TRISKEMINFOS



TRISKEM SHARING INNOVATION

## Cesium Resins

TrisKem has introduced 2 new resins in its catalogue this year: AMP-PAN and KNiFC-PAN resins for the separation of cesium from acidic and neutral liquid media respectively. These resins have been developed by Dr. Šebesta at the Czech Technical University in Prague.

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 **A**mmonium **M**olybdo**P**hosphate, AMP) and potassium nickel hexacyanoferrate(II) (also potassium **Ni**ckel **F**erro**C**yanate, KNiFC).

AMP-PAN resin is based on ammonium phosphomolybdate, an inorganic ion exchanger known for its high selectivity for Cs even at elevated acid concentrations, quick kinetics and radiation stability [1].

One of the main restraints to the use of AMP is its unfavorable microcrystalline structure accordingly considerable work has been performed to improve its granulometry. Embedding the AMP in an organic matrix allows for controlling particle size, topography, porosity, hydrophilicity and cross-linking of the resin matrix as well as the amount of AMP embedded in the resin.

Šebesta and Štefula showed that embedding the AMP in a PAN matrix only has limited impact on its Cs uptake kinetics, which remain very rapid, and on the Cs capacity of the embedded AMP [1]. It could further be shown that the resin is chemically stable even under relatively harsh conditions such as 1M $HNO_3$ / 1M $NaNO_3$ or 1M NaOH / 1M $NaNO_3$, even after storing the resin under these conditions for 1 month no visible mechanical damage could be observed, $K_D$ values, sorption kinetics and capacity also remained unchanged [2]. Radiolysis stability of the resin was evaluated in acidic solution by exposing it to doses up to $10^6$ Gy, again no changes in $K_D$ or sorption capacity were found.

**(NEXT) PAGE 2**

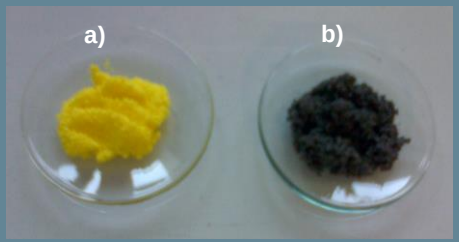



***Figure 1 : a) bulk AMP-PAN Resin, b) bulk KNiFC-PAN Resin***

## DEAR CUSTOMERS

**As every year we use summer time to revise our production equipment with the aim to maintain the high quality of our products and to enhance ergonomics of the working places.**

**This year I like to draw your attention especially to our column production. To assure the homogeneous filling of our columns and to avoid musculoskeletal disorders for our manufacturing team, we had decided from the beginning to privilege the automation of our column production.**

**You might notice small air bubbles under the upper frit of our columns. These bubbles are sometimes due to our manufacturing process but more often they occur during transport. Please note that these air bubbles have no impact on the performance of your analysis.**

**In case you wish to remove these bubbles please lift the upper frit, remove the bubble, insert the frit or replace it by lab cotton or quartz wool. If you prefer to push the frit onto the resin bed, please do this carefully to avoid compacting of the beads. This could otherwise reduce column flow rate.**

**For your convenience you find this information in each column box.**

**Please do not hesitate to contact us and to advice in case you need any further information. Our customer service is at your entire disposal at *contact@triskem.fr*.**

**Michaela Langer**
***TRISKEM CEO***

N°10
July
2013

TRISKEM INFOS
Publication Manager : Michaela Langer • Writting : Aude Bombard
Graphic Designer : Essentiel – Rennes • ISSN 2116-6145

## Tips and Tricks

- ***Empty cartridges***

We now propose empty cartridges of 2 and 12mL

***Figure 3: Empty cartridges of 2mL and 12mL.***

- ***CL Resin***

It is now indicated on the CL Resin packagings that the resin comes NOT loaded with Ag+. CL Resin is to be activated with Ag+ solution prior to use. The CL Resin method (TKI_CL01_V14) has been updated accordingly and is available online.

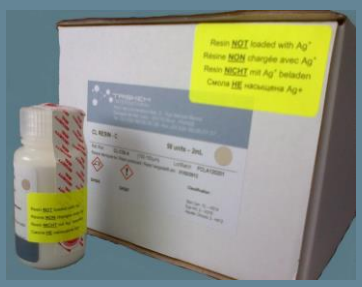
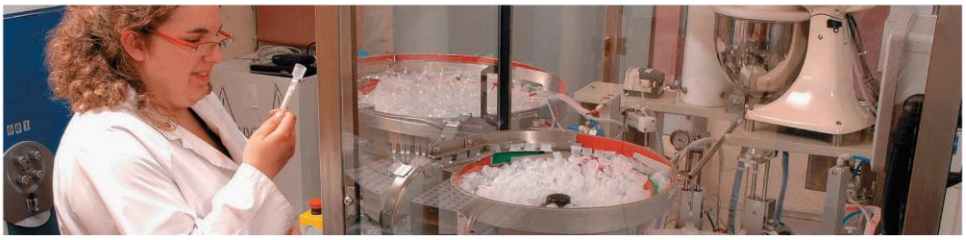


***Figure 4: Additional labelling on CL Resin products.***



Desorption of the cesium is only possible using concentrated ammonium salts, 10 bed volumes of 5M $NH_4Cl$ for example elute 92% of Cs from a column [1] (alternatively $NH_4NO_3$ might be used [3]) or by destruction of the AMP using strong alkaline solutions (like 5M NaOH).

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. [3] 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. Small scale tests were performed using 1.5 mL columns and two feed solutions, one simulated tank waste (spiked with 100 $Bq.mL^{-1}$ Cs-137) and one actual tank waste. Both solutions were filtered, and pumped through the column using a pump system at a flow rate of 26 – 27 bed volumes per hour, aliquots were taken at regular intervals and analyzed for Cs-137 activity. After the experiment the AMP-PAN columns were eluted using 30 bed volumes of 5M $NH_4NO_3$, reconditioned and the effluents were passed over the column a second time. For the real waste samples a Cs breakthrough of 0.15% was observed after a sample loading volume of 1000 bed volumes during the first loading cycle (corresponding to a Cs decontamination factor greater than 3000) and 0.53% after 830 bed volumes during the 2nd loading cycle. Cs recoveries in the respective eluates were 87%.

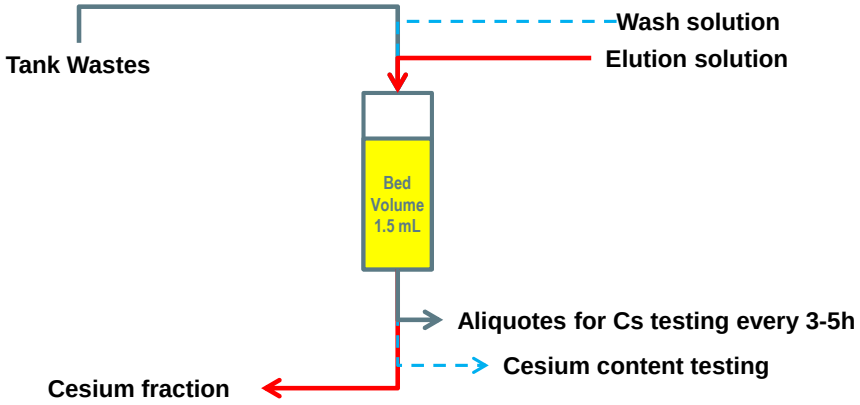



***Figure 2: Separation scheme for the separation and determination of cesium in waste effluent [3].***

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.

Pike et al. [4] used AMP-PAN for concentrating and purifying Cs from 20L seawater samples (acidified to pH 1 – 2, stable Cs was added for yield determination by ICP-MS). The authors employed 5 mL columns and worked at a flow rate of 35 $mL.min^{-1}$. After extraction the resin was rinsed from the column using 0.1M $HNO_3$ and analyzed by gamma spectrometry. Yields were found to be 93.5% +/- 5.0% (n=55). The authors further analyzed an internal lab standard (WHOI) in triplicate and IAEA sea water reference material, results are summarized in table 1.

| Sample reference | Reference value / $Bq.m^{-3}$ | Obtained value / $Bq.m^{-3}$ |
|---|---|---|
| WHOI | 3.4 +/- 0.4 | 3.7 +/- 0.2 |
| IAEA-443 | 340 - 370 | 369 +/- 8 |

***Table 1: Results in Cesium obtained using reference material on AMP-PAN resin.***

***For more information, do not hesitate to contact us and/or to download the technical data sheets from our website www.triskem-international.com***

Even larger seawater samples were analysed by Kamenik et al. [5]. The authors evaluated, in addition to the AMP-PAN resin, also the use of KNiFC-PAN resin, which is based on potassium-nickel hexacyanoferrate(II) embedded in a PAN matrix.

The authors passed 100L of acidified seawater samples (in case of KNiFC-PAN unacidified seawater 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 100L samples in less than 6h. As described before stable Cs was added to the seawater 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 using a coaxial HPGe detector with 43% rel. efficiency in Petri dish geometry. Chemical yields obtained are summarized in table 2. Yields are generally high, 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.

| Resin | Matrix | Chemical yield / % |
|---|---|---|
| AMP-PAN | sea water (pH 1) | 88,1 +/- 3,3 |
| KNiFC-PAN | sea water (pH 1) | 92,9 +/- 1,1 |
| KNiFC-PAN | sea water | 90,2 +/- 2,7 |

***Table 2: Comparison of obtained chemical yields, 100 L sea water samples, AMP-PAN and KNiFC-PAN [5]***

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%.

The authors calculated the minimum detectable activity (MDA) for 100L samples at 50 – 70 h counting time and average chemical yields. For Cs-137 they calculated an MDA of 0.15 Bq.m$^{-3}$ and 0.18 Bq.m$^{-3}$ for Cs-134.

KNiFC-PAN resin was further used for the determination of Cs isotopes in milk [6] and urine [7]. Scheme of the procedure is shown Fig. 5. The chemical yield in Cs was about 95% for both urine and milk. For milk, the MDA obtained was 2mBq.l-1 for 137Cs in 5 liters milk sample (HPGe detector, relative efficiency 140%, counting time 600000 s, $\rho$ = 1g.cm$^{-3}$).

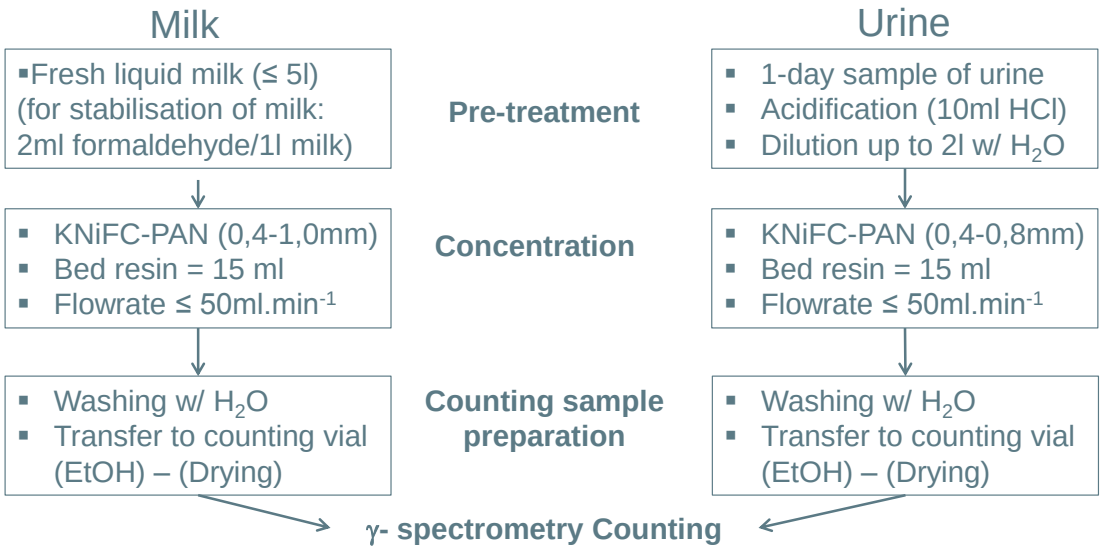



***Figure 5: Separation scheme of Cs in milk and urine samples.***

Other than for Cs separation AMP based ion exchangers have also been used to separate Rb from other alkalines in acidic media [8, 9].



*For more information contact or visit our website*
*http://www.triskem-international.com/*

## AGENDA

We'll be participating to the following upcoming conferences and are very much looking forward to meeting and discussing with you there!

° 8th LSC Anwendertreffen, 30/09 – 01/10/13, Tübingen (Germany) *www.uni-tuebingen.de/einrichtungen/zentrale-einrichtungen/isotopenlabor-strahlenschutz/veranstaltungen.html*

° NKS Workshop on Radioanalytical Chemistry, 02/09 – 06/09/13, Roskilde (Denmark) *http://www.nks.org/en/seminars/upcoming_seminars/nks-b_radioanalysis.htm*

° Asia-Pacific Symposium on Radiochemistry - APSORC 2013 22/09-27/09/13, Kanazawa (Japan) *www.radiochem.org/apsorc13/*

° Russian-Nordic Symposium on Radiochemistry, 21/10 – 24/10/13, Moscow (Russia), *http://rnsr.org/*

**You will find an update on our participations to conferences on our website www.triskem-international.com**

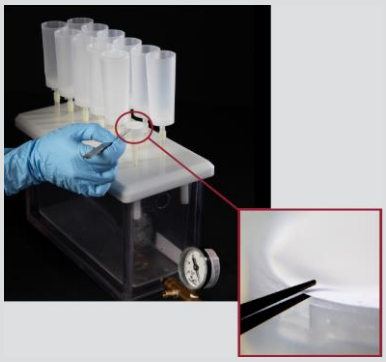


***Figure 6: filtration units using Resolve Filters (Ref RF-DF25-25PP01).***


**Bibliography**

(1) Sebesta F, Stefula V (1990) Composite ion exchanger with ammonium molybdophosphate and its properties. J Radioanal Nucl Chem 140(1):15 - 21

(2) John et al. (1999) Application of new inorganic-organic composite absorbers with polyacrilonitril binding matrix for separation of radionuclides from liquid radioactive wastes. Choppin and Khankhasayev (eds.) Chemical separation technologies and related methods of nuclear waste management, 155 - 168

(3) Brewer et al. (1999) AMP-PAN column tests for the removal of Cs-137 from actual and simulated INEEL high-activity wastes. Czechoslov J Phys 49(S1):959-964

(4) Pike et al.. (2012) Extraction of cesium in seawater off Japan using AMP-PAN resin and quantification via gamma spectroscopy and inductively coupled mass spectrometry. Radioanal Nucl Chem. DOI 10.1007/s10967-012-2014-5

(5) Kamenik et al. (2012) Fast concentration of dissolved forms of cesium radioisotopes from large seawater samples. J Radioanal Nucl Chem. DOI 10.1007/s10967-012-2007-4

(6) Kamenik J et al. (2009) Long term monitoring of Cs-137 in foodstuffs in the Czech Republic. Appl Radiat Isot 67(5):974-977

(7) Bartuskova et al. (2007) Ingestion doses for a group with higher intake of Cs-137. In: IRPA regional congress for Central and Eastern Europe, Brasov, Romania

(8) Coetze CJ : The separation of a sodium-rubidium mixture on an ion exchanger. (1972) J Chem Edu 49(1): 33

(9) Smit, J van R, Robb W, Jacobs JJ: Cation exchange on ammonium molybdophosphate—I: The alkali metals (1959) J Inorg Nucl Chem, 12(1-2): 104-112


## InBrief:

### New product

Starting this summer, we provide/offer a new accessory allowing the preparation of alpha spectrometry sources via micro-precipitation directly using the vacuum box. The filtration funnel units (Ref RF-DF25-25PP01) come preloaded with our Resolve® filters (see Fig. 6).
The funnel units are designed to maximize analyte recovery and the resolution of the filter. They are also designed with a notch, allowing for easy removal of filters with tweezers for planchet placement.

### Liquid scintillation cocktails NPEs free

NPE's and APE's are widely used as surfactant not only in liquid scintillation cocktails but also in detergent/textile industries/… European Directive 2003/53/EC bans the use of NPE's/APE's at concentration above 0.1% in these industries as the leaking of these molecules to the environment has engendered environmental issues due to high toxicity (endocrinian perturbators) and low biodegradability. Although their use in liquid scintillation cocktails is not restricted by the EU directive, the supply of these reagents might become an issue in the next decade. **Meridian Biotechnologies Ltd, manufacturer of the LS cocktail product line distributed by TrisKem,** has already moved to a new generation of NPE free LS cocktails with their ProSafe line of scintillation cocktails. For more information do not hesitate to visit our website or to contact us.

We remain at your disposal to study your specific needs.

***DO NOT HESITATE TO CONTACT US FOR MORE INFORMATION OR TO OBTAIN OUR NEW PRICE LIST***


**TRISKEM INTERNATIONAL**
Parc de Lormandière Bât. C • Rue Maryse Bastié • Campus de Ker Lann • 35170 Bruz • FRANCE
Tel +33 (0)2.99.05.00.09 • Fax +33 (0)2.99.05.07.27
www.triskem-international.com • email : contact@triskem.fr