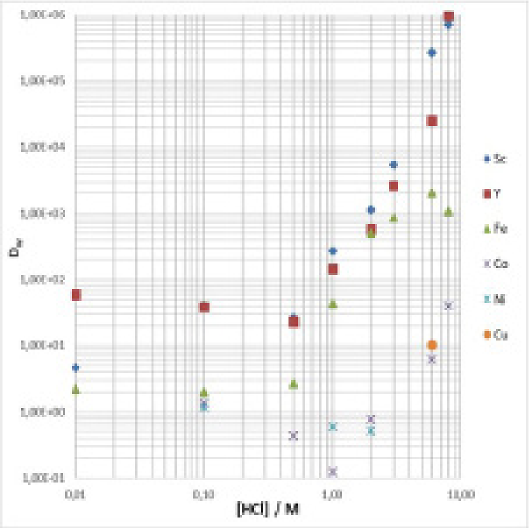
The TK221 Resin generally retains tetravalent elements such as Zr and Hf at elevated HNO_3 concentrations.

The TK221 Resin shows very high retention of lanthanides at HNO_3 concentrations ≥ 0.1M HNO_3, heavy lanthanides are even well retained in more dilute HNO_3 (≥ 0.01M). The retention of the lanthanides is significantly stronger than on TRU Resin.



D_W values of selected elements on TK221 in HNO_3

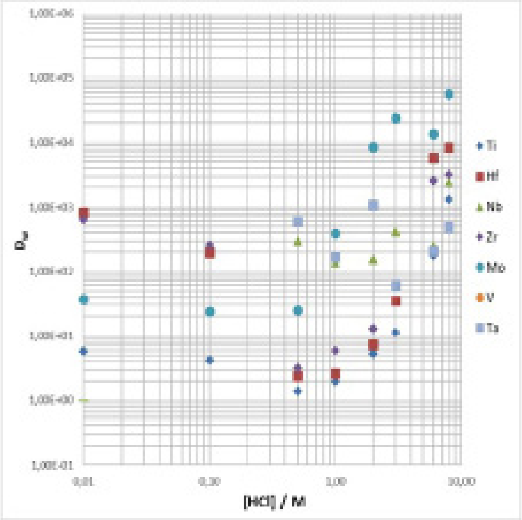
U and especially Bi are well retained over the whole HNO_3 concentration range, while Th is well retained at HNO_3 > 0.1M. U retention is significantly higher than on other diglycolamide based resins such as DGA Resin. Pb and Sn are only weakly retained.



D_W values of selected elements on TK221 in HCl

In HCl medium, none of the tested alkaline and earth-alkaline elements were retained on the TK221 Resin the same is true for Al.

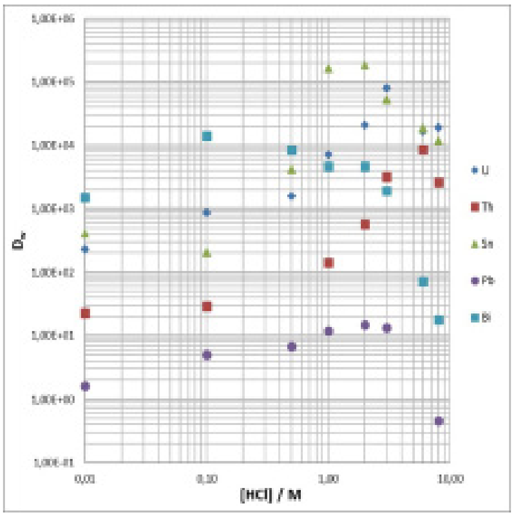
Y and Sc are very strongly retained from HCl at elevated concentration (≥ 2M HCl). Fe(III) is also well retained at HCl concentration ≥ 3M.



D_W values of selected elements on TK221 in HCl

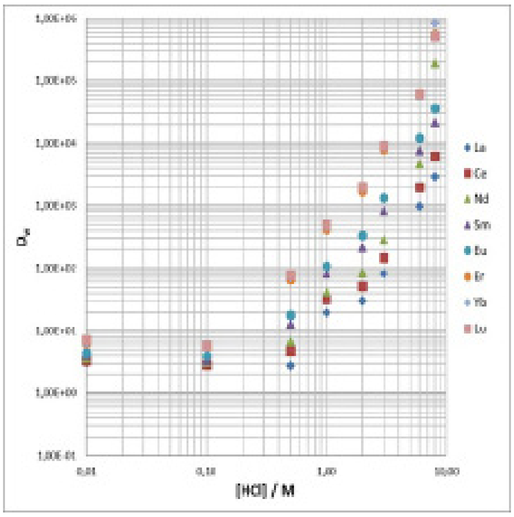
Elements with a valency of +IV and higher such as Nb, Zr, Hf and Mo are very well retained at elevated HCl concentrations.

Other than many other transition metals, Zn and Ga are very well retained from ≥ 2M HCl. Both may be easily eluted in dilute HCl.



D_W values of selected elements on TK221 Resin in HCl

U, Sn and Bi are well retained over the whole HCl concentration range, while Th is only well retained at ≥ 3M HCl. Pb is generally only very weakly retained.

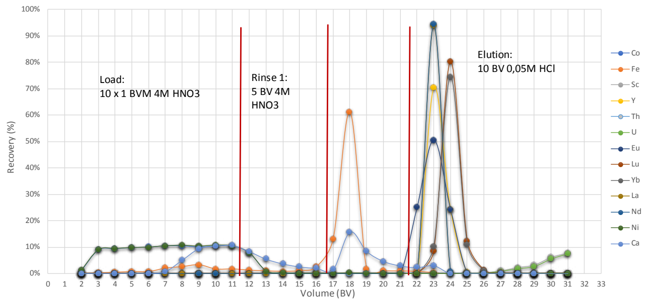


D_W values of selected elements on TK221 in HCl

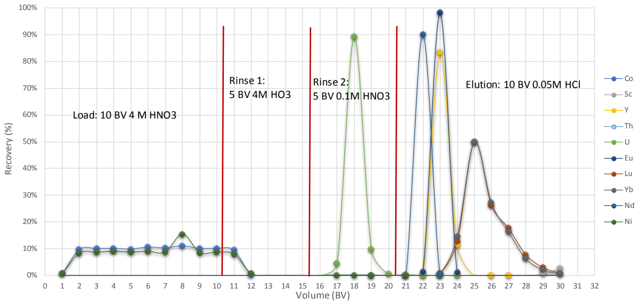
Lanthanides are generally very well retained at HCl concentrations ≥ 3M HCl, heavy lanthanides even at ≥ 1M, and they may be eluted in dilute HCl.

One of the main applications of TK221 Resin is the concentration, purification and conversion of heavy lanthanides such as Lu from highly acidic solutions into dilute HCl (typically ~0.05M HCl) conditions.

It allows e.g. to elute Lu in a smaller volume than DGA,N Resin. Accordingly, it may e.g. find use in the production of Lu-177.



Elution study, various elements on TK221 Resin



Elution study, various elements on DGA, normal

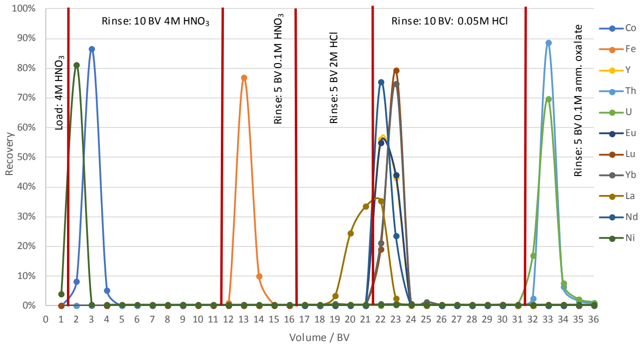
A number of separation methods based on the TK221 Resin are currently being developed particularly for ca and nca Lu-177 purification, as well as the use of TK221 as part of the separation of nca Lu-177 from up to 500mg Yb-176.

The final product obtained using the TK221 Resin is typically additionally passed through a 1mL A8 cartridge for trace nitrate removal.

Similar separations are also applicable to the purification of Ac-225.

The fact that the TK221 Resin is showing higher U retention compared to e.g. DGA,N Resin might further allow for its use in a two column separation method for sequential actinides separation.

The following figure shows an elution study of various elements including U on TK221.



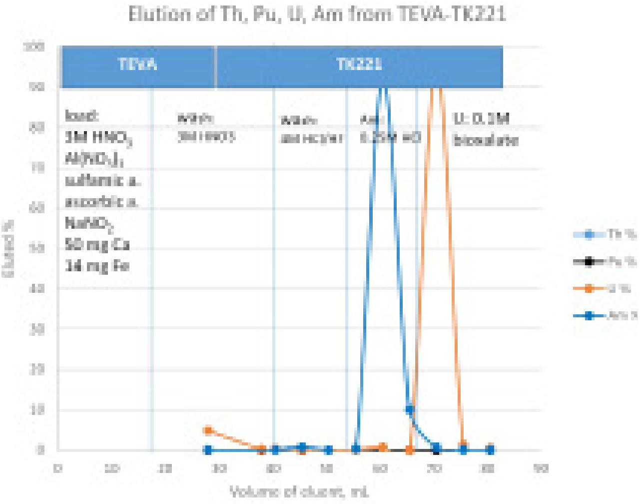
Elution study, various elements on TK221

U is very well retained under all employed HNO_3 and HCl concentrations and may finally be eluted in 0.1M oxalate. Am is expected to be eluted before U in dilute HCl.

With respect to the TK221 selectivity a stacked TEVA/TK221 method for the separation of U, Th, Pu, Am/Cm and Np seems very well possible.

In such a case Np(IV), Pu(IV) and Th(IV) would be retained, and separated, on TEVA while U and Am would pass through TEVA onto TK221 where both would be retained. It should then be possible to first elute Am with dilute HCl and finally U with dilute oxalic acid.

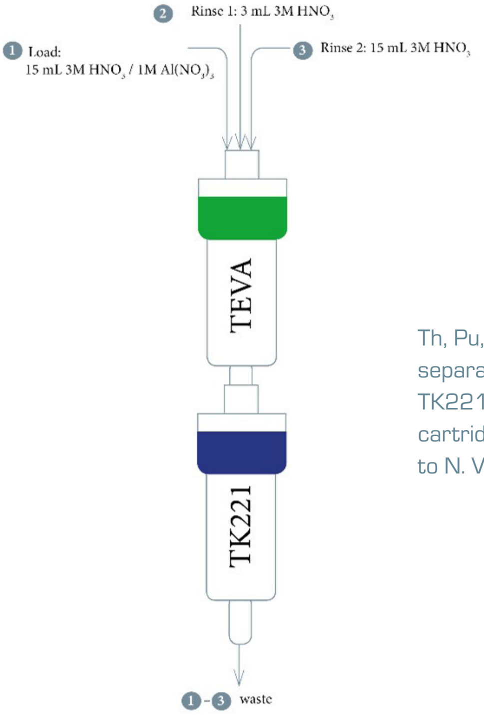
N. Vajda et al. developed such a method for the separation of Th, Pu, Am and U from water samples based on the TEVA/TK221 system. The development work was based on a typical Ca-Phosphate preconcentration step, and took into account the possible presence of Fe(III) originating from the oxidation state adjustment. Through a very thorough optimisation of the Am elution volumes a clean separation of Am and U on the TK221 could be achieved as shown below.



Th, Pu, Am and U separation on a 2mL TK221 cartridge (data courtesy of N. Vajda et al.)

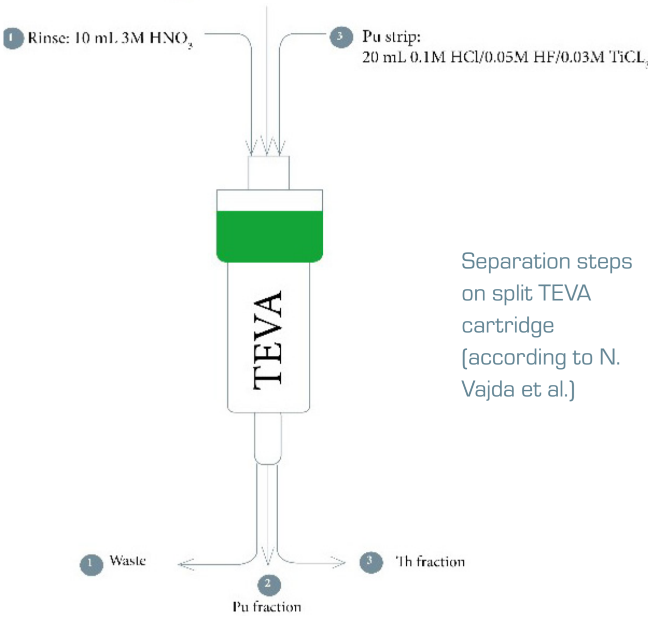
Overall, the authors reported high chemical yields (92 – 106%) under the given conditions, and very good decontamination of the obtained actinide fractions (cross-contamination <1% respectively).

The developed separation protocol is summarized in the following figures.

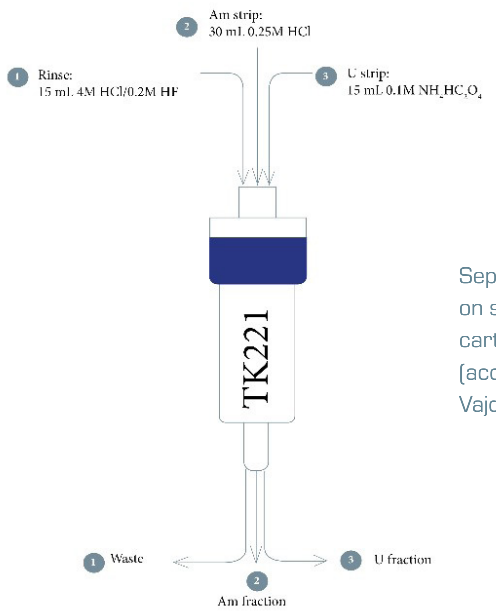


Th, Pu, Am and U separation on TEVA/ TK221, stacked cartridges (according to N. Vajda et al.)

The dissolved CaPhosphate precipitate is first passed through stacked TEVA and TK221 cartridges. The cartridges are then rinsed with 3M HNO_3 to assure matrix removal and quantitative transfer of U and Am onto the TK221 cartridge. Both cartridges are then separated: Pu and Th are separated on the TEVA cartridge, while U and Am are separated on the TK221 cartridge. The use of TK201 or TK200 instead of TEVA is currently being tested.



Separation steps on split TEVA cartridge (according to N. Vajda et al.)

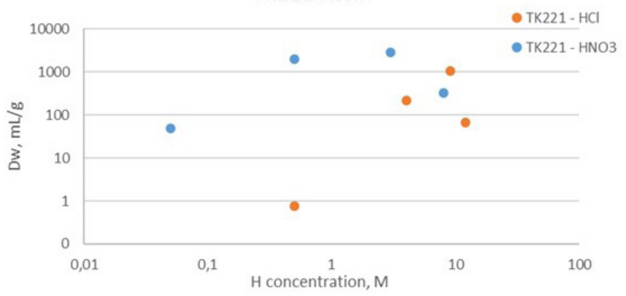


Separation steps on split TK221 cartridge (according to N. Vajda et al.)

When applying the developed method to the alpha spectrometric determination of tap and a sea water samples spiked with Th-230, Pu-239, Am-241 and U-233 they could confirm the clean separation of the actinides, as well as the fact that high chemical yields may be obtained.

Even for a highly charged matrix such as a sea water sample, chemical yields were in the order of ~90% for U, Pu and Am and ~70% for Th, only about 10 – 20% lower compared to the tap water samples (90 – 108%), making this a very promising alternative to the classical TEVA/TRU methods with the additional benefit of a more robust Am retention.

N. Vajda et al further examined the TK221 Resin with respect to Ac retention.



Weight distribution ratios (D_W) of Ac on TK221 Resin. Data courtesy of N. Vajda (RadAnal)

Overall, the D_w values for Ac are elevated in HNO_3, even at very high (e.g. 8 - 10M) or low (e.g. 0.05M) concentrations. In HCl Ac retention is high at elevated HCl concentrations (9M HCl) while decreasing significantly at higher and lower acid concentrations. Especially at low HCl concentrations Ac D_w values are very low, indicating suitable elution conditions.

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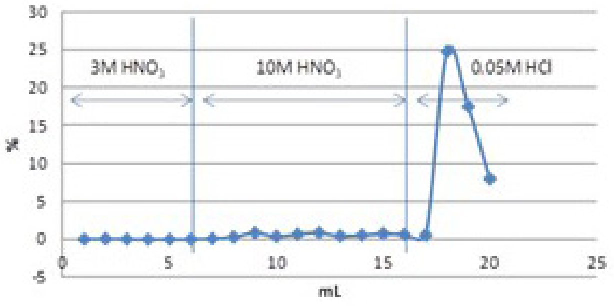
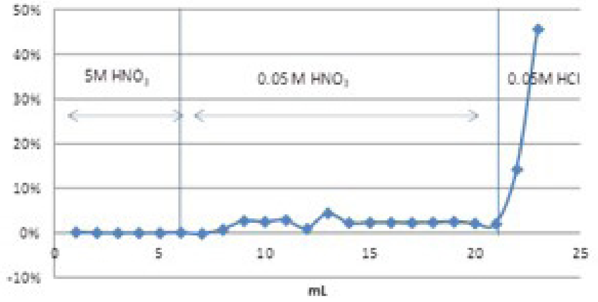
The high retention of Ac over a wide HNO_3 concentration range could further be shown through elution experiments.



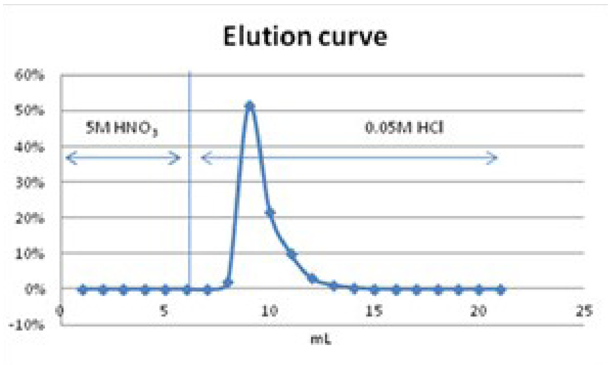
Ac elution study on 1mL TK221 cartridge (100 – 200µm), 10M HNO_3 rinse. Data courtesy of N. Vajda et al.

Other than the DGA Resins the TK221 doesn't allow for Ac elution in 10M HNO_3, a step frequently used for Ac/Lanthanide separation. For TK221 even higher HNO_3 concentrations are required (~14M).



Ac elution study on 1mL TK221 cartridge (100 – 200µm), elution with 0.05M HNO_3. Data courtesy of N. Vajda et al.

TK221 does not allow, contrary to the DGA Resins, Ac elution in dilute HNO_3.



Ac elution study on 1mL TK221 cartridge (100 – 200µm), 0.05M HCl elution. Data courtesy of N. Vajda et al.

The fact that TK221 retains Ac from dilute HNO_3 and allows its elution in dilute HCl might open the possibility of converting Ac solutions from dilute HNO_3 to dilute HCl using TK221.

It should be noted that TK221 is now also available in 50 – 100µm particle size now, which should allow for narrower elution.

Overall, a separation method similar to Lu-177 purification seems possible: Ac retention from elevated HNO_3, (or HCl) rinse with dilute HNO_3 to remove impurities and lower HNO_3 concentration on the resin, followed by elution in HCl. Ideally the final product should be loaded through a small anion exchange resin cartridge (e.g. 1mL A8) to remove last traces of nitrate.

Additional work on the Ac separation on TK221 Resin is currently on-going.

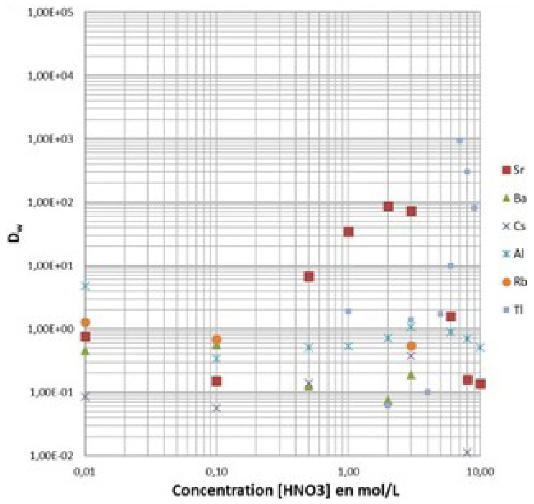
Main applications

- Separation and concentration of lanthanides (e.g. ca and nca Lu-177)
- Separation of actinides
- Separation of actinium

TK222 Resin

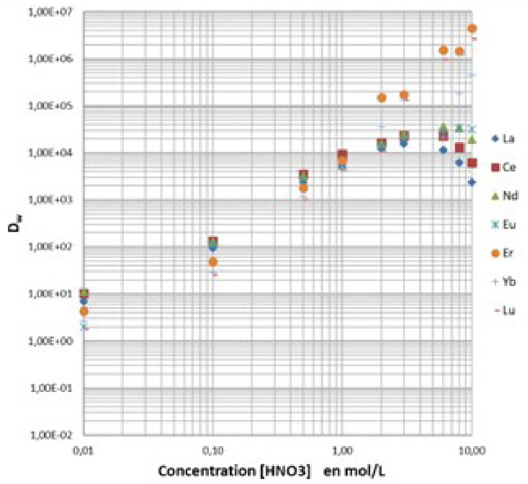
The TK222 Resin is based on a mixture of a branched diglocylamide and a phosphine oxide. It also contains a small amount of a long-chained alcohol. Further, the organic phase is impregnated onto an inert support containing aromatic groups for increased stability against radiolysis.

The following graphs show the selectivity of the TK222 Resin for a wide range of elements in HNO_3 and HCl. All D_W values shown in these graphs were obtained through ICP-MS measurements.



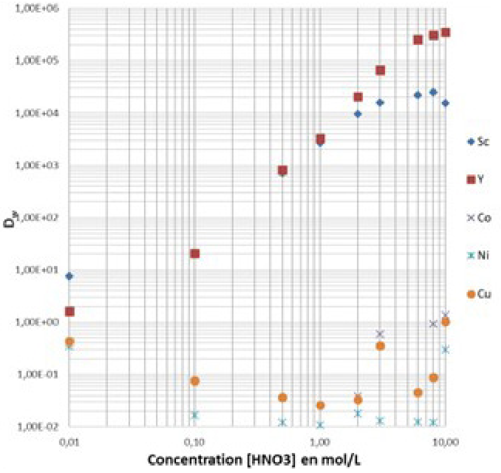
D_W values of selected elements on TK222 in HNO_3

Out of the shown elements only Sr at medium high HNO_3 concentration (2 – 3M) and Tl at elevated concentrations (~8M) are retained.



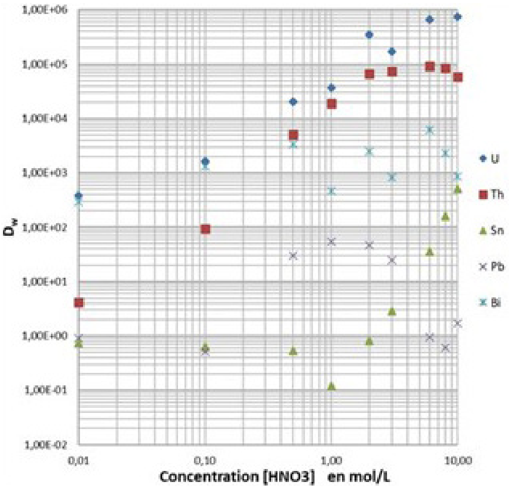
D_W values of selected elements on TK222 in HNO_3

Lanthanides are generally very well retained at elevated HNO_3 concentrations (≥0.5M), this is particularly true for heavy lanthanides. This point is particularly interesting with respect to the separation of lanthanides from Ac. D_W values are generally low at low HNO_3 concentrations.



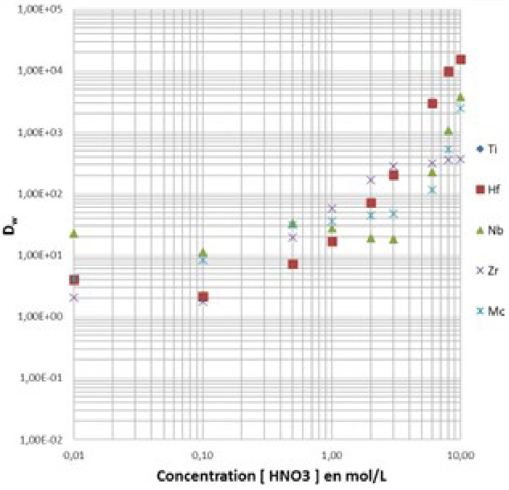
D_W values of selected elements on TK222 in HNO_3

Y and Sc are very well retained at elevated HNO_3 concentrations, while Co, Ni and Cu are not retained.



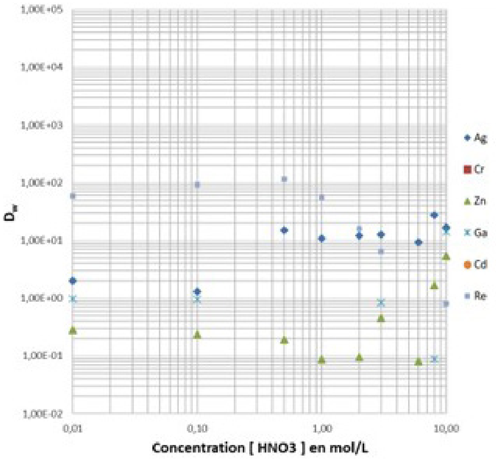
D_W values of selected elements on TK222 in HNO_3

U and Th are very well retained from elevated HNO_3 concentrations. Bi, too is well retained, to a lesser extent than U and Th though. Sn shows some retention at elevated HNO_3. Pb is generally only rather weakly retained with a maximum between 0.5 and 3M HNO_3.



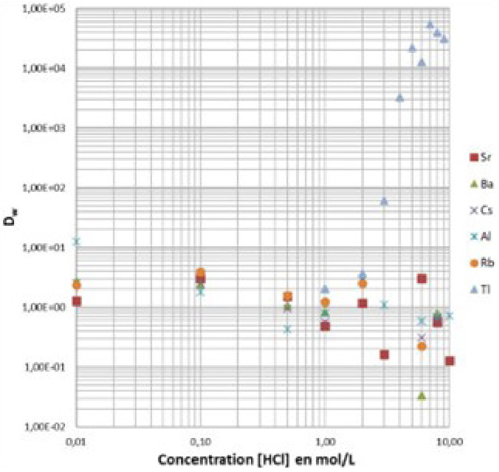
D_W values of selected elements on TK222 in HNO_3

Elements of higher valency such as Hf, Zr, Nb and Mo are well retained from HNO_3 of high concentration.



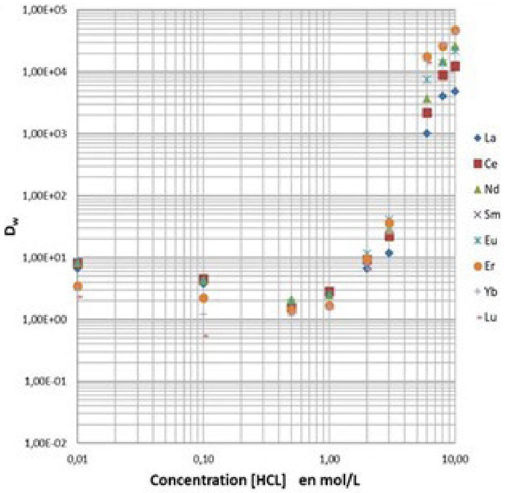
D_W values of selected elements on TK222 in HNO_3

None of the shown elements show significant retention on TK222 from HNO_3.



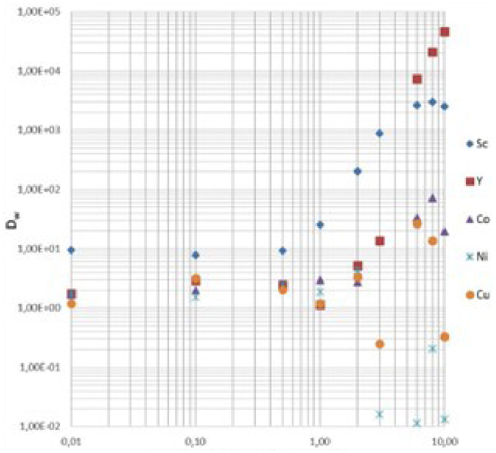
D_W values of selected elements on TK222 in HCl

Out of the shown elements only Tl is well retained at high HCl concentrations.



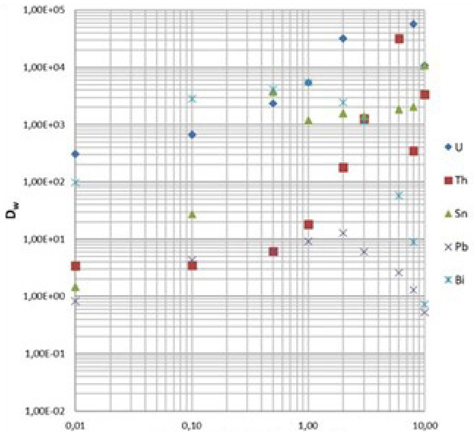
D_W values of selected elements on TK222 in HCl

Lanthanides are strongly retained at high HCl concentrations (≥ 6M) for example. As for HNO_3 this is an important information with respect to the separation of lanthanides from Ac.



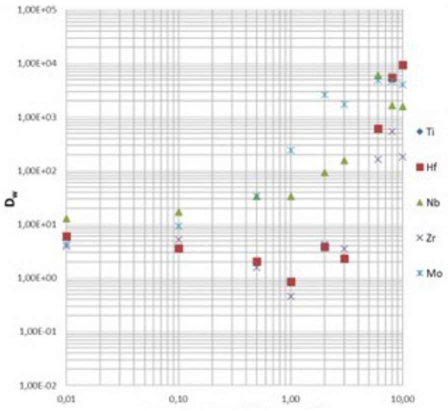
D_W values of selected elements on TK222 in HCl

Like the Lanthanides Y and Sc are very well retained at high HCl concentrations. Co, Ni and Cu are not or only weakly retained.



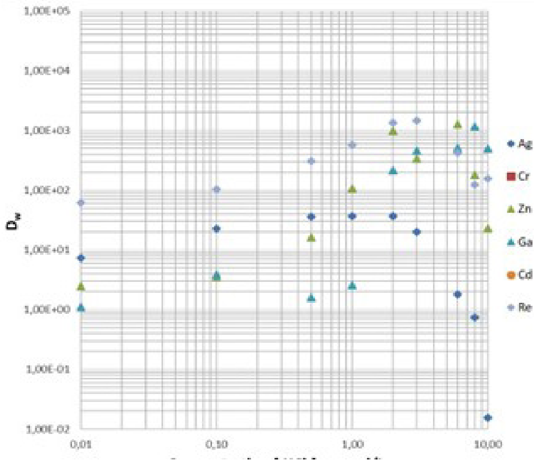
D_W values of selected elements on TK222 in HCl

U, Th and Sn show strong increase of D_W values with increasing HCl concentrations. Pb is only very weakly retained from HCl. Bi is well retained between 0.1M and 2M HCl, its retention then sharply drops with increasing HCl concentration. 10M HCl may e.g. be used to elute Bi from the TK222.



D_W values of selected elements on TK222 in HCl

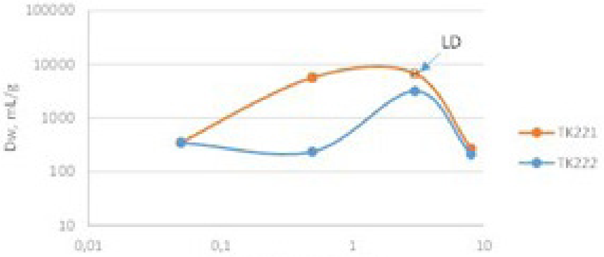
Like for HNO_3, elements of higher valency like Mo, Nb, Zr and Hf are well retained at high acid concentrations.



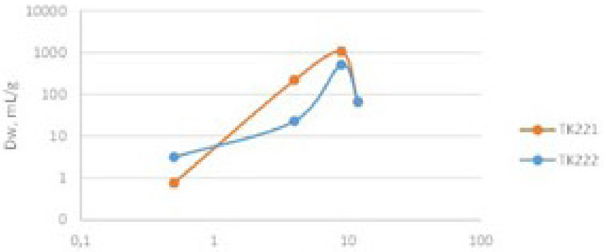
D_W values of selected elements on TK222 in HCl

At elevated HCl concentrations Zn and Ga are quite well retained, while the other elements shown are not retained.

The following graphs show the behavior of Ac on TK221 and TK222 (data courtesy of Nora Vajda, RadAnal, all obtained via LSC).



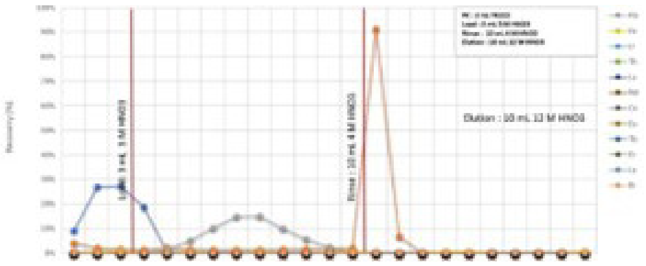
D_W values of Ac on TK221 and TK222 in HNO_3
(data courtesy of N. Vajda, Radanal)



D_W values of Ac on TK221 and TK222 in HCl
(data courtesy of N. Vajda, Radanal)

These graphs compare D_W values for Ac on TK221 and TK222 from HNO_3 and HCl. As can be seen TK221 retains Ac significantly stronger than the TK222 Resin. The latter is, on the other hand, easier to elute. Both show rather low D_W values at very high HCl concentrations (> 10M), this should, with respect to the resin's selectivity for lanthanides, allow for the separation of Ac from the lanthanides. Elution in HNO_3 will require significantly higher HNO_3 concentrations (≥ 14M HNO_3) to elute Ac.

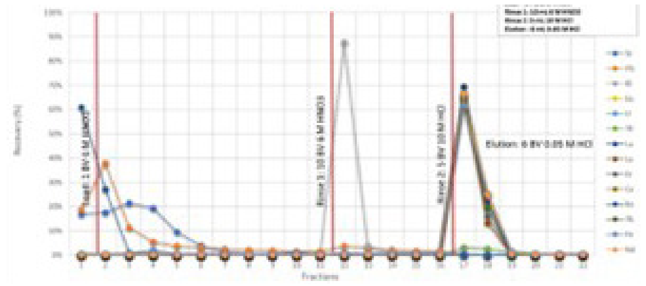
The elution studies below were performed with stable elements and ICP-MS measurements.



Elution study, 2mL TK222 cartridge,
1 BV fractions, various elements.

Ba (the same should be true for Ra) and Pb are removed at elevated HNO_3 concentrations (2 – 4M HNO_3), for Sr elution even higher HNO_3 concentrations are required (here 12M HNO_3).

Under these conditions lanthanides, U and Th remain retained on TK222 Resin, while Ac is expected to elute which should result in a suitable separation of Ac from these elements.



Elution study, 2mL TK222 cartridge, 1 BV fractions, various elements.

When loading the TK222 Resin from 6M HNO_3, followed by a rinse with the same acid, Pb, Ba and Sr are removed. Bi may then be removed using 10M HCl. As can be seen, under the usual Ac elution conditions (0.05M HCl) lanthanides would co-elute, accordingly they need to be removed as described before via the Ac elution from TK222 (or TK221) in very high HNO_3.

Main applications

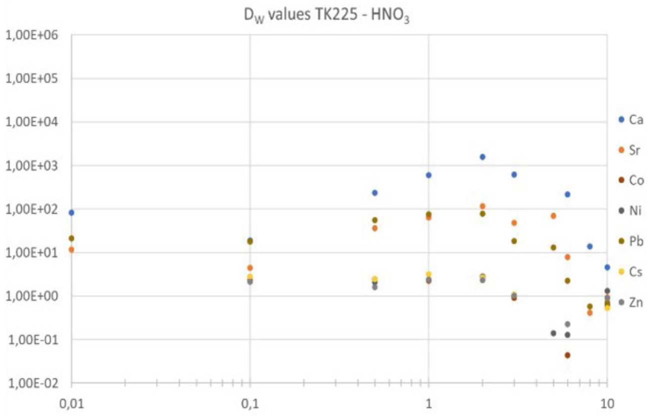
- Ac purification
- Lu-177 purification

TK225 Resin

The TK225 Resin is based on a mixture of a diglycolamide and an ionic liquid. The organic phase is impregnated onto an inert support containing aromatic groups for increased stability against radiolysis.

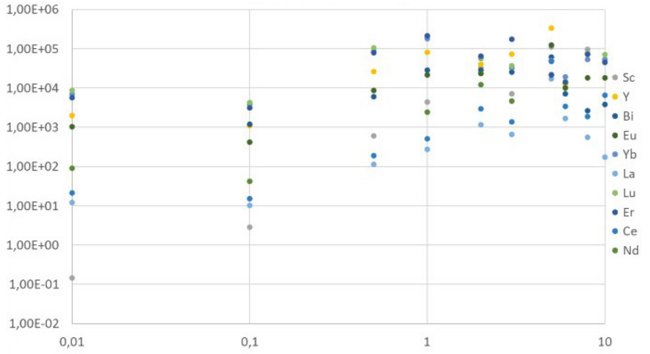
The main application of the TK225 Resin is the removal of radiolanthanides from acidic solutions, particularly from solutions of elevated HNO_3 concentration, for decontamination purposes.

The following graphs show the selectivity of the TK225 Resin for a wide range of elements in HNO_3 and HCl. All D_w shown in these graphs were obtained through ICP-MS measurements.



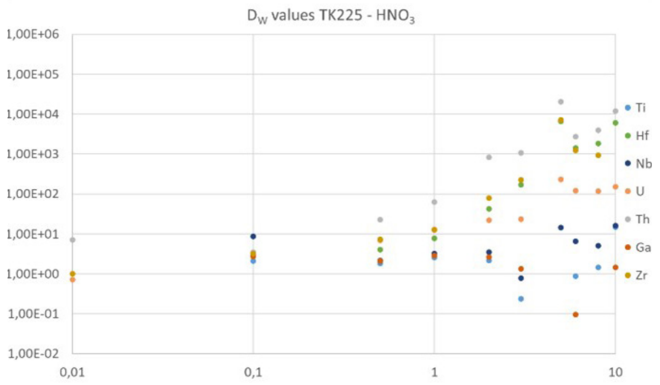
D_W values of selected elements on TK225 in HNO_3

Out of the tested elements only Ca is quite strongly retained at elevated HNO_3 concentrations. Sr and Pb, too are retained under these conditions to a lesser extent though.



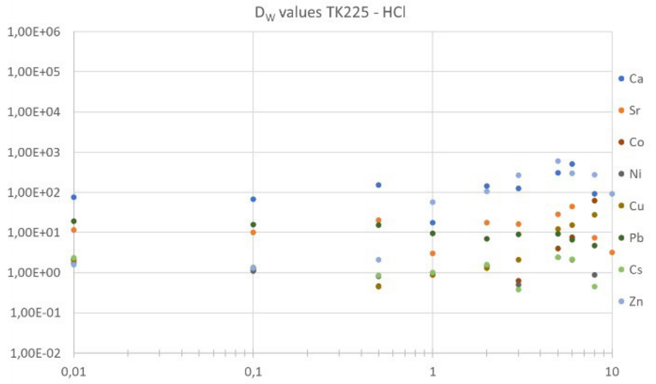
D_W values of selected elements on TK225 in HNO_3

Lanthanides, especially heavy lanthanides, Y and Sc are very strongly retained from HNO_3 of elevated concentration. Especially for heavy lanthanides the D_w values remain very high, even at low HNO_3 concentrations.



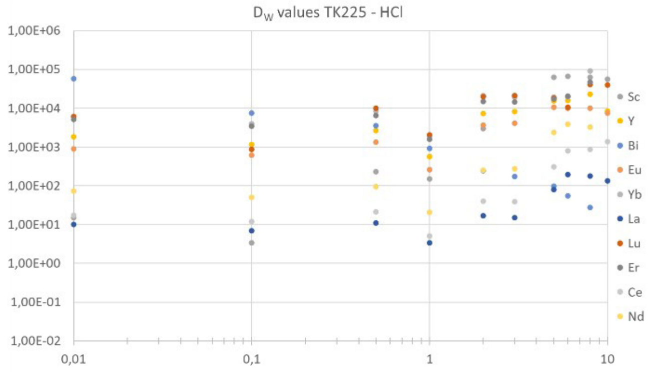
D$_W$ values of selected elements on TK225 in HNO$_3$

The TK225 Resin generally retains tetravalent elements such as Zr, Hf and Th at elevated HNO$_3$ concentrations quite strongly.



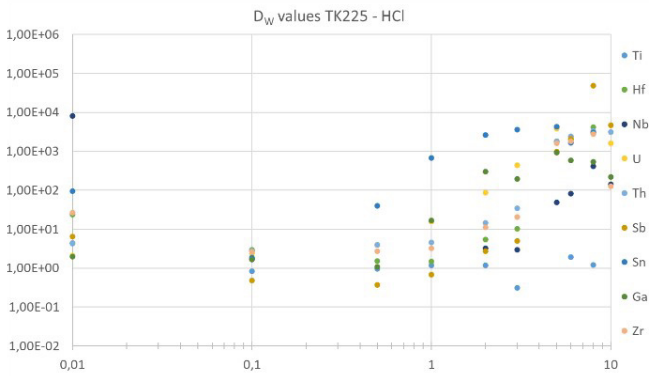
D$_W$ values of selected elements on TK225 in HCl

The TK225 Resin shows elevated retention of Ca and Zn at high HCl concentrations. Other elements shown are not or only very weakly retained.



D$_W$ values of selected elements on TK225 in HCl

Especially heavy lanthanides are well retained over a broad HCl concentration range, with highest retention being observed at high HCl concentrations.
At high HCl concentrations Y, Sc and lighter lanthanides are well retained, too.



D$_W$ values of selected elements on TK225 in HCl

Higher valent elements such as Sb, Sn, Zr and U are well retained at high HCl concentrations, while showing very little retention at low HCl concentrations. TK225 Resin is mainly used for the removal of radiolanthanides, especially heavy radiolanthanides such as Lu-177, Yb-175, Tb-161,... from acidic solutions.

Especially the heavy lanthanides are near impossible to elute, accordingly the resin is mainly suitable for the decontamination of acidic effluents and waste solutions.

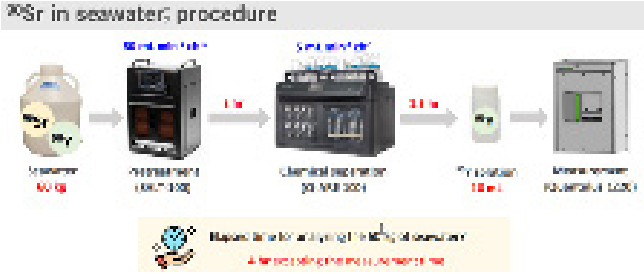
Main applications

- Lanthanides removal from acidic effluents

TK227 RESIN

The TK227 Resin is based on a diglycolamide (TO-DGA), small amounts of an ionic liquid and a long-chained alcohol. The ionic liquid is added to increase Y retention at lower HNO_3 concentrations. Its main application is the determination of Sr-90 in seawater samples via Y-90 separation and measurement as described by Kim et al.. The published method is a rapid procedure for the extraction and purification of Y-90 from large sea water samples (e.g. 60kg) that allows obtaining results in a few days (including measurement, the actual separation takes less than 4h) instead of several weeks. It thus represents a potential gain of time that very significant.

The TK227 Resin showed better performance than the originally employed DGA, N Resin and can thus replace the latter in the above-mentioned method. An overview over the method can be found below.



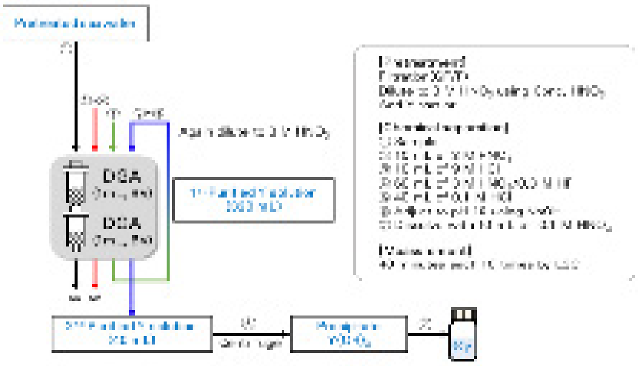
Workflow Sr-90 determination in seawater according to Kim et al.

Typically, 60kg seawater are pretreated (filtration, acidification to 3M HNO_3 (2M HNO_3 for TK227 Resin), followed by the addition of 3 – 4mg Y carrier).

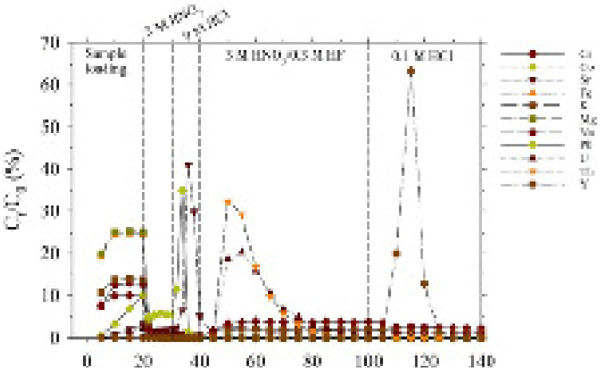
Y is preconcentrated from sea water samples on an equipment specifically developed for this type of applications (SALT-100, WITHTEC Ltd, South Korea) at 80mL/min per channel using a combination of two 1mL DGA or TK227 Resin cartridges per channel (overall 16 cartridges). With eight channels being used the overall flow rate is in the order of 640mL/min.

Once the Y has been concentrated on the cartridges the eight sets of stacked cartridges are moved into a Hidex Q-ARE 100 (Hidex Oy, Finland) system that allows for an automized multi-step purification of the Y.

The respective steps are shown in the following graph, while the subsequent figure gives an overview which impurity is eliminated in which step.



Scheme of the Y separation from seawater



Separation of Y from interferences on DGA

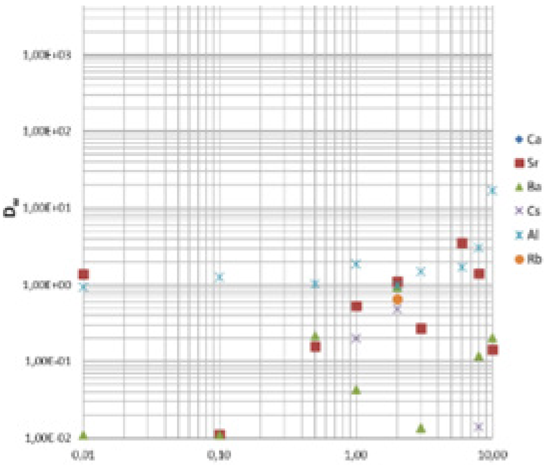
The separation on the Q-ARE 100 is performed at 5mL/min. Y is eluted from the eight channels (16 cartridges) with 40mL 0.1M HCl per channel cartridges (overall 320mL).

To lower the volume of the Y eluate, and to allow further purification, a set of two stacked 1mL DGA cartridges is moved to the SALT-100 unit. The Y eluate (320mL of 0.1M HCl) is adjusted to 3M HNO_3 and loaded onto the cartridges at 80mL/min. The stacked cartridges that are now containing the Y are then moved back to the Q-ARE 100 unit to go through an additional purification cycle (see above) and finally elution in 40mL 0.1M HCl.

The Y-90 sample is prepared for counting by precipitation at pH 10 and redissolution in 10mL 0.1M HNO_3. An aliquot of the sample is taken and analysed by ICP-OES for Y yield determination. The remaining sample is analysed for 10 x 40minutes in Cerenkov mode.

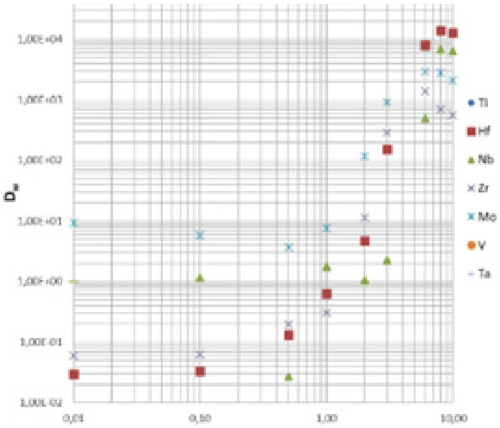
For 50L of seawater and the described counting modalities an MDA of 0.16 ± 0.1mBq/m^3 was reported by Kim et al.. Y yields were typically greater 90%.

The following six graphs show the selectivity of the TK227 Resin for a wide range of elements in HCl and the six graphs following these in HNO_3. All D_W values shown in these graphs were obtained through ICP-MS measurements.



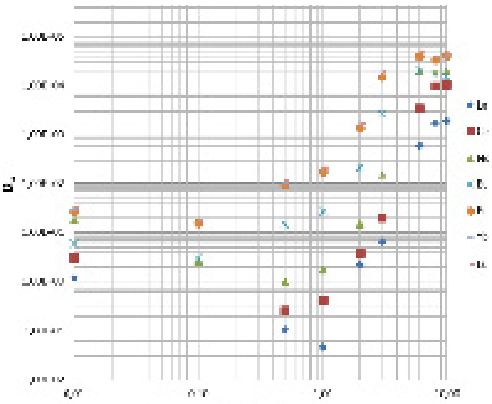
D_W values, TK227, selected elements, HCl

None of the tested elements show significant retention on TK227 in HCl.



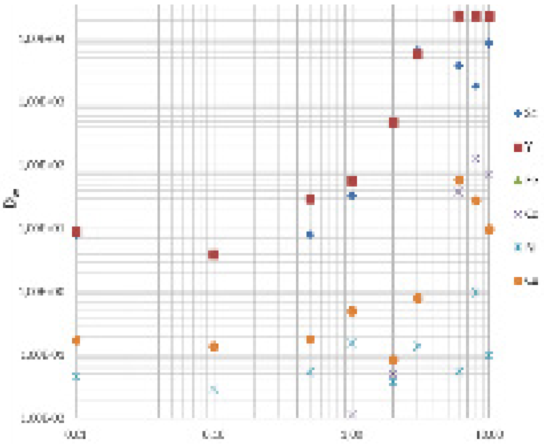
D_W values, TK227, selected elements, HCl

Higher valent elements such as Hf, Zr, Mo and Nb are well retained at elevated HCl concentrations. At lower HCl concentrations the retention decreases quite steeply.



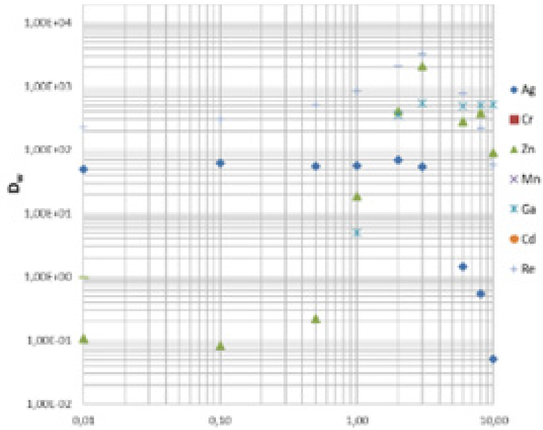
D_W values, TK227, selected elements, HCl

All lanthanides are well retained at high HCl concentrations. To be noted, the retention of the heavy lanthanides is significantly higher than the retention of light lanthanides. In general, an elution of the lanthanides may be performed at low HCl concentrations.



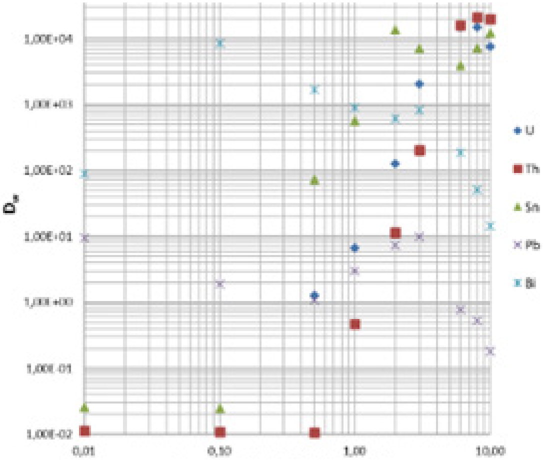
D_W values, TK227, selected elements, HCl

Y and Sc show a behaviour similar to the lanthanides, high to very high retention in high HCl, elution in dilute HCl. Tested transition metals are generally not well retained, with the exception of Cu and Co which show some retention at 6-8M HCl.



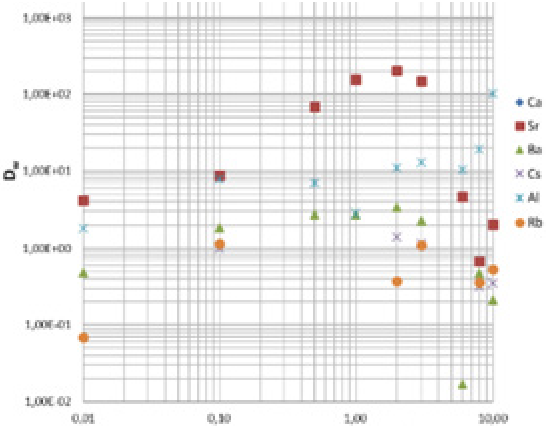
D_W values, TK227, selected elements, HCl

Elements such as Zn, Ga show fairly high retention from 2M HCl onwards. Ag is to a certain extent retained at lower HCl concentrations, while showing strong decrease at high HCl concentrations.



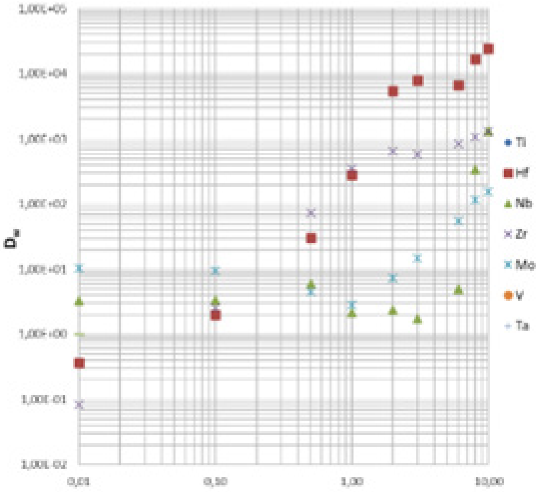
D_W values, TK227, selected elements, HCl

U, Th and Sn show negligeable retention at less then 2M HCl, above this HCl concentration though retention increases strongly. Bi shows contrary behaviour, strong retention at low HCl and decreased retention at high HCl concentration (e.g. 10M).



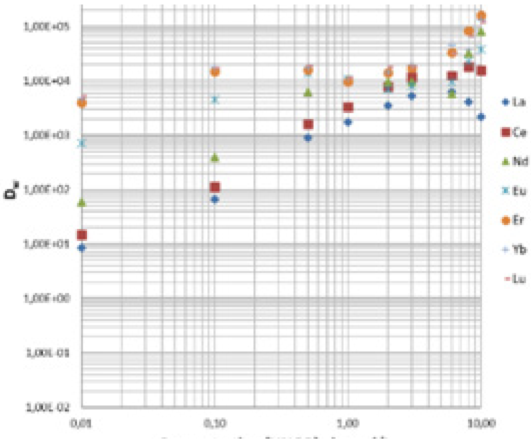
D_W values, TK227, selected elements, HNO_3

Out of the tested elements only Sr is well retained on TK227 between 1M and 3M HNO_3.



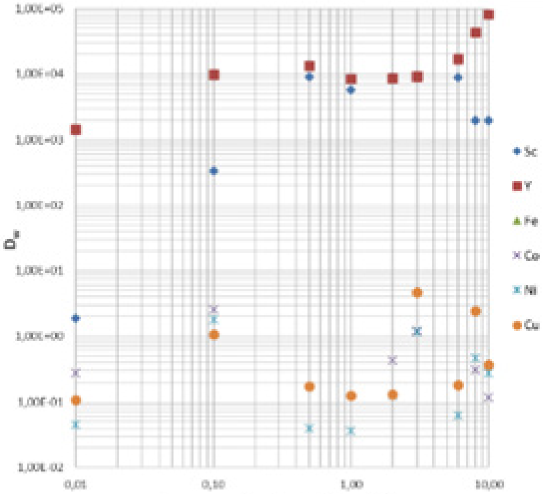
D_W values, TK227, selected elements, HNO_3

Similar to HCl, higher valent elements such Hf, Zr, Nb and to a lesser extent Mo, are well retained at elevated HNO_3 concentrations.



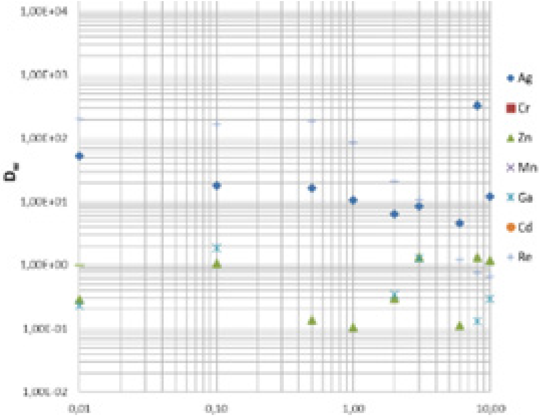
D_W values, TK227, selected elements, HNO_3

All lanthanides are very well retained at elevated HNO_3 concentrations. Heavier lanthanides remain well retained even at low acid concentrations. Overall dilute HCl seems to represent the best option for lanthanide elution.



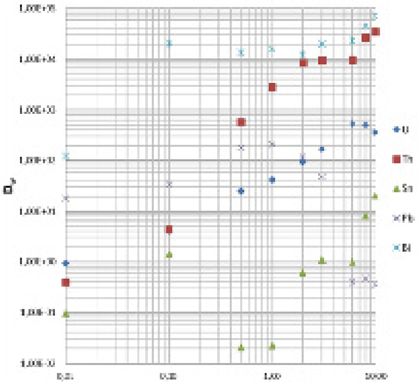
D_W values, TK227, selected elements, HNO_3

Y and Sc are very well retained at high HNO_3 concentrations. While Sc retention decreases at low HNO_3 concentrations, the Y retention remains very high. Indeed, Y requires elution with dilute HCl, similar to the lanthanides. Tested transition metals showed no significant retention in HNO_3.



D_W values, TK227, selected elements, HNO_3

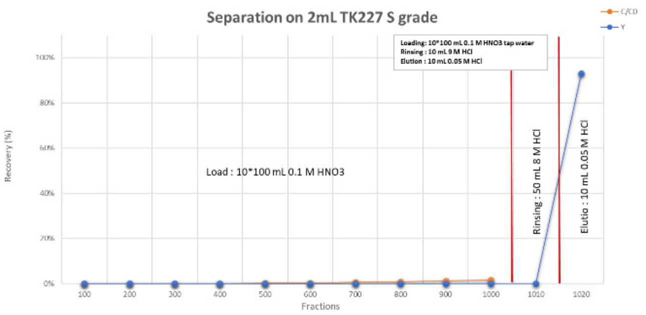
None of the tested elements show strong retention on TK227 from HNO_3.



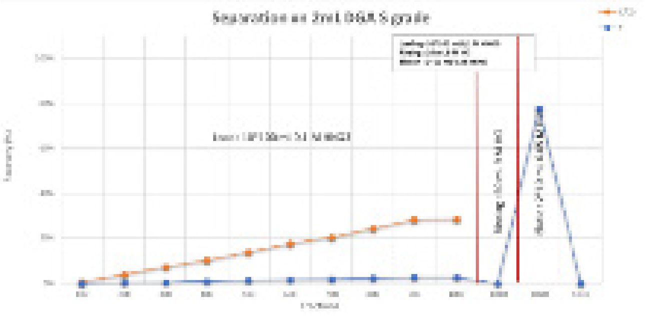
D_W values, TK227, selected elements, HNO_3

U and Th show good to high retention on the TK227 from elevated HNO_3. Bi is overall well retained, even at rather low HNO_3 concentrations. Pb is quite well retained from 0.5 – 1M HNO_3.

The following graphs show the results of elution studies with the aim of concentrating and separating Y from various types of water samples. In these tests 2mL cartridges and 1L water samples acidified to 0.1 – 0.2M HNO_3. Were employed. To show the effect of the ionic liquid added to the composition of the TK227 Resin results are compared to results obtained with DGA, N Resin. Important: it should be noted that Y needs to be present in mg amounts in such experiments, at trace concentrations Y will not be eluted quantitatively.

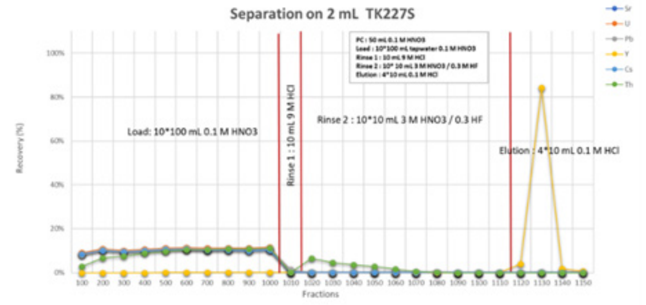


Elution study, 1L tap water, loading from 0.1M HNO_3, Y separation, 2mL TK227 Resin cartridge



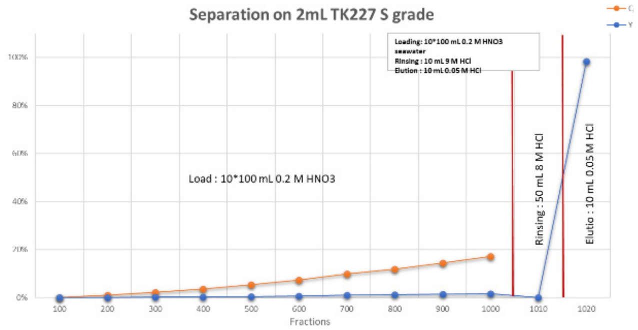
Elution study, 1L tap water, loading from 0.1M HNO_3, Y separation, 2mL DGA, N Resin cartridge

It can be seen that while TK227 Resin allows for quantitative retention of Y from 0.1M HNO_3, DGA, N Resin shows significant breakthrough under these conditions.

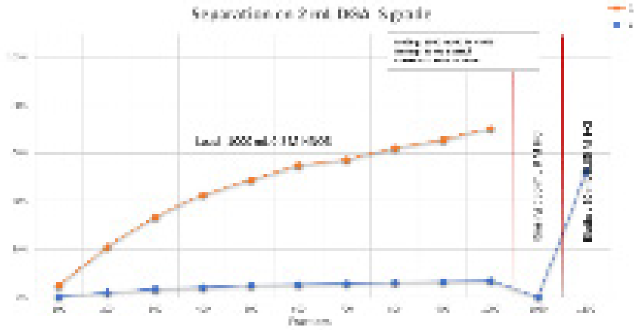


Elution study, 1L tap water, loading from 0.1M HNO_3, Y separation from selected elements, 2mL TK227 Resin cartridge

The elution study above shows that Y can be preconcentrated from tap water (0.1M HNO_3) and then purified. The shown separation scheme allows for the removal of all considered impurities.
Sea water samples show a similar trend to tap water samples. Due to the high matrix load these samples have to be acidified a more strongly (0.2M HNO_3) to achieve elevated yields.



Elution study, 1L sea water, loading from 0.2M HNO_3, Y separation, 2mL TK227 Resin cartridge



Elution study, 1L sea water, loading from 0.2M HNO_3, Y separation, 2mL DGA, N Resin cartridge

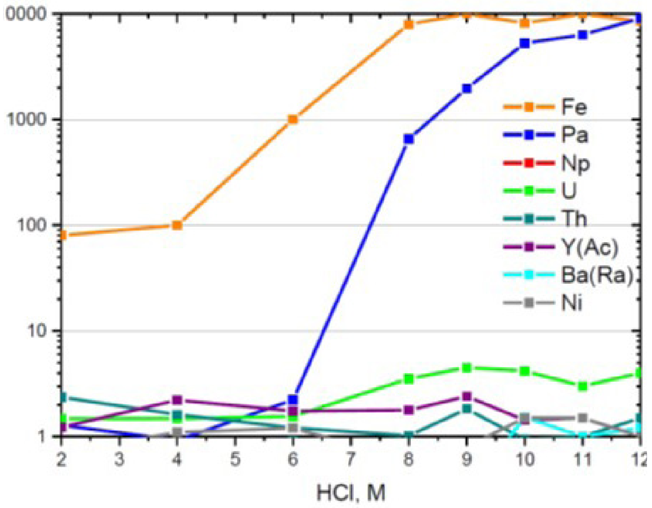
For sea water samples TK227 shows some breakthrough, too – much less than DGA, N though. In order to obtain even higher Y yields for sea water samples the HNO_3 concentration of the sample before load needs to be increased further.

Main applications

- Determination of Sr-90 in large seawater samples via Y-90

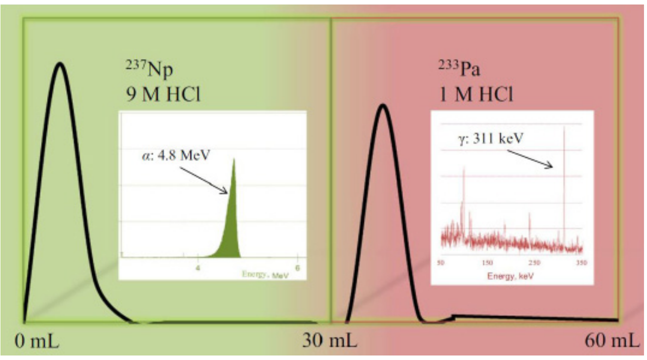
TK400 Resin

The TK400 Resin is an extraction chromatographic resin that is comprised of octanol impregnated onto an inert support. Knight et al. showed that long-chained alcohols, especially octanol, show very interesting selectivity towards Pa at high HCl concentrations, allowing for facile Pa/Np separation using column chromatography. Jerome and al. characterized a number of elements including Pa, Np, U and Th.



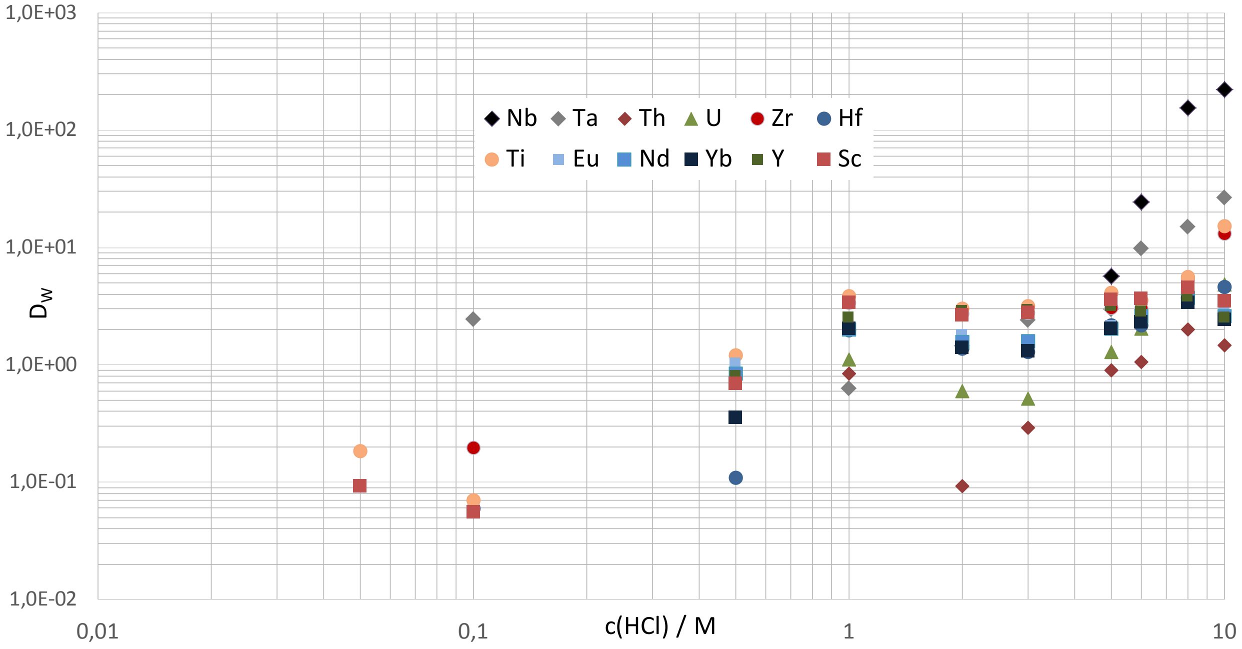
D_W values of selected elements on TK400 Resin in HCl at varying concentration [Data provided by Ivanov et al]

They found that Pa retention sharply increases at high (≥ 9M) HCl concentrations whereas other elements tested are not retained. At HCl concentrations < 8M HCl on the other hand D_w values of Pa were found to be low allowing for its elution in a small volume. Ostapenko et al. found a similar trend for Pa retention with k’ values being high for Pa at high HCl concentrations (9M). These results correspond overall well to the selectivity observed by Knight et al. when performing Np/Pa separation.

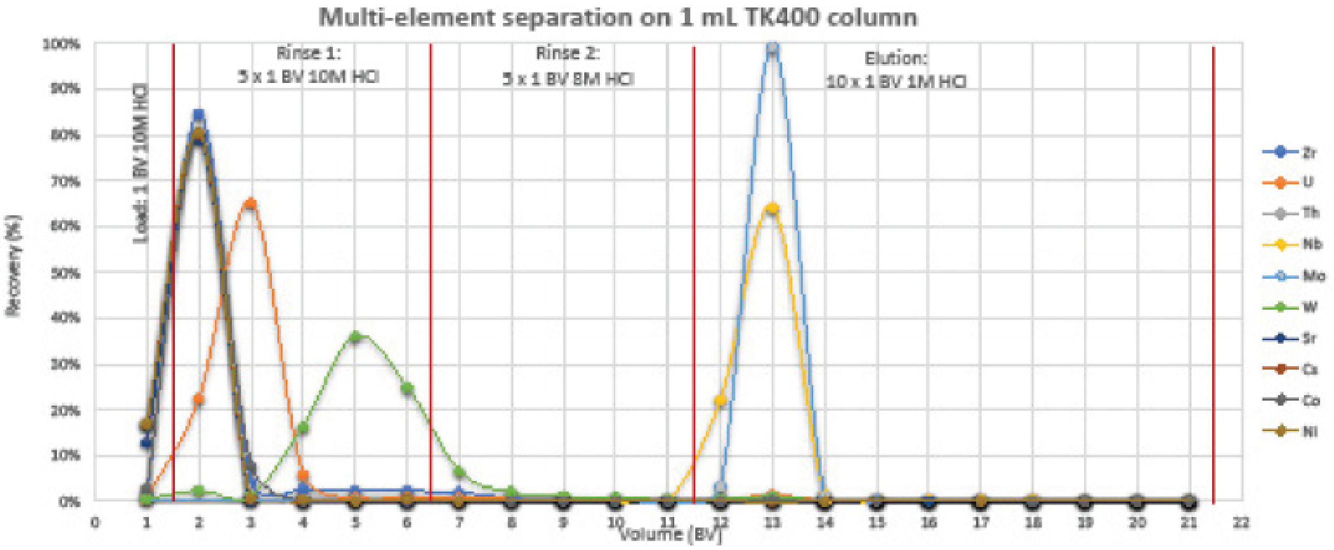


Elution study, Np/Pa separation on long-chained alcohol resin [Taken from Knight et al.]

The resin shows high selectivity for Nb at high HCl concentrations over other elements tested such as Ta, Zr, Hf and lanthanides which are not, or only very poorly as in the case of Ta, retained by the resin.

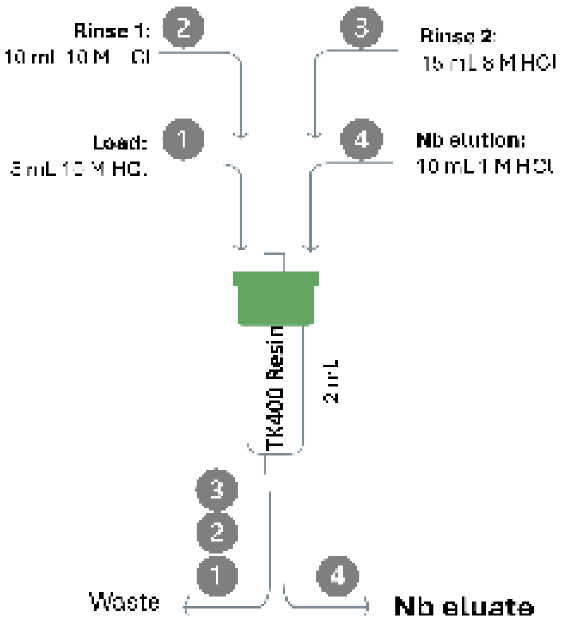


D_W values of selected elements on TK400 Resin in HCl at varying concentration

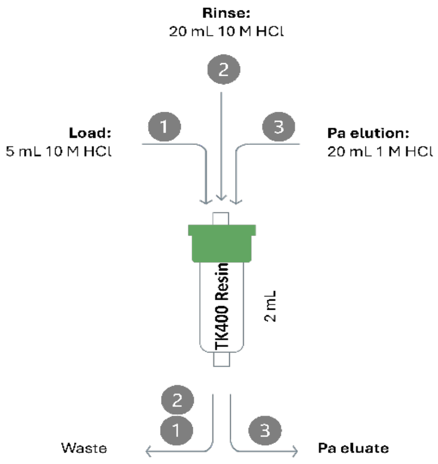


Elution study, Nb separation from selected cations, 2mL TK400 column

With respect to its selectivity the TK400 Resin shows the potential for allowing a number of interesting separations such as Nb/Zr and Pa/U/Th. The results of an elution study on the separation of Nb from a number of elements, including Zr, and the separation method used to obtain these results are shown in following figures. Jerome et al. employed the TK400 Resin for the separation of Pa from its descendants following the procedure shown here. They found that U, Th, Ac, Ra and Pb were removed from the resin during load and rinse, allowing for obtaining a clean Pa fraction with high chemical yield (environ 83%).



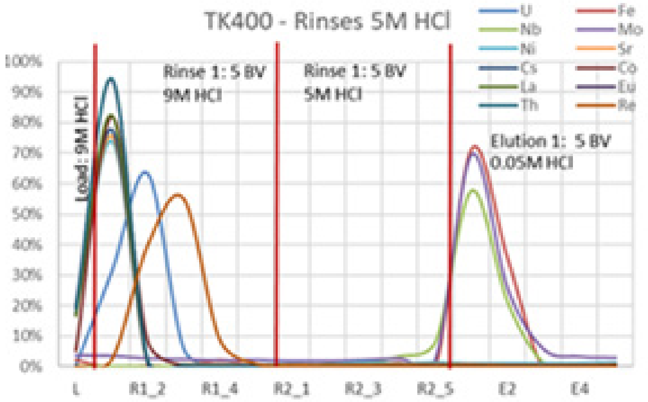
Nb separation on TK400 Resin



Pa separation from its descendants

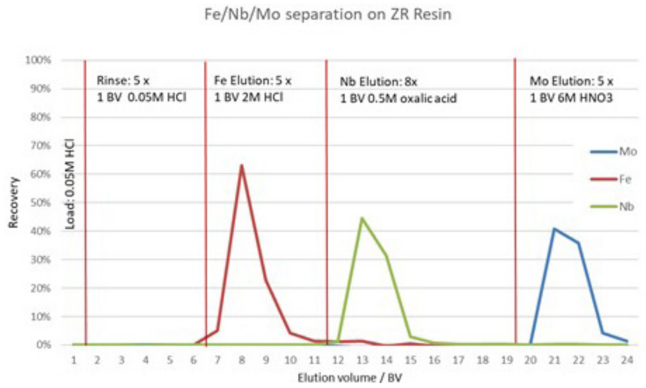
Another interesting application of the TK400 Resin was described by Tieu et al. and Svedjehed et al. Both authors used the TK400 for the separation of Ga-68 from irradiated solid Zn targets. Svedjehed et al. showed that it is advantageous for solid targets to use the TK400 instead of ZR Resin in combination with A8 and TK200.

The fact that the TK400 Resin shows higher Fe capacity than e.g. TRU Resin makes its use in the analysis of i.e. decommissioning samples interesting. A method combining the use of the TK400 Resin (separation of Fe, Nb and Mo from most matrix elements) and ZR Resin (subsequent separation of Fe from Nb and Mo) is currently being optimized. The following graphs show typically obtained elution profiles.



Fe/Nb/Mo separation from selected elements, TK400 Resin

As may be seen, while Fe, Nb and Mo are well retained a large number of other elements, such as e.g. Zr, U, Th, Cs, Co,... are removed during load and rinse. These three elements may then be eluted in dilute HCl and directly loaded onto ZR Resin for further separation.

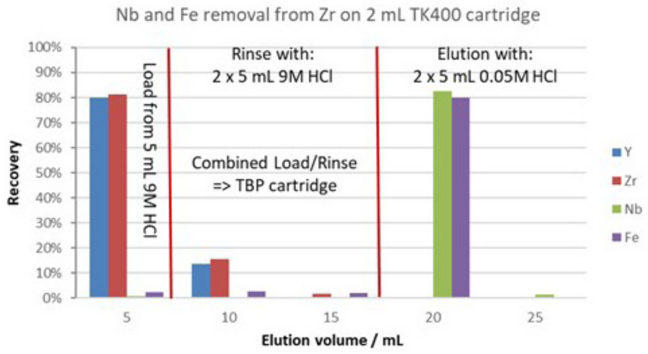


Fe, Nb and Mo separation on ZR Resin

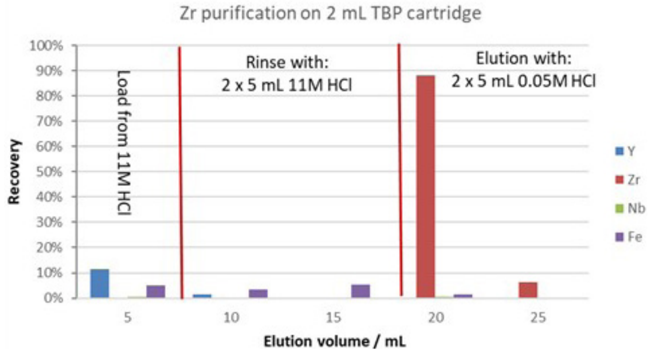
The fact that the TK400 Resin shows high selectivity for Fe and Nb but not for Zr may also allow its use in the separation of Zr-89 from solid Y targets, with the aim to further lowering the amount of these impurities in the final product.

First tests showed that loading a simulated dissolved target solution containing Zr, Y, Nb and Fe through a TK400 Resin cartridge at 9M (or 10M) HCl, followed by a rinse under the same conditions will allow retaining Nb and Fe on the TK400 while Zr (and Y) will pass through. Combining the load and rinsing fractions containing Zr, adjusting them to 11M HCl and loading this solution through a TBP Resin cartridge (similar to the method described by Graves et al. will then allow a clean Zr separation with high chemical yield. It should be noted that alternatively 10M HCl may be used as loading condition on both cartridges. This could simplify the separation, as the intermediate HCl concentration adjustment is not necessary. It might further allow for the use of stacked cartridges in the initial loading step, this modification will require further testing though. Zr is finally eluted from the TBP cartridge using dilute HCl.

If desired, Nb and Fe may be recovered from the TK400 cartridge using dilute HCl. The graphs below show typically obtained elution studies under the described conditions.



Nb and Fe removal from Zr (and Y) on a 2mL TK400 Resin cartridge



Zr purification on a 2mL TBP Resin cartridge

Main applications

- Separation of protactinium
- Separation of gallium
- Separation of iron
- Separation of niobium

Guard Resin

The Guard Resin is a hydrophobic, highly crosslinked, porous polydivinylbenzene based adsorbent. Due to its high hydrophobicity it will remove certain organic impurities, notably organic impurities that are hydrophobic, more efficiently than e.g. the Prefilter Resin. The Guard Resin is generally used in reversed phase chromatography and solid phase extraction, and for the adsorption of biomolecules of up to 14kDa. It has a surface area of > 600m²/g and a typical porosity in the order of 300 – 500 Å. The resin shows high mechanical and chemical stability, and it may be used over the whole pH range.

The Guard Resin is TSE/BSE/GMO free.
Another application of the Guard Resin is the separation, in combination with the ZR Resin, of Ge-68 from GaNi or GaCo targets. The actual separation of Ge from the target material is performed on two consecutive ZR Resin cartridges. The Guard Resin may then be used in the final step of the purification, namely the conversion of final product Ge-68 from dilute citric acid to dilute hydrochloric acid.

Main applications

- Removal of organic impurities
- Ge-68 (in combination with ZR Resin)

Prefilter Resin

Prefilter Resin is a hydrophilic, macroporous, acrylic ester polymeric resin with a high surface area and a high capacity for various organic compounds.

The resin is mainly used for the removal of organic impurities from aqueous solutions, such as colourizing agents to avoid quench effects in liquid scintillation counting, or for the removal of co-eluted extractants from column or cartridge eluates which might interfere with electrodeposition.

Main applications

- Removal of organic impurities

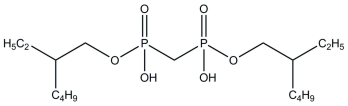
AC Resin

AC Resin contains bis(2-ethylhexyl) methane-diphosphonic acid ($H_2DEH[MDP]$, DIPEX®) as extractant.

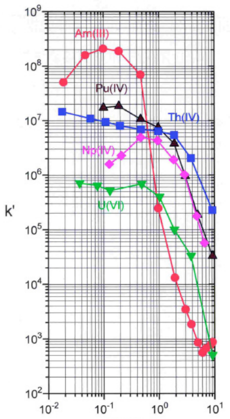
The Actinide Resin (AC Resin, in the literature also referred to as DIPEX® Resin) is mainly used for the preconcentration and separation of actinides from acidic solutions obtained from environmental samples (soil leachates and large volume water samples) .

The AC Resin shows a higher affinity for actinides in general and in particular a better selectivity for americium, compared to its ion exchange analogue Diphonix®. This high affinity is also used for the determination of the gross alpha activity of urine and water samples via liquid scintillation counting.

The same resin is also used for the separation of beryllium from environmental and industrial matrices. It is used to purify Be from other elements that could interfere with its determination via ICP-AES. Be is strongly retained on the AC Resin between pH 1 and pH 2 and can be eluted using nitric acid of a concentration greater than 1M.



DIPEX® extractant



Acid Dependencies of the uptake of several Actinides by AC Resin

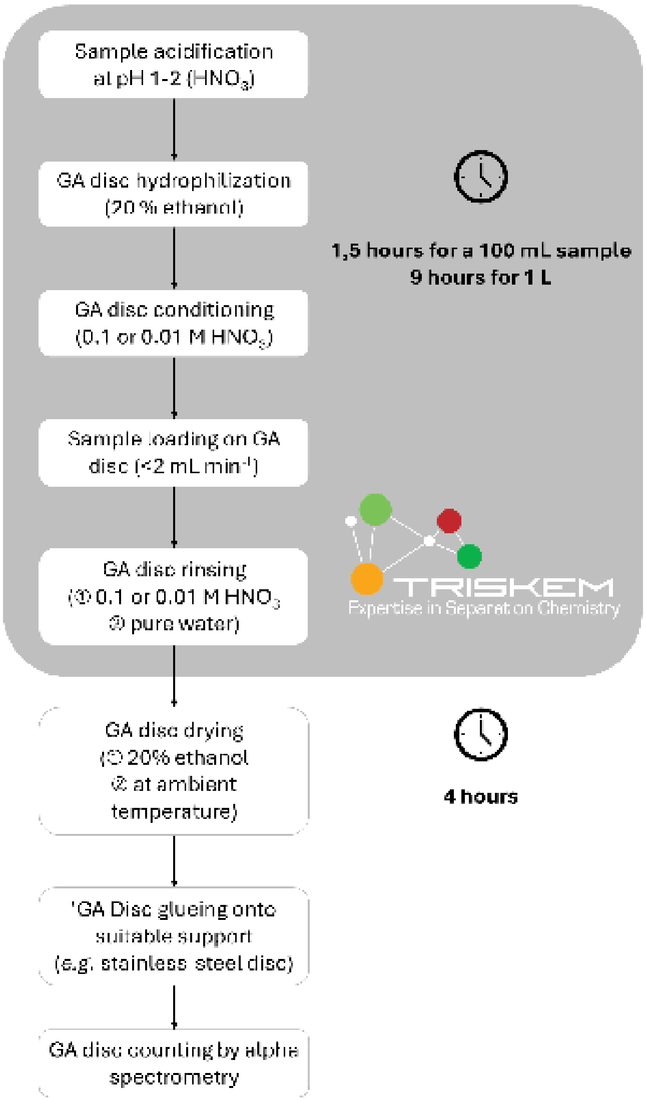
Main applications

- Gross-alpha activity in water and urine samples
- Preconcentration of actinides from environmental samples
- Separation of Be for ICP-AES measurement
- Separation of Mn from Cr targets

TK-GA DISCS

The TK-GA (Gross Actinides) Discs are specialty membrane filters impregnated with an extractant system that shows very strong retention of actinides from acidified (pH 1 or 2, for some actinides even up to 3M HNO_3) water samples. They are the first product of a new range of such impregnated membrane filters ('Discs'). It's main application is the screening for actinides in aqueous samples via alpha spectrometry [Bailly et al.]. Other potential applications of such Discs include passive sampling [Wagner et al.] or analyte preconcentration via filtration [Horstmann et al.].

An outline for a typical actinide screening procedure in aqueous samples is shown below.



Sample preparation scheme TK-GA Discs for alpha spectrometry

The aqueous sample is first acidified to pH 1 or 2 and, in case this is necessary, filtered to remove fine particles that could interfere with the alpha spectrometric measurement. The TK-GA Disc is then preconditioned with 20% EtOH (hydrophilization step) followed by rinses with water and 0.01M or 0.1M HNO_3 (for Th e.g. it is preferable to work at pH 1).

The aqueous sample is then filtered through the TK-GA Disc, for best recovery and peak resolution of the alpha spectrum the filtration should ideally be performed at ≤2mL/min.

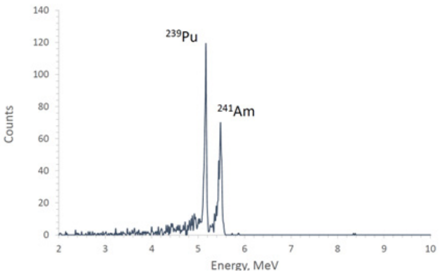
It should be noted that the TK-GA Discs are available in two diameters: 25mm and 47mm. 25mm discs are typically used for up to ~100mL samples, the 47mm discs for up to 1L samples.

Once the sample has been completely passed through the Disc it is subsequently rinsed with dilute acid, water and 20% EtOH, and air dried.

The dried disc is then glued onto a suitable support (e.g. stainless steel disc), and is then introduced into an alpha spectrometry system for measurement.

The following graphs show some examples of alpha spectra obtained using the TK-GA Discs.

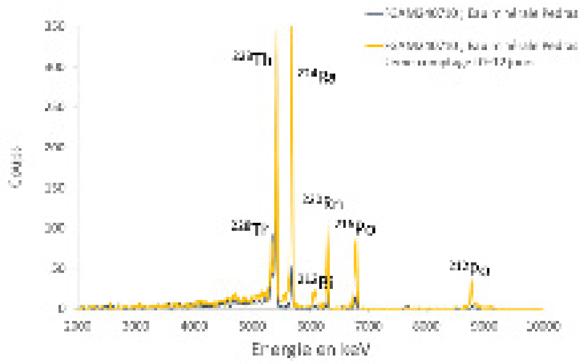
All shown spectra were obtained by Bailly et al. as part of the LabCom 'TESMARAC' cooperation.



Alpha spectrum, Am-241 & Pu-239, each ~50mBq, 100mL pH 2 HNO_3

As can be seen, the spectral resolution of the Am and Pu peaks are good to very good (30 – 100keV) for a filtration based sample preparation. Typically 80 - >90% of the respective actinides are detected by alpha spectrometry, indicating that the actinides are to a very high extent fixed on the surface of the Discs.

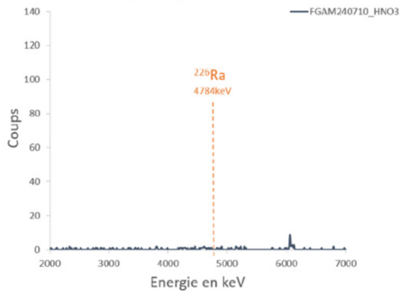
The following fig. shows that Th is very well retained from acidified Pedras mineral water (acidified to ~0.1M HNO_3), Ra on the other hand is not retained, and Po only weakly. The third figure further shows the alpha spectrum of Pedras water obtained directly after filtration, and after 12 days of ingrowth of its daughters.



Alpha spectrum, 47mm TK-GA Disc,
Pedras mineral water, 100mL pH 1.25

The fig. below shows the alpha spectrum of a Ra-226 containing solution at 0.01M HNO_3.

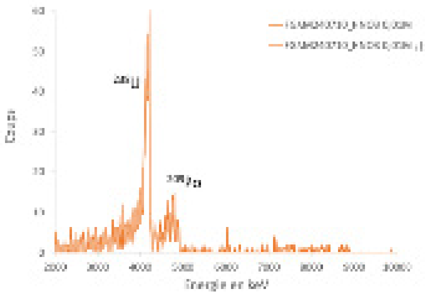
It could be confirmed that Ra-226 is not showing any signal in the alpha spectrum, it is indeed not retained as indicated in the Pedras water test. In an additional experiment at pH 4 it could be shown that while Ra is retained on the disc at this pH again no signal could be detected in the alpha spectrum, indicating that Ra is retained within the disc, and not on the surface.



Alpha spectrum, 47mm TK-GA Disc,
100mL Ra-226 containing solution, 0.01M HNO_3

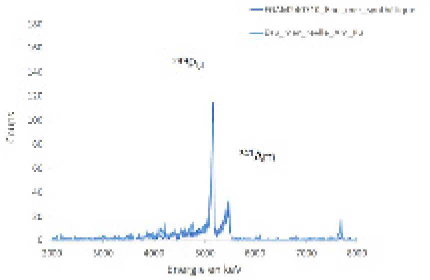
A similar test was performed with a solution containing U-238 and Po-209 in 0.01M HNO_3. While U is very well retained, Po is only showing partial retention.

Here, the influence of Ca especially on the U retention was tested by running the test also at elevated Ca concentrations (0.1g/L), no adverse effect was found to be induced by Ca.

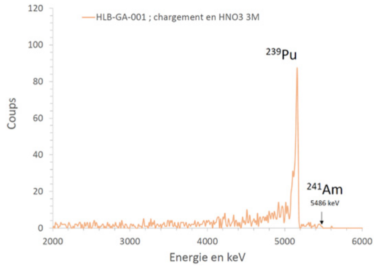


Alpha spectra, 47mm TK-GA Disc,
100mL U-238 and Po-209 containing solutions, 0.01M HNO_3

Pu is even strongly retained from high matrix samples such as sea water and highly acidic samples such as 3M HNO_3. Under the same conditions Am is distinctively less well (sea water) or not retained (3M HNO_3).

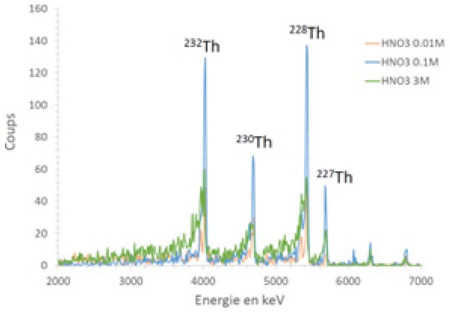


Alpha spectrum, 47mm TK-GA Disc, 100mL natural and syntethic sea water samples containing Am and Pu, acidified to pH 2



Alpha spectrum, 47mm TK-GA Disc, 100mL solution containing Am and Pu, acidified to 3M HNO_3

Th is also retained from 3M HNO_3, not quite as well as from pH 1 or 2 though, and the peak resolution is distinctively less sharp.



Comparison of alpha spectra obtained with 47mm TK-GA Discs for Th solutions of varying acid concentrations (0.01M, 0.1M and 3M HNO_3)

Main applications

- Screening/alpha spectrometry measurement of actinides

TK-TcScint

The TK-TcScint Resin is the first of a range of resins based on “Impregnated Plastic Scintillation microspheres” developed by García, Tarancón and Bagán at the University of Barcelona.

This range of new products will be comprised of plastic scintillation microsphere (PSm), supplied by the group at the University of Barcelona, that are impregnated with selective extractants.
The TK-TcScint Resin is, as the name indicates, mainly dedicated to the quantification of Tc-99. The extractant used in its fabrication is Aliquat 336, it further contains small amounts of a long-chained alcohol. Accordingly, its selectivity will generally be very similar to the TEVA Resin.

The figures below (a and b) show SEM pictures of the non-impregnated PSm, and the impregnated microspheres (TK-TcScint), respectively.

The TK-TcScint is generally employed as pre-packed 2mL cartridges for use with vacuum box systems, or automized separation equipments based on pump systems

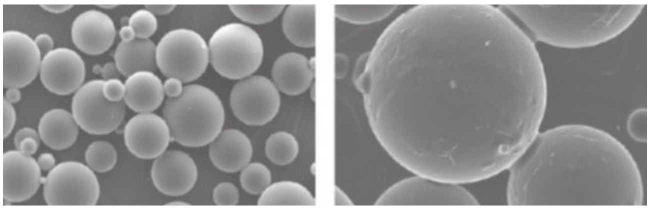


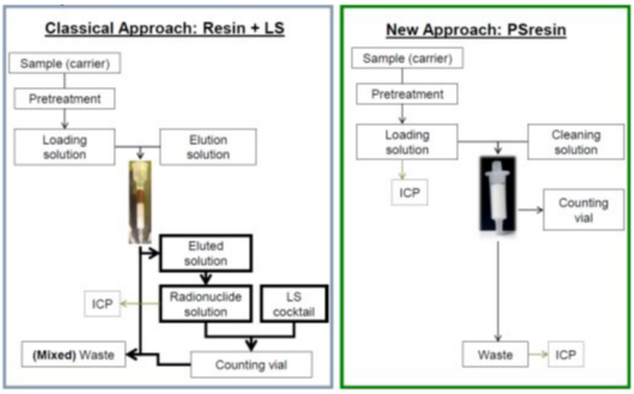
Figure1 (a and b): left - non-impregnated plastic scintillation microspheres (PSm), right - impregnated PSm (TK-TcScint). Taken from Garcia et al.

The PSm support employed in the TK-TcScint Resin is itself acting as scintillating medium, this allows for a direct measurement of the Tc-99 retained on the cartridge, no elution and mixing of the eluate with a liquid scintillation cocktail is necessary.
This has a number of advantages:

- Gain of time which is particularly interesting in emergency situations
- No mixed liquid radioactive waste
- No Tc elution with HNO_3 of elevated concentration and no evaporation / aliquoting of the eluate
- No cutting of columns or cartridges to push the resin into LSC vials

Especially the latter two points are interesting in terms of radiation protection when samples of elevated activity are being analyzed.
Ideally the chemical yield is determined via ICP-MS or ICP-OES using Re as internal standard.

The following graph compares this new approach based on impregnated PSm Resins such as the TK-TcScint with classical methods.
In order to easily handle and avoid contaminating the LSC counter the cartridges should be placed in a standard 20mL LSC vial for its measurement.

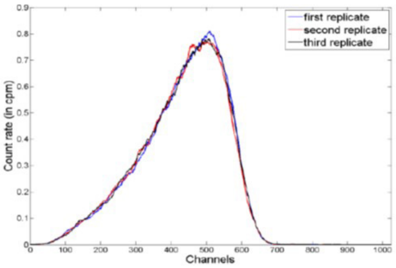


Overview classical radioanalytical method and PS Resin approach. Taken from Garcia et al.

Typical samples analysed include urine and various types of water sample. In case of surface water samples generally a breakthrough volume of >200mL can be achieved using 2mL cartridges, making this technique not only interesting in emergency situations or as screening tool in decommissioning, but also for use in routine biomedical or environmental monitoring. For water samples the chemical yield is generally >98.8%.

The detection efficiency for Tc-99 obtained with the TK-TcScint is very high, in the order of 89.5(0.6)%, while the background of the standard 2mL cartridges is low with ~1.09CPM (obtained in Quantulus™ detector in the high-energy and low-coincident bias configuration).
Further the TK-TcScint cartridges show reproducibly low quench with a mean SQP(E) of 787(7).

The figure below shows typically obtained Tc-99 spectra, as can be seen spectra obtained for three replicates match very well.



Liquid Scintillation Spectrum of Tc-99 on TK-TcScint. Taken from Garcia et al.

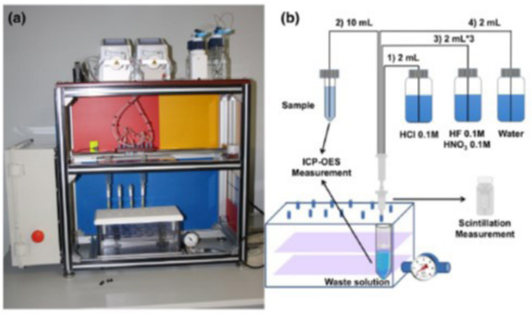
The analysis of water samples such as e.g. river and sea water (typically 50mL) using TK-TcScint cartridges is rather straightforward

After filtration, if necessary, samples are heated to 90°C for 60min after addition of a few mL of 30% H_2O_2 to assure that Tc is present as pertechnetate. The solutions are then adjusted to 0.1M HCl using conc. HCl. Once the samples reach room temperature, they are ready for separation.
After loading of the sample the cartridge is typically rinsed successively with 0.1M HCl, 0.1M HNO_3/0.1M HF (only necessary in case Th is expected to be present) and finally water.
These rinses allow eliminating possible interferences while Tc (and the internal standard Re) remain on the cartridge. Load and rinse fractions are combined and analysed for Re content to allow calculating the chemical yield of the separation. The TK-TcScint cartridge can then be directly counted on an LSC counter.
The authors found very good match between expected and measured activities, for the spiked water samples as well as for two spiked MAPEP samples.

Using 50mL samples and 180min counting time allowed the authors obtaining a limit of detection of 0.15Bq/L.

As could further be shown by the authors, this method can easily be automized. In their case they developed their own separation unit called OPENVIEW-AMSS, a modular, vacuum box based equipment. They could show that both, manual and automized separations allow for obtaining high chemical yields and detection efficiencies, no significant differences were observed when analysing samples in parallel. However, with respect to hands-on time and radiation protection automatization provides significant advantages.
Further to the unit developed by the authors the TK-TcScint cartridges are also compatible with commercially available equipment such as the Hidex Q-ARE 100

Besides water samples urine samples were also analysed using TK-TcScint cartridges.



OPENVIEW-AMSS system and scheme of a typical separation of water samples. Taken from Coma et al

With respect to their higher matrix load this kind of samples requires a thorough sample pre-treatment. The described method is based on 100mL urine samples that are first wet-ashed using conc. HNO_3, followed by an additional ashing step in a muffle furnace at 550°C.
The obtained ash is then dissolved in 3mL of conc. HNO_3 and diluted to 100mL using deionized water. To assure Tc is present as pertechnetate the solutions are heated, after addition of a few mL hydrogen peroxide, to 90°C for 60min. As described before, Re was used as internal standard.
By analysing spiked urine samples the authors could show that accurate results can be obtained using the impregnated PSm approach. A minimum detectable activity (MDA) of 0.036 Bq/L for 100mL samples and 24h counting was reported.

Further to the analysis of Tc-99, PSm Bagán et al. showed that Aliquat 336 impregnated resins may also be used for the analysis of $[^{14}C]SCN^-$ used as radiotracer for study of oil reservoir dynamics.

With respect to the selectivity of the Aliquat extractant, the compound giving TEVA Resin its selectivity, a use of the TK-TcScint cartridges for the screening of other radioelements such as e.g. Pu isotopes or Po-210 seems well possible.

Main applications

- Separation and LSC measurement of technetium

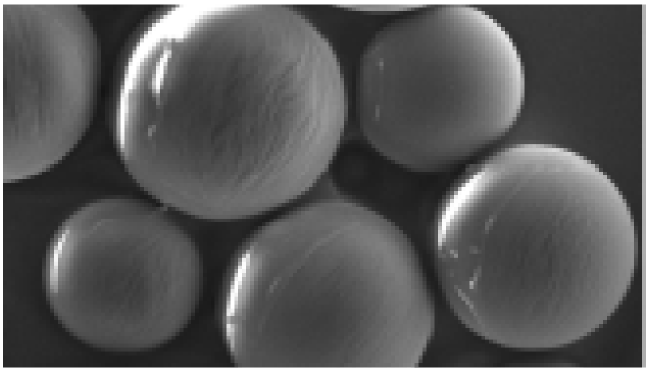
TK-SrScint Resin

The TK-SrScint Resin is, like the TK-TcScint, a resin based on “Impregnated Plastic Scintillation microspheres” developed by Tarancón, and Bagán at the University of Barcelona.

These new materials consist of plastic scintillation microspheres (PSm), supplied by the group at the University of Barcelona, which are impregnated with selective extractants.

The TK-SrScint Resin is designed for use in similar separation methods to those employing SR or TK102 Resins. The selective extractant used in the production of this PS resin is a crown ether (also used in SR Resin) dissolved in a fluorinated alcohol (used in TK102 Resin). Consequently, its selectivity will generally be very similar to that of the aforementioned resins, which are primarily used for Sr or Pb separations.

The figure below shows SEM pictures of the impregnated microspheres (TK-SrScint).



Impregnated PSm (TK-SrScint). Courtesy of Tarancon et al.

The TK-SrScint is available as pre-packed 2mL cartridges for use with vacuum box systems or systems that can be easily automated using pump systems.

As with the also available TK-TcScint Resin, the PSm support acts as a scintillating medium, allowing for direct measurement of the isotope retained on the cartridge.

Thus, there is no need to elute the target radionuclide and mix the eluate (i.e. radioactive solution) with a liquid scintillation cocktail (i.e. organic product). Incorporating this PS resin thus helps to reduce the amount of mixed (radioactive + organic) waste generated during the determination procedure.

This has a number of advantages:

- Less hands-on time consumed which is particularly interesting in emergency situations
- No mixed liquid radioactive waste
- No Sr, Pb or Ba elution required and no evaporation / sample preparation of the eluate
- No cutting of columns or cartridges to push the resin into LSC vials

Especially the latter two points are interesting in terms of radiation protection when samples of elevated activity are being analyzed.

Ideally the chemical yield is determined via ICP-MS or ICP-OES using Sr as internal standard (comparison between the initial amount added/ present and the strontium not retained on the PS Resin).

Like for the TK-TcScint it is advisable, to facilitate handling and avoid contaminating the LSC counter, that the cartridges are placed in a standard 20mL LSC vial for its measurement.

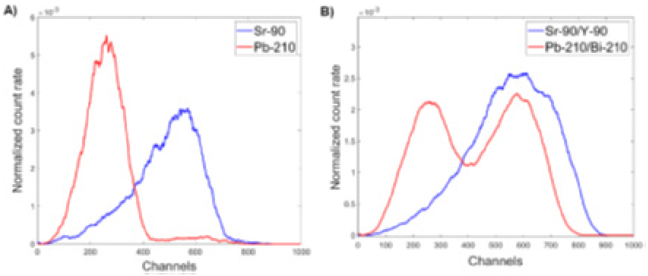
The TK-SrScint Resin has been tested in various water samples, including river, MAPEP, and CSN interlaboratory samples. Results were compared with the two most common methods for Sr-90 determination: extraction chromatography using SR Resin combined with LSC, and successive precipitations combined with LSC. Sr-90 activity evaluated using the different methods ranged from 0.49-4.9Bq/L in spiked river water samples, to 5.65-10.48Bq/L in MAPEP interlaboratory samples, and 4.1Bq/L in CSN interlaboratory samples. Moreover, in CSN samples, interferences such as Co-57, Co-60, Cs-134, Pu-238, Ra-226, Pb-210, and Ra-228 were evaluated.

Prior to applying the PS method using TK-SrScint Resin, various precipitation methods to remove Pb from the sample were investigated. Iodate precipitation was selected as the most suitable method for Pb removal, and further optimization was performed to increase Sr recovery while decreasing Pb presence (~3% remaining). The final procedure included precipitating Pb by adding iodate and calcium as coprecipitating agents and boiling of the sample. Once Pb was removed, Sr was precipitated using hydrogen phosphate in a basic medium, and the resulting precipitate was dissolved with 8M HNO_3 for loading the sample onto the TK-SrScint Resin.

After analyzing water samples spiked with CSN, Sr chemical yields of approximately 87% were achieved. The analysis considered a background signal of 0.18CPM, and no other radionuclides, including Pb-210, were detected in the samples.

For larger samples, such as 1L spiked river water samples, a carbonate precipitation step was added to reduce sample volume. In these cases reported chemical recoveries ranged between 63% and 81%.

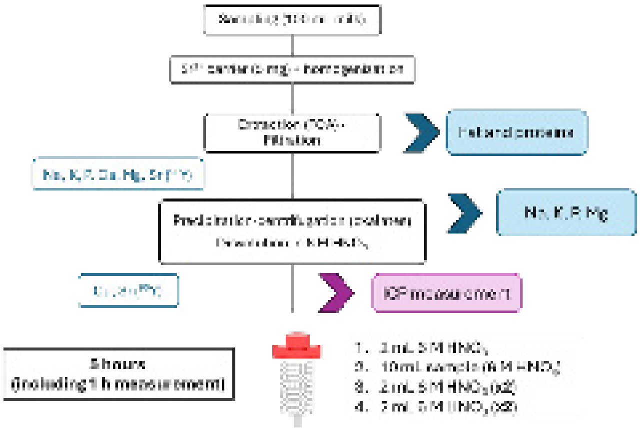
LS spectra of the target radionuclide (Sr-90) and the potential interference (Pb-210) are shown below at different times to account for Y-90 ingrowth.



Liquid Scintillation normalized Spectra of Sr-90/Y-90 (blue) and Pb-210/Bi-210 (red) on TK-SrScint at time 0 (A) and with ingrowth daughters at time >21 days (B). Courtesy of Tarancon et al.

The detection efficiency of Sr-90, evaluated at time 0, was found to range between 86% and 51% for complete and optimal window settings, respectively. The optimal window was selected to minimize the possible contribution of Pb-210 to the LS spectrum. A lower background signal of 0.3CPM, compared to standard methods, was achieved with TK-SrScint resin, resulting in a lower detection limit of 27 mBq/L (1h counting time). As mentioned earlier, the inclusion of PS Resin in the Sr-90 determination method reduced the procedural turnaround time to 5-6 hours.

Determination of Sr-90 in milk samples has also been investigated by Tarancón et al. Their tests resulted in the detection of approximately 65% of the total Sr-90.



Sr-90 determination procedure in milk samples. Courtesy of Tarancon et al.

According to the results obtained by Tarancón et al. (2024) when testing IAEA milk powder samples, the Sr-90 activity quantified using the TK-SrScint Resin was in agreement with the reference activity. Moreover, interferences such as Ca, Na, and K were shown to be removed by observing a clear Sr-90/Y-90 spectrum and through measurements taken with ICP.

Main applications

- Separation and LSC measurement of strontium (Sr-89/90)

MnO_2- PAN Resin

The analysis of Ra isotopes in water samples is becoming more and more important. To meet existing requirements on detection limits, especially for Ra-228, elevated samples volumes have to be used for analysis. MnO_2 Resin allows to preconcentrate Ra isotopes efficiently from water samples, drinking water as well as sea water.

MnO_2 Resin is currently used with LN Resin and DGA Normal resin in a method developed by Sherrod Maxwell of Westinghouse Savannah River. In his method the MnO_2 is used to pre-concentrate Ra from 1 to 1.5L water samples, with 1.25g/L of MnO_2 resin used per sample.

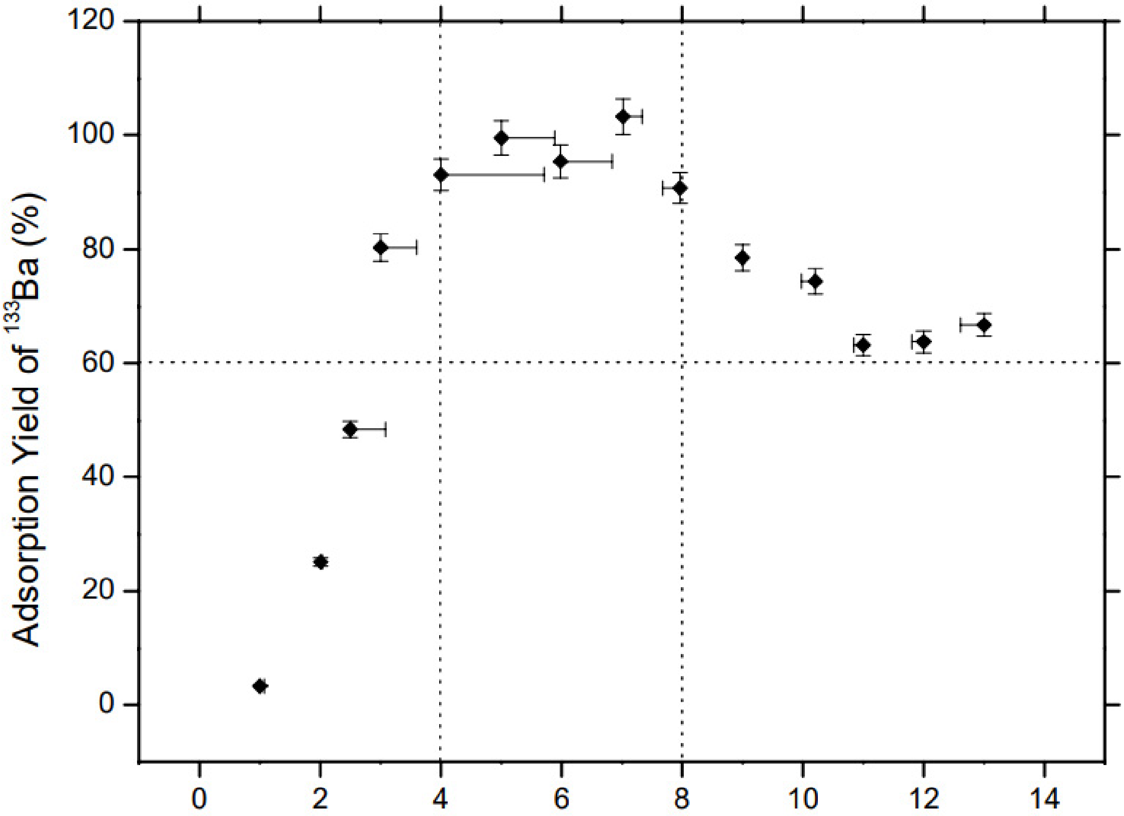
Ra is then eluted and loaded (in case Ra-228 is to be determined after a minimum of 36 hours for Ac-228 ingrowth) onto DGA, Normal Resin (retention/separation of Ac-228). Ac-228 is eluted from the DGA Resin and finally micro-precipitated with CeF_3 and counted via GPC; the precipitated source can be used for gravimetric yield determination. Ra-226 can be determined, directly or after additional clean-up via LN Resin, by $BaSO_4$ micro-precipitation and alpha spectrometry.

TrisKem International is providing the MnO_2-PAN resin (G2 Resin) developped by Dr. Šebesta (ČVUT).

It consists of very fine MnO_2 particles embedded into a modified polyacrylonitrile (PAN) binding polymer offering a very stable MnO_2 resin with a very high surface area.

Main applications

- Determination of Ra-226/8 in water samples



Absorption of Ba-133 on MnO_2 Resin

Cs Resins

Both resins, AMP-PAN and KNiF-PAN have been developed by Dr. Šebesta from the Czech Technical University in Prague (CVUT). Like the MnO_2-PAN resin both resins are based on very fine and selective inorganic materials embedded in an organic matrix based on polyacrylnitrile (PAN) in order to improve their mechanical characteristics. The active components are the widely employed ammonium phosphomolybdate (also Ammonium MolybdoPhosphate, AMP) and potassium nickel hexacyanoferrate(II) (also potassium Nickel FerroCyanate, KNiFC).

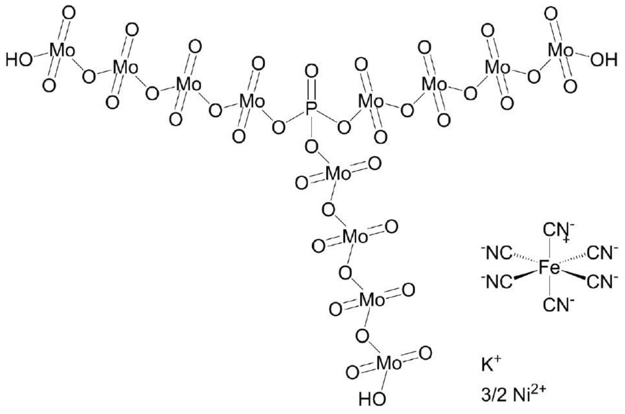
Both resins are used for the concentration and separation of Cs from various liquid samples.

Ammonium phosphomolybdate is an inorganic ion exchanger known for its high selectivity for Cs even at elevated acid concentrations, quick kinetics and radiation stability.

Its high selectivity for Cs even under harsh chemical conditions and high levels of radioactivity make the AMP-PAN resin a candidate resin for the treatment of radioactive waste solutions. Brewer et al. tested the resin for the removal of Cs-137 from real and simulated acidic high-active liquid radioactive waste containing high amounts of potassium and sodium.

AMP-PAN's robustness against high salt concentrations also makes it interesting for use in environmental analysis, especially the analysis of Cs-134/7 in sea water. Kamenik et al. evaluated AMP-PAN and KNiFC-PAN resins for use in Cs-134/7 analysis in sea water samples.

The authors passed 100L of acidified seawater samples (in case of KNiFC-PAN unacidified seawater samples were tested as well) through 25mL beds of AMP-PAN or KNiFC-PAN resin at flow rates up to 300mL/min allowing for processing 100L samples in less than 6h. Stable Cs was added to the seawater samples to allow for the determination of the chemical yield e.g. via ICP-MS.



Ammonium MolybdoPhosphate (AMP) and potassium Nickel FerroCyanate (KNiFC)

After loading resins were rinsed from the columns, dried and measured by gamma spectrometry in Petri dish geometry. Chemical yields obtained are generally high (>90%), KNiFC-PAN showing slightly higher yields for the acidified seawater samples than AMP-PAN resin and comparable chemical yields for acidified and non-acidified seawater samples. Higher flow rates were tested for the processing of non-acidified sea water samples on KNiFC-PAN resin; even at a flow rate of 470mL/min Cs yield is still greater than 85%.

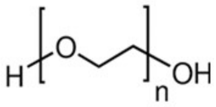
KNiFC-PAN resin was further used for the determination of Cs isotopes in milk and urine.

Main applications

- Concentration and separation of Cesium

TK202 Resin

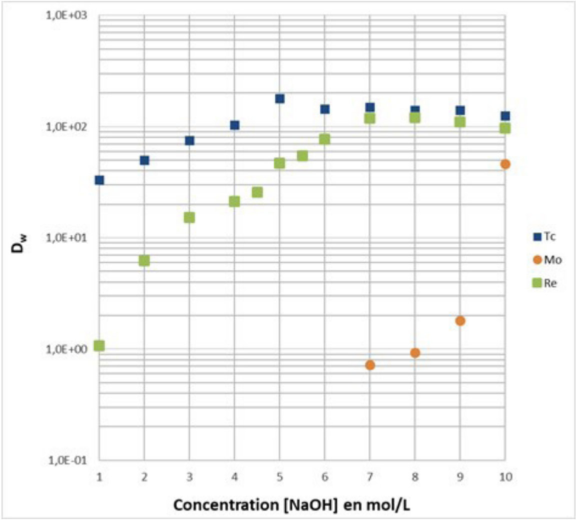
The TK202 Resin is based on Polyethyleneglycol (PEG) groups, with high molecular weight, that are covalently bound onto a polymer support.



PolyEthyleneGlycol (PEG).

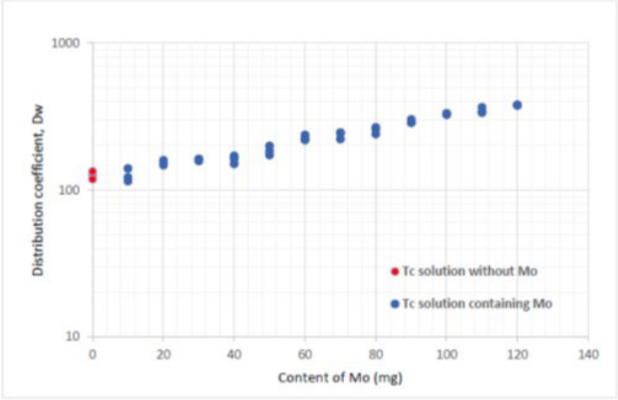
The TK202 Resin is based on an aqueous biphasic system (ABS) extraction mechanism with the covalently bound PEG acting as solid separation support. In presence of aqueous solutions with high ionic strength and high content of water-structuring (kosmotropic) anions like SO_4^{2-}, CO_3^{2-}, OH^-, as well as MoO_4^{2-} or WO_4^{2-}, it will extract chaotropic ions, according to Spear et al. notably TcO_4^- and ReO_4^- while other non-chaotropic elements will not be retained, molybdenum being an important example. Accordingly, Tc (and Re) retention will improve with increasing concentration of these anions.

The following graph shows D_w values for Tc, Re and Mo on TK202 Resin at increasing OH concentrations. Ideally the NaOH concentration should be between 5 and 7M NaOH during load and rinse, as Tc (and Re) retention is highest while Mo retention is very low.



D_W values for Tc, Re and Mo on TK202 Resin, at varying NaOH concentrations. Tc data taken from Cieszykowska et al.

As mentioned before, MoO_4^{2-} itself is a kosmotropic anion. Accordingly, increasing its concentration will lead to higher Tc (and Re) retention in ABS systems like the TK202 Resin, as shown below. A distinct increase of the Tc retention with increasing amounts of Mo is observed. This is particularly relevant in case of the separation of Tc from elevated amounts of Mo (e.g. irradiated Mo targets).



D_W values for Tc in 5M NaOH using 40mg TK202 Resin, increasing amounts of Mo. Data taken from Cieszykowska et al.

Cieszykowska et al. estimated from column studies that 6 to 8g of Mo per g of TK202 Resin allow obtaining high Tc recovery (> 90%). Further increasing the amount to 12g Mo/g of resin lead to a decrease of the Tc recovery to ~82% in their experiments.

The retained Tc and Re can then be eluted with water as the ABS systems breaks down, due to the low concentration of kosmotropic anions, under these conditions.

As mentioned, one potential application of the TK202 Resin is the separation of Tc-99m from irradiated Mo targets. Accordingly, its high selectivity for Tc over Mo, and the fact that the presence of elevated amounts of Mo in solution increases the Tc retention, makes the TK202 Resin particularly suitable for this type of applications.

Indeed, besides the production of Mo-99 for the fabrication of Mo-99/Tc-99m generators via U-235 fission there are a number of other ways to produce Mo-99, and thus Tc-99m. Three of these methods are based on the irradiation of Mo targets:

- Neutron activation of Mo-98 via (n, γ) reactions (Mo-98 (n, γ) Mo-99), preferably performed in a reactor with high neutron flux.
- Photon-induced (γ, n) reaction of Mo-100 (Mo-100 (γ, n) Mo-99) using photons (γ) obtained through irradiation of heavy targets (converter) e.g. with electron beams.
- Direct Tc-99m production on a cyclotron using Mo-98 targets (Mo-98 (p, 2n) Tc-99m). This latter method represents, with respect to the short half-life of Tc-99m, certain logistical challenges and will generally rather allow for supplying users close to the production facility. This method requires a clean, and very rapid, separation of the produced Tc-99m from the target material.

The first two described methods tend to result in Mo-99 of limited specific activity, especially compared to Mo-99 obtained from U fission. Accordingly using them e.g. in alumina column based generator systems will, due to the generally limited Mo capacity of these columns, result in rather lower activity Mo-99/Tc-99m generators.

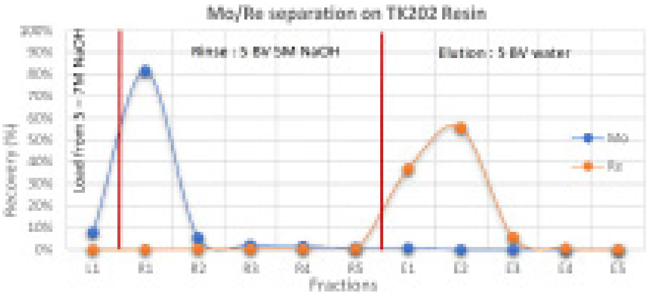
In such cases employing a resin, such as the TK202 Resin, to extract the Tc-99m originating from Mo-99 decay while letting Mo pass through ("inverted generator") is often a preferred option.

It should be noted that in all three cases the recovery and recycling of the enriched Mo is of very high importance due to the pricing and limited availability of the target material.

The production methods described above all require the use of a resin with high selectivity for Tc over large amounts of Mo. Ideally, as the Mo targets are very frequently dissolved in NaOH solutions of elevated concentration, the resin should show this selectivity under these conditions.
This is the case for the TK202 Resin. As indicated before, Tc may then be recovered using water, although further separation steps will be necessary to adjust pH and Na^+ concentration of the final product.

Initial elution studies using Re instead of Tc confirmed the high selectivity for Re (and, as could be confirmed in separate tests, also Tc) over Mo.

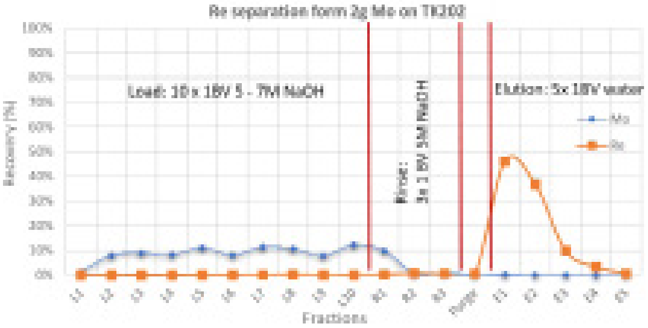
The graph below shows an elution study performed with trace amounts of Mo and Re. As it can be seen, a clean separation of both elements is obtained. Mo is removed during load and following rinses (both may be performed with 5 – 7M NaOH), while Re elutes in a small water volume.



Elution study, trace amounts of Mo and Re on a 2mL TK202 Resin cartridge, load and rinse at 1BV/min, elution at 0.25BV/min.

As the general selectivity could be confirmed further tests were performed using larger amounts of Mo.

The ellution study below shows the separation of traces of Re from 2g of Mo, as e.g. typically required in case of Tc-99m production from Mo-98 irradiation in a cyclotron, as described e.g. by Bénard et al.



Elution study, separation of trace Re and 2g Mo on a 2mL TK202 Resin cartridge, load and rinse at 1BV/min, elution at 0.25BV/min.

Elution study, separation of trace Re and 2g Mo on a 2mL TK202 Resin cartridge, load and rinse at 1BV/min, elution at 0.25BV/min.

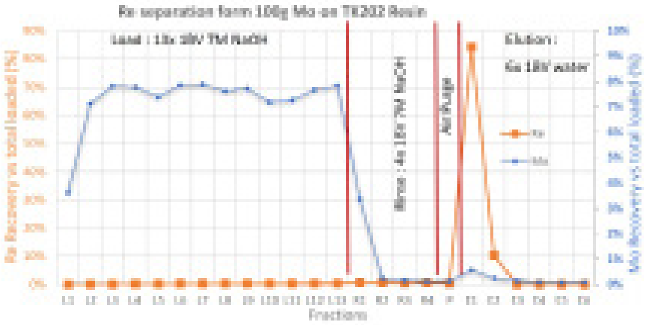
It should be noted that purging the resin, e.g. with air, after the rinse and before its elution with water, to remove NaOH from the cartridge/column, is of high importance to reduce the Na^+ and OH^- load of the final Tc/Re fractions.
It could further be shown that lower flow rates during elution of the TK202 Resin will result in narrower elution peaks, and thus lower elution volumes.

As discussed previously, the TK202 Resin may also be used to extract Tc-99m from Mo-99 decay present in an alkaline solution. In such cases much larger Mo targets are generally irradiated.
With respect to this, the separation of trace of Re from 100g Mo was tested. As shown below a clean separation of Mo and Re could be obtained here, too. Nevertheless, with respect to the very large amount of Mo present, an additional purification of the obtained Tc will be required.

Elution study, separation of trace amounts of Re and 100g Mo on a 75mL TK202 Resin cartridge, load at 0.5BV/min, rinse at 1BV/min, elution at 0.2BV/min.

Methods for the separation of Tc from larger Mo amounts (e.g. ≥200g) are currently being tested.

As shown e.g. by Bénard et al., one convenient option for this additional Tc purification is the use of a cation

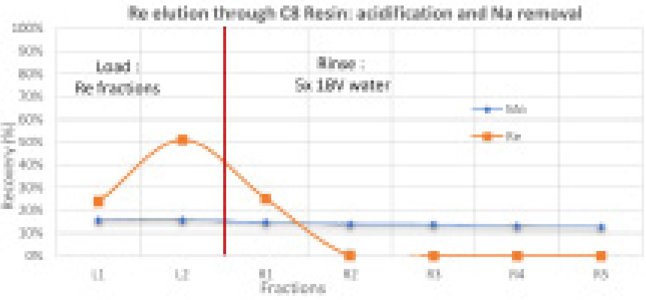


Elution study, separation of trace amounts of Re and 100g Mo on a 75mL TK202 Resin cartridge, load at 0.5BV/min, rinse at 1BV/min, elution at 0.2BV/min.

exchange resin (for Na^+ removal and pH adjustment to below pH 7), followed by an alumina cartridge (for Re/Tc concentration and further Mo removal) such AlOxA. Especially for the cation exchange cartridge it is important to adjust the size of the cartridge to the amount of Mo previously present in the sample/and so the size of the TK202 Resin cartridge employed.

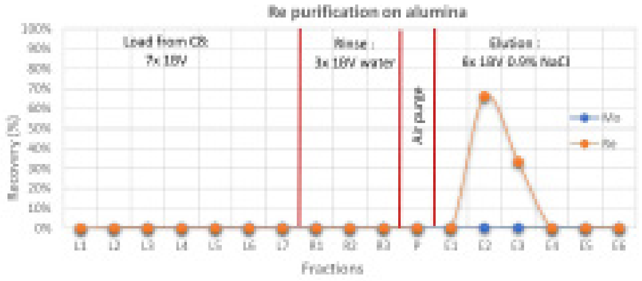
For the size of the alumina cartridge on the other hand the amount of residual Mo will be a decisive parameter.

The graph below shows the continuation of the 2g Mo separation test shown before.
The obtained Re fractions (E1 – E4) were combined and loaded through a C8 cation exchange resin cartridge which was then rinsed with water. The load fractions and the first rinse were collected, analyzed, and then combined for the final step of the separation, as they contain all the Re (or Tc).



Continuation of the separation of Re from 2g Mo (see above). Na^+ removal and pH adjustment of Re fractions on 2mL C8 Resin cartridge, load and rinse at 2BV/min.

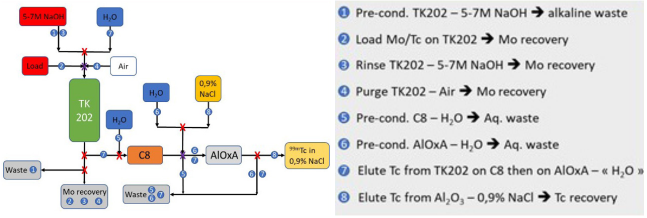
Continuation of the separation of Re from 2g Mo (see above). Na^+ removal and pH adjustment of Re fractions on 2mL C8 Resin cartridge, load and rinse at 2BV/min.
As stated before, at this stage the Re fraction should be below pH 7 (typically 3 – 5) and largely free of Na^+ cations.
It could be shown that under these conditions, acidic alumina will retain Re/Tc (and Mo). A 0.9% NaCl solution then allows eluting Re/Tc in a small volume (2 – 3 BV) while Mo remains very strongly retained, thus further improving the purity of the recovered Re/Tc.
The indicated air purge is not necessary in case of the AlOxA Resin (acidic alumina).
This further has the advantage of allowing to obtain the Tc in the same matrix (0.9% NaCl) as delivered by a Mo-99/Tc-99m generator.



Re concentration, purification and conversion to 0.9% NaCl solution on 1mL AlOxA Resin at 2BV/min.

Overall, in the cold tests Re recoveries in the order of >90% could be obtained.

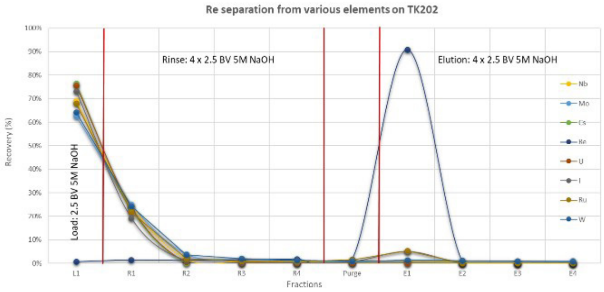
A schematic overview of the suggested separation method is given below. The method may be applied for the separation of Tc from Mo targets of various sizes, cartridge/column volumes will need to be adjusted accordingly.



Schematic overview, Tc separation from Mo.

Further to the TK202 Resin TrisKem also supplies C8 Resin and AlOxA Resin.
All resins are available in various columns and/or cartridges of different sizes (depending on the size of the Mo target).

Besides the described radiopharmaceutical use the TK202 Resin may also be employed in radioanalytical applications, notably in the analysis of Tc-99 in samples that were solubilized via alkaline fusion.
In such cases the solubilized samples (e.g. concrete samples resulting from decommissioning work) may, after removal of insoluble material, be adjusted to 5 – 7M NaOH and then passed through TK202 Resin to separate Tc.



Re separation from selected elements on 2mL TK202 Resin cartridge, load and rinse at 1BV/min, elution at 0.25 BV/min.

In order to further increase the purity of the obtained Tc fraction it might be passed, as described above, through a C8 Resin, and potentially even AlOxA Resin.

Main applications

- Separation of technetium
- Separation of rhenium

ANALYTICAL GRADE ION EXCHANGE RESINS

Anion and Cation Exchange Resins for Analytical Applications

TrisKem supplies an expanding range of cation and anion exchange resins, purified for the needs of analytical applications. The analytical grade ion exchange resins are purified with successive rinsing of methanol, NaOH and HCl to remove residual organic molecules remaining from the manufacturing processes in order to provide you with a high quality product. The line of cation exchange resins for analytical applications are type 50W strong acid cation exchange resins (sulfonic acid exchange groups on styrene divinyl benzene polymeric beads).
The resins are available in 4% and 8% DVB cross linkages. All cation exchange resins are provided in
hydrogen form and are supplied in the following dry mesh ranges: 50 - 100, 100 – 200, and 200 – 400.

The line of analytical anion exchange resins are type 1 strong base anion exchangers (quarternary amine functional groups on styrene divinyl benzene polymeric beads.) DVB cross linkages of 4% and 8% are currently available in dry mesh sizes of 50 – 100, 100 – 200, and 200 - 400. All resins are available in the chloride form.

Main applications

- Preconcentration of cations (e.g. Sr, Pb)
- Separation of Pu

Tritium Columns

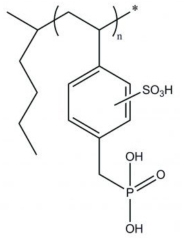
Tritium columns (H-3 columns) are used for the separation and determination of free tritium as tritiated water and are an alternative to direct measurement or measurement after distillation. Since the columns are not concentrating tritium, they can only be used when the required detection limit can be obtained by measurement of a sample volume of 5 – 10mL. The free tritium is passing through the column while other elements of the matrix are retained on the three components of the Tritium column.
The Diphonix resin retains cations in exchange of protons; its theoretical capacity is 0.8 mEq per column.
The 1X8 anion exchange resin (Cl- form) retains anions that might interfere with the Tritium measurements. Its theoretical capacity is 0.8mEq per column. It is recommended to work at sample pH values greater than 1. The Prefilter Resin is used to bind traces of organic impurities. Its theoretical capacity is 50mg per column.

Monophos Resin

Monophos Resin is based on a Polystyren-DVB support functionalized with monophosphonic acid.

The Monophos Resin mainly finds use in process scale applications. Within these applications it is most frequently used in order to control of the Fe(III) concentration in Cu, Ni and Co electrowinning electrolytes.

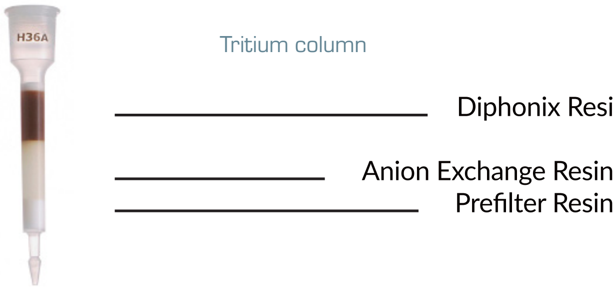
It further allows retaining actinides in the oxidation states III, IV and VI. At the same time other cations which are frequently found in environmental samples (e.g. Ca and Fe(II)) are not well retained, accordingly it may be used to preconcentrate actinides from leached soil samples.



Monophos® resin

Main applications

- Fe(III) removal from Cu electrowinning solutions
- Preconcentration and removal of actinides



Tritium column

Main applications

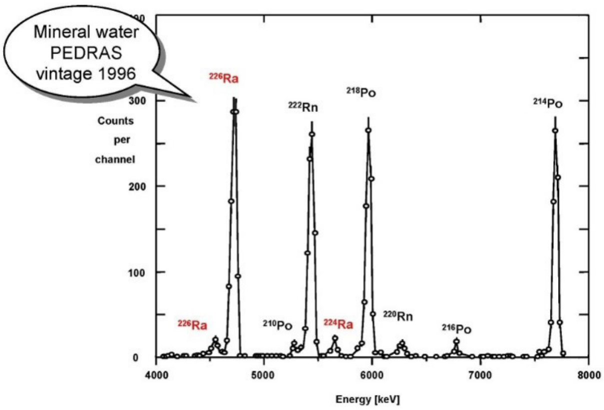
- Determination of H-3 in aqueous samples

Ready to use method H3W02 Tritium in water available at:
www.triskem.com

SAMPLE PREPARATION

Nucfilm Discs

Recent improvements in the fabrication of MnO_2 layers by Dr. Heinz Surbeck (Nucfilm GmbH) now allow the fabrication of selectively adsorbing MnO_2 coated as a thin film onto the surface of a polyamide disc. These coated substrates are available as Ra NucfilmDiscs. Due to their high selectivity for Ra the discs allow the direct determination of Ra isotopes in water samples without applying additional radiochemical separation methods.



Alpha spectrum of a radium adsorbing thin film exposed to a Portuguese mineral water.

The discs are contacted with the untreated water samples (pH 4 – 8, typical volume = 100mL) under stirring for 6h. Under these conditions the Ra extraction is typically greater 90%. The dried disc can then be measured with a solid state alpha detector.

The energy resolution of the obtained sources is very good as demonstrated in the following figure, typically FWHM in the order of 30 to 40keV are obtained. The analysis of a 100mL sample (counting time t = 80000 s, 900mm^2 Si-detector at 10 mm distance) typically results in a detection limit (LLD) of 5mBq/L for Ra-226.

Main applications

- Determination of Ra-226 in water samples

Resolve™ Filters

Filters typically are manufactured to meet specifications for removal of particles to result in defined solution purity characteristics.

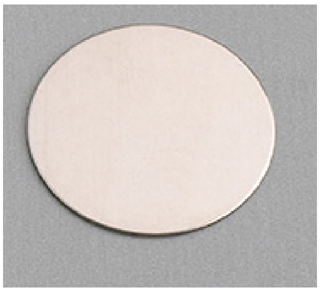
For example, a 0.1µm rated pore size filter is typically defined as capable of removing 99.98% of particles 0.1µm from a liquid. The pores present on the surface, however, may be much larger or smaller than 0.1µm. Maximum peak resolution in alpha spectroscopy requires a filter surface as uniform as possible to ensure a uniform deposition of the rare earth fluoride precipitate.

Resolve™ filters (0.1µm PE, 25 or 47mm diameter) are manufactured to specifications appropriate for alpha source preparation in order to allow obtaining alpha spectra with high resolution. The 25mm Resolve® filters are also available in filtration units for use with vacuum boxes

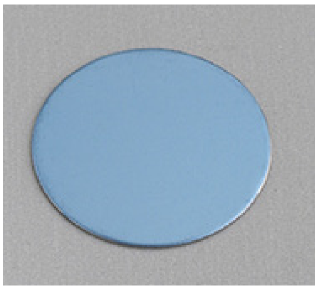
Discs for alpha spec source preparation

TrisKem is providing stainless steel discs for electrodeposition source preparation. Silver and nickel discs as well as an auto-deposition kit are available for the particular case of polonium auto-deposition.

Ra Nucfilm Disc

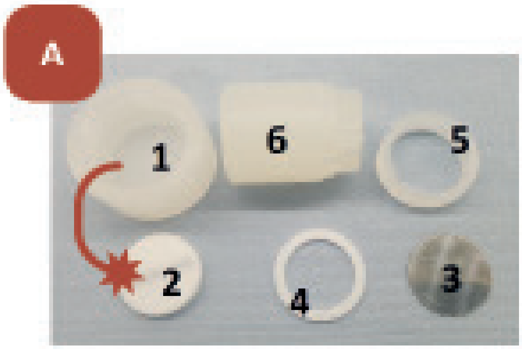


Nickel Disc

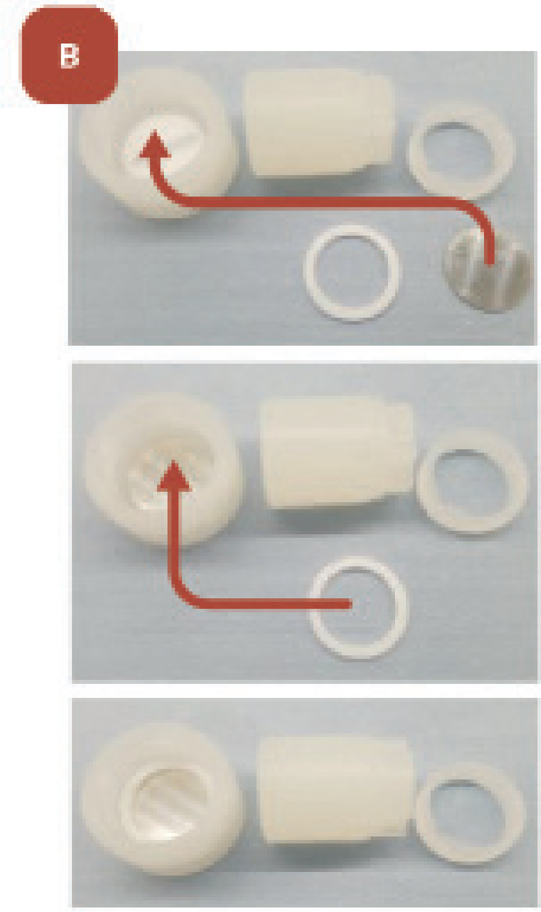


Silver Disc

Resolve filters



(1) Plastic rotative unit, (2) Flat circular magnet Ring, (3) Disk, (4) Ring, (5) Clamping ring, (6) Retaining clip



Insert (2) Flat circular magnet Ring into (1) Plastic rotative unit then the (3) Disk and the (4) Ring

Place the (6) Retaining clip in the (5) Clamping ring

The system is ready for deposition in a beaker on stirring plate

Tighten the (5) Clamping ring in the (1) Plastic rotative unit

ACCESSORIES

Vacuum box and accessories

Vacuum-assisted separation setups based on a vacuum box and cartridges are gaining increasing popularity. TrisKem provides 4, 12 and 24 position vacuum boxes, empty 1, 2, 8 and 20mL cartridges, associated accessories such as reservoirs, valves allowing for individual flow rate adjustment and spare parts for the vacuum box.

Column accessories

In order to allow our users to pack columns themselves TrisKem provides empty 2, 5 and 20mL columns. We further provide funnels (20mL for 2mL columns and 250 mL for 5 -20mL columns) and racks (for 2mL columns and for 5 - 20mL columns) for empty and prepacked columns.

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Products	Applications*
UTEVA® Resin	U, Zr, Th, Np, Pu
TRU Resin	Fe, Pu, Am/Cm, Th, Pa, U, Np
TEVA® Resin	Tc, Th, Np, Pu, Am/lanthanides
SR Resin	Sr, Pb, Po
PB Resin	Pb
DGA Resins	Am, Ac, Actinides, Y
DGA Sheets	Quality control of Ra-223, Pb-212, Ac-225/Bi-213, Ge-68/Ga-68
LN Resin Series	Lanthanides, Ra-228
NI Resin	Ni, Pd
RE Resin	Rare earth, Th, U, Np, Pu, Am, Cm
CL Resin	Cl, I, Ag
CU Resin	Cu
CU iSheets	Quality control of Cu labelled compounds
ZR Resin	Zr, Ti, Ge, Ga, Nb, Mo
TBP Resin	Sn, Zr, Actinides, Sc
TK100 Resin	Sr, Ra, Pb (direct separation)
TK101 Resin	Pb (direct separation), Ra
TK102 Resin	Ra/Ba separation, Pb, Sr
TK200 Resin	Ga-67/8, Actinides
TK201 Resin	Tc, Cu, Pu
TK202 Resin	Tc, Re from alcaline samples
TK221 Resin	Actinides, Lanthanides separation and purification (e.g. Lu-177), Ac-225
TK222 Resin	Ac, Lu
TK225 Resin	Lanthanides removal from acidic effluents
TK227 Resin	Sr-90 determination in water samples via Y-90
TK211/2/3 Resins	Lanthanide separation (e.g. nca Lu-177, nca Tb-161)
TK400 Resin	Pa, Ga, Fe, Nb, Mo
TK-TcScint	Tc-99 by direct LSC measurement
TK-SrScint	Sr-89/90 by direct LSC measurement
Guard Resin	Removal of organic impurities
AC Resin	Actinides separation/ gross alpha measurement, Be, Mn
TK-GA Discs	Screening for actinides in water samples by alpha spectrometry
Pre-Filter Resin	Removal of organic impurities
MnO_2 PAN Resin	Ra in water samples
CS Resins	Cs-134/7
Analytical grade Ion Exchange Resins	Preconcentration
Monophos Resin	Actinides and transition metals
Tritium Columns	H-3
Nucfilm discs	Ra in water samples
Resolve™ Filters	Microprecipitation
Stainless steel discs	Electrodeposition
Ag and Ni discs / Autodeposi	Po autodeposition

*Main applications are shown in grey

Upcoming new products :

TK250 Resin, TK300 Resin, SE Resin,...

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