EXTRACTION CHROMATOGRAPHY
Technical Documentation - New Developments

Extraction chromatographic resins

Specialty ion exchange resins

Technical Sheets

Applications
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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)
• 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 cocktails, filters and discs for alpha spectrometry, and Pyrolyser systems for the determination of H-3, C-14, Cl-36, I-129,... in a variety of matrices including decommissioning samples.

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.
TrisKem International is an ISO9001 certified French independent company producing and developing extraction chromatographic resins.

Extraction Chromatographic Resins combine the selectivity of liquid-liquid extraction and the rapidness and the ease of use of chromatography.

Due to their high selectivity, methods using TrisKem’s Extraction Chromatographic Resins, are shorter, demand less hands-on time and reduce, or even avoid, the use of toxic and dangerous reagents.

The resins are comprised of microspheric beads with a very high surface impregnated with an organic extractant. The large surface of the beads and the high content of extractant allow reducing the size of the columns, and therefore also the amount of reagents used – resulting in the generation of less waste.

Extraction Chromatographic Resins can be prepared using a wide choice of different extractants, and thus selectivities:
- Liquid anion exchangers
- Liquid cation exchangers
- Solvating and chelating extractants
- Macrocycles (e.g. crown ethers)
- Ionic liquids

Extraction Chromatographic columns, cartridges and discs are facile to handle and can easily be used in vacuum based systems or in automated separation setups e.g. for the separation of high active samples in glove boxes or hot cells, as for example needed for the preparation of radionuclides for nuclear medicine.
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.

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.

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, ...

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

A number of ready-to-use methods are available through our new website: www.triskem.com
The CL Resin is based on an extraction system that is selective for platinum group metals, gold and silver and is mainly used for the separation of chloride and iodide, especially in the context of Cl-36 and I-129 analysis, and the removal of radioiodine from effluents.

The selectivity for halides is introduced by loading the resin with Ag⁺ allowing good selectivity for anions, especially halides, forming sparsely 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 ensure their presence as halides.

Radioiodine is considered to be one of the most dangerous radioelements in terms of radiological effects in case of accidental release. Accordingly efforts are made to capture iodine from liquid effluents already during the production process before waste storage, to avoid a possible gas release from stored liquid waste at a later date.

Caroline Decamp of the Institute of Radioelements (IRE) has developed a method for the removal of radioiodine from acidic (1M HNO₃) radioactive process waste solutions. The IRE combined the CL Resin with XAD-4 resin and packed this mix (4g XAD-4 and 3g CL Resin) into columns. These mixed bed columns were then introduced into their process setup in order to remove radioiodine from their multi-curie production process solutions.

One prerequisite of the removal step was that it should not slow down the process; the radioiodine removal was thus tested at the same flowrates applied during the process. Flow rates up to > 180 mL/min and effluent volumes between 12L and 17L were tested, and it was found that under these conditions between 85 and more than 95% (overall mean retention yield for all solution volumes is 88% ± 5% (N = 14, k = 1) of the radioiodine present were retained on the column, thus lowering the I-131 activity of the effluents by a factor of 10 from 100 GBq/L to about 10 GBq/L. 2000 GBq of I-131 could be removed per decontamination cycle and stored as solid waste.
Besides the removal of radioiodine from effluents the CL Resin is also used for analytical purposes. It allows for the preconcentration of isotopes e.g. from waste water upfront to gamma spectrometry and, more importantly, for the determination of I-129 and Cl-36, both long-lived, volatile radionuclides, in environmental and decommissioning samples.

In case of analytical applications, 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 S²⁻ solution.

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

Warwick et al. have developed a method for the analysis of decommissioning samples [e.g. spent resin] based on the thermal decomposition of the sample to be analysed using a Raddec ‘Pyrolyser’ furnace. Volatilized chlorine species are transported by a stream of moistened air into a bubbler containing a 6 mM Na₂CO₃ solution where they are trapped. The authors modified the separation procedure so that the bubbler solution could 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 5 mL 0.1M H₂SO₄ 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 H₂O₂, removal of the sulphate by precipitation with Ba followed by centrifugation, and finally an 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.
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 here.

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 of elevated amounts of Zn and Ni, even at 1 g of Zn or Ni 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 – 1.5 mL) in less than 10 min using a vacuum assisted separation setup.

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

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

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 20 cm, 10 x 10 cm or 20 x 20 cm. Other formats and custom dimensions are however also available upon request.

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

DGA application notes available on our website: www.triskem.com/tki-methods.php
The ZR Resin is based on the hydroxamate functionality frequently used for the separation of Zr, especially from Y target materials for later use in radiopharmaceutical applications. Dirks et al. have characterized the resin with respect to its selectivity for selected elements in HNO₃, HCl and oxalic acid.

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 weaker from 1 – 6M HCl. As expected the resin shows very little selectivity for Sc and Y, a separation of e.g. Zr from Y and Ti from Sc is thus possible.

The resin shows rather similar selectivity in HNO₃, Zr, Ti and Nb are well retained up to 5M HNO₃, Fe(III) is well retained up to 1M HNO₃. At higher HNO₃ concentrations the nitric acid starts attacking the extractant, 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 HNO₃.

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 high D_w values even at 0.05M oxalic acid, Zr and Nb may thus be separated by adjusting the oxalic acid concentration accordingly.

Zr can be recovered near quantitatively in 1.5 mL dilute oxalic acid even in presence of up to 300 mg stable Y (using a 100 mg ZR Resin column).

The loading is preferably performed under reducing conditions in order remove Fe from the column before Zr elution.

Other than for Zr the ZR Resin also shows very interesting selectivity for Ti, especially with respect to Sc. While Sc is not retained e.g. from 10M HCl Ti is very well fixed. 0.1M citric acid may then be used to elute Ti from the resin; beside citric acid, hydrogen peroxide, oxalic acid or HNO₃ of high concentration may be employed.

As Ti is retained over a very wide range of HCl concentrations, including dilute HCl, its potential use as support for a Ti/Sc generator is currently evaluated.

Other than for Zr, Nb and Ti the resin also shows very interesting selectivity for Ge over Ga, allowing for its separation from macro quantities of Ga.

Main applications:
- Separation of zirconium
- Separation of titanium
- Separation of germanium
- Separation of gallium
TK200 Resin

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

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

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 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.

It should be noted though that only very little additional Ga/Zn separation is taking place on the TK200 Resin.
This allows for using the TK200 resin for the preconcentration of actinides from acidified water samples, and their subsequent separation on the same column.

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 following figure, U and Th are very well retained over the whole HNO₃ concentration range, including 0.01M.

Dₘ values of selected elements on TK200 Resin in HNO₃

Elution of Pu from 1g TK200 resin cartridge

(data courtesy of Nora Vajda)

U and Th are also very well retained from HCl.

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

Dₘ values of selected elements on TK200 Resin in HCl

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.

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 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.

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 Russel 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 Russel 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 Russel et al. (NPL)
The TK201 Resin generally shows rather limited selectivity in HNO₃, similar to Tc(VII) Re is well retained at low HNO₃ concentrations (0.01 – 0.1M HNO₃). At elevated HNO₃ concentrations Pu is well retained and Th fairly well, other actinides are not retained under these conditions.

Out of the other elements tested only Bi (at about 0.5M HNO₃) and Mo (at low HNO₃ concentrations) are retained. It is important to note that Mo is not retained at HNO₃ concentrations above 0.5M while Tc and Re are well retained (shown on the 1st figure), allowing for their clean separation.

It could further be shown by Vajda et al. that Dₜ values for Tc(VII) are very low in dilute NH₄OH: in 0.1M NH₄OH Tc(VII) shows a Dₜ of only ~2, accordingly it is easily eluted by ≥ 0.1M NH₄OH.

Additional elution studies indicated that an efficient Mo separation from Re is possible using 0.7M HNO₃ for Mo removal and dilute NH₄OH for Re elution.

Mo/Tc separation. Most suitable conditions for Tc elution were found to be NH₄OH greater or equal to 0.2M.

Another application of the TK201 Resin is the separation of Cu isotopes, in combination with the CU Resin, from irradiated solid Ni targets. While CU Resin shows very high selectivity for Cu over Ni, Zn, Ga... it requires loading at pH ≥ 2 which is not easily compatible with solid Ni target dissolution and separation chemistry, as these are generally dissolved in strong HCl.

TK201 Resin may be used to retain Cu from 6M HCl, while letting Ni pass for subsequent recycling. The Cu may then be eluted under suitable conditions [e.g. using a mixture of NaOH and acetate buffer at pH 3] allowing for direct loading onto CU Resin for further purification. It might also be used to convert the Cu fraction eluted from the CU Resin from highly acidic solution [e.g. 6 - 8M HCl] to conditions more suitable for labeling [e.g. dilute HCl] as indicated in the following figure.
The TBP Resin is comprised of an inert support impregnated with Tributylphosphate (TBP). TBP is a widely used extractant, it finds 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.

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 have shown 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.
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 0.1 M 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 a long-lived beta emitter Sn-126, 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.

The TBP Resin has further been characterized with respect to the uptake of various elements in HNO₃ and HCl.

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 1 M 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 0.1 M HCl.
In HNO₃ of all tested elements only the actinides (at elevated HNO₃ concentrations), and Ag (at low HNO₃ concentrations), are retained on the resin. Based on the obtained data, Dirks et al. have developed a procedure for the separation of Sn from various elements.

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 6 mL 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).

Main Applications:
- Separation of tin
- Separation of zirconium
- Separation of scandium
- Separation of actinides

Separation scheme Sn separation on TBP Resin.
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 the TK400 Resin with respect to its selectivity for a number of elements including Pa, Np, U and Th.

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.

![Distribution coefficients of Pa, Np, U, Th, Th(IV), Ac(III) and Ra(II) was investigated and a highly Ac resin is chromatography resin expressed as Vs chromatography resin.](image1)

Elution study, Np/Pa separation on long-chained alcohol resin

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

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.

![Distribution coefficients of Pa, Np, U, Th, Th(IV), Ac(III) and Ra(II) was investigated and a highly Ac resin is chromatography resin expressed as Vs chromatography resin.](image2)

$D_w$ values of selected elements on TK400 Resin in HCl at varying concentration
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 (~83%).
TK100 Resin & TK101Resin

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.

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 ($D_W >> 100$).

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.

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 500 mM, K concentrations of 400 mg/L, Mg concentrations of 1300 mg/L and Ca concentrations of 500 mg/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 1 L samples at pH 7 loaded onto a 2 mL TK100 column in aliquots of 100 mL at a flow rate of 5 mL/min showed that K and Ca breakthrough during load without being retained.

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

Dirks et al. could indeed show that Sr can be separated with high yields from 250 mL (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 – 10 mL/min. Y, Pb and U on the other hand are very well retained, even when a sample of 1 L is loaded. Y can be quantitatively removed using 8M HNO₃. 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.

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.

Main Applications:
- Direct separation of Sr from water samples
- Direct separation of Ra from water samples
- Direct separation of Pb from water samples

1 L loading - 100 mL fractions - TK100 Resin - 5 mL/min

Elution study, various elements, 1L sample, 100 mL aliquots, TK100 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, leading to high Sr retention at low pH values. Sr retention then decreases with increasing acid concentrations to a minimum at about 1M HNO₃. At higher HNO₃ concentrations the Dₗ values increase as expected when liquid-liquid extraction mechanism becomes predominant.

This behavior corresponds very well to the behavior of the crown-ether / HDEHP system. However, as very little additional selectivity is introduced by the ionic liquid, compared with TK100 Resin, it is much easier to obtain a clean Pb fraction. Unfortunately the Sr retention turned out to be significantly weaker than for the TK100 Resin, limiting the application of this system to the separation of Pb.

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 1 L samples or more at flow rates of 5 – 10 mL/min.
Cs Resins

Both resins, AMP-PAN and KNiF-PAN have been developed by Dr. Šebesta from the Czech Technical University in Prague. Like the MnO₂-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-PANs 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 resin for use in Cs-134/7 analysis in sea water samples.

The authors passed 100L of acidified sea water samples (in case of KNiFC-PAN unacidified sea water samples were tested as well) through 25 mL beds of AMP-PAN or KNiFC-PAN resin at flow rates up to 300 mL.min⁻¹ allowing for processing 100 L samples in less than 6h. Stable Cs was added to the sea water samples to allow for the determination of the chemical yield e.g. via ICP-MS.

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 sea water 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 470 mL.min⁻¹ 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
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 1.5 L water samples, with 1.25 g/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 Ac228 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) developed by Dr. Šebesta.

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
SAMPLE PREPARATION

Nucfilm Discs

Recent improvements in the fabrication of MnO₂ layers by Dr. Heinz Surbeck (Nucfilm GmbH) now allow the fabrication of selectively adsorbing MnO₂ 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.

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

The energy resolution of the obtained sources is very good, typically FWHM in the order of 30 to 40 keV are obtained. The analysis of a 100 mL sample (counting time t = 80000 s, 900 mm² Si-detector at 10 mm distance) typically results in a detection limit (LLD) of 5 mBq.L⁻¹ for Ra-226.

Discs for alpha spectrometry 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.
Pyrolyser-6 Trio

Raddec Pyrolyser-Trio Furnace System

Efficient and rapid extraction of tritium and C-14 from any material

The Pyrolyser-6 Trio & Pyrolyser-2 Trio furnace systems have been scientifically designed and evaluated to provide a safe and efficient means of extracting H-3 and C-14 (and other volatile radionuclides) from almost any type of sample (foodstuffs, biota, soil, sediment, concrete and other building materials, metals and bioassay samples).

Pyrolyser Mini

Raddec Pyrolyser Mini Furnace System

A compact furnace for the efficient and rapid extraction of H-3 and C-14 from any material.

The Pyrolyser Mini system is a compact two stage combustion furnace designed to complement the existing Pyrolyser-Trio family of combustion furnaces. The Pyrolyser Mini has been designed to be compact enabling the system to be installed and operated in confined spaces.
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*the main applications are shown in orange

Also available:
- ICP-MS standards
- LSC cocktails