TRISKEM INFOS

TRISKEM SHARING INNOVATION

DEAR CUSTOMERS

We wish you a good return from summer time. We hope to meet you in the different conferences that will take place starting in September: TrisKem Users' Group Meeting in Bath (UK), ERA12 in Bath (UK), ISTR 2014 in La Baule (FR), TrisKem Users' Group Meeting in Moscow (RU)…

This issue of the TrisKem Infos is dealing with examples of rapid methods, more specificaly with methods for the determination of actinides and strontium-90 in large seawater samples.

Our customer service is at your entire disposal at *contact@triskem.fr*.

Michaela Langer
TRISKEM CEO

Separation of Actinides and Strontium in large seawater samples

Requirements on detection limits in environmental monitoring are in general very strict. The Fukushima accident, on the other hand, has stressed the need for laboratories to provide results in a short time. In case of incident situations this can be easily achieved as applicable alarm/action levels are rather elevated (small sample masses and short counting times). Adjustments of these methods are required when applied to routine environmental samples in order to meet requested low detection limits. Then to remain with reasonable analytical processing times, work with larger sample volumes/masses helps keep counting time short.

This issue of the TrisKem Infos is dealing with examples of such methods, more precisely with methods for the determination of actinides and radio-strontium in large seawater samples. Recently important work has been done on sample pre-treatment methods drastically reducing time consumption and matrix content of column loading solutions. Sample pre-treatment of up to 80L can be achieved in 4-8h with overall chemical yields (incl. sample-pretreatment, separation and source preparation) in the range of 85-95% [1].

Figure 1 shows the preconcentration steps employed for the separation of Pu/Np and Am/Cm from 80L seawater samples. An iron hydroxide/Ti^{3+} precipitation is used as first step to co-precipitate and concentrate the actinides, careful control of the pH reduces the amount of Ca co-precipitating. The second precipitation with LaF_3 allows for iron and titanium removal. Pu and Np are then separated on TEVA Resin and Am/Cm on DGA Resin according to the steps shown in Figure 2.

If Th or Po are present, they will be retained on the TEVA Resin from 3M HNO_3. Th is easily eluted with 9M HCl, its removal can be improved by further increasing the volume of the rinsing step. Po^{4+} is strongly retained on TEVA Resin from hydrochloric acid at concentrations between 0.1M and 10M. It does not interfere with Pu determination by ICP-MS, and it will not typically co-precipitate during CeF_3 microprecipitation, but in case the alpha spectrometry source is prepared by electrodeposition it might introduce spectral interference. In that case, an additional removal step might be necessary, for example, evaporation to dryness at high temperature from HCl solutions. U is retained on the DGA Resin and can be removed together with La and Ca with 0.05M HNO_3. If any Po^{4+} passes on to the DGA Resin, it would also be eluted in this rinsing step.

The overall chemical yields obtained for samples of 16-80L are greater than 85%: the chemical yield for ^{242}Pu is 86.4% +/- 3.9%, and for ^{243}Am 94.0% +/- 3.4%.

The minimum detectable activity (MDA) obtained for 16h counting time, an alpha detection efficiency of about 25%, 40L sample volume and a chemical yield of 90% is about 13µBq/L. The MDA obtained for 7 days counting with an alpha detection efficiency of about 25%, 80L sample volume and a chemical yield of 90% is about 1µBq/L. Plutonium isotopes may also be assayed using ICP-MS, with enhanced separation steps to ensure high decontamination from ^{238}U [2].

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News

- ***New lines of products:***

We're very glad to announce that we're further increasing our product range. In addition to our chromatographic accessories we'll now also provide ***classical labware materials*** *(beakers, centrifuge tubes…) and* ***ICP-MS*** *single and multi-element* ***standard solutions.***

For more information please contact us at contact@triskem.fr

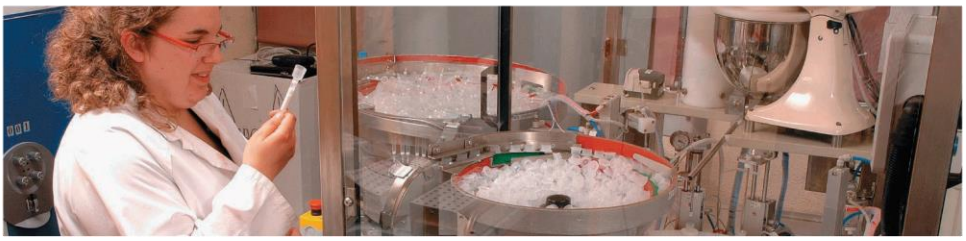
- ***New procedures online:***

Step by step protocols for ***Pu and Np in seawater*** *and* ***Sr-90 in seawater*** *are available from Triskem on request*

Literature

We'd like to draw you attention to three publications that have been published recently:

- *Narek Gharibyan from Ralf Sudowes workgroup at UNLV examined the extraction of Am and Cm from acid solutions on various extraction chromatographic resins (http://dx.doi.org/10.1080/07366299.2014.884888).*
- *Jake Surman from Jackie Pates workgroup at Lancaster University has been working on the characterisation of the upcoming TK100 Resin (http://dx.doi.org/10.1016/j.talanta.2014.06.041).*
- *Emmanuelle Nottoli did part of her PhD in CEA on the use of CL Resin as part of a process for the determination of I-129 from spent resin by AMS (http://dx.doi.org/10.1016/j.apradiso.2014.01.010).*



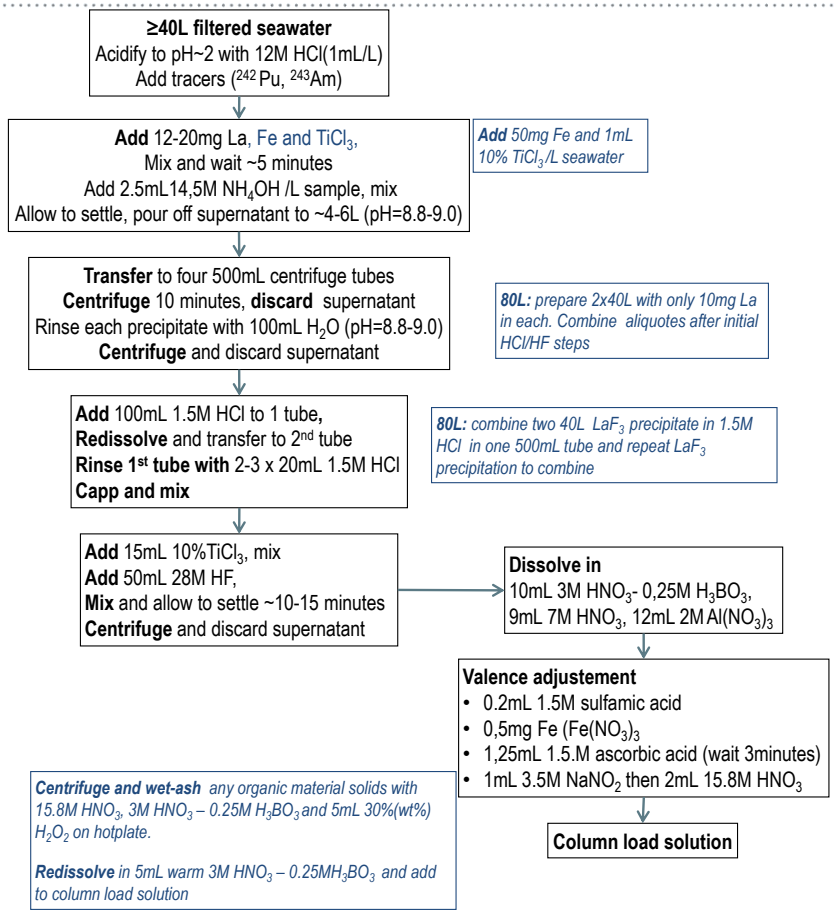


Figure 1: Pre-concentration scheme for actinides in 80L seawater sample [1].

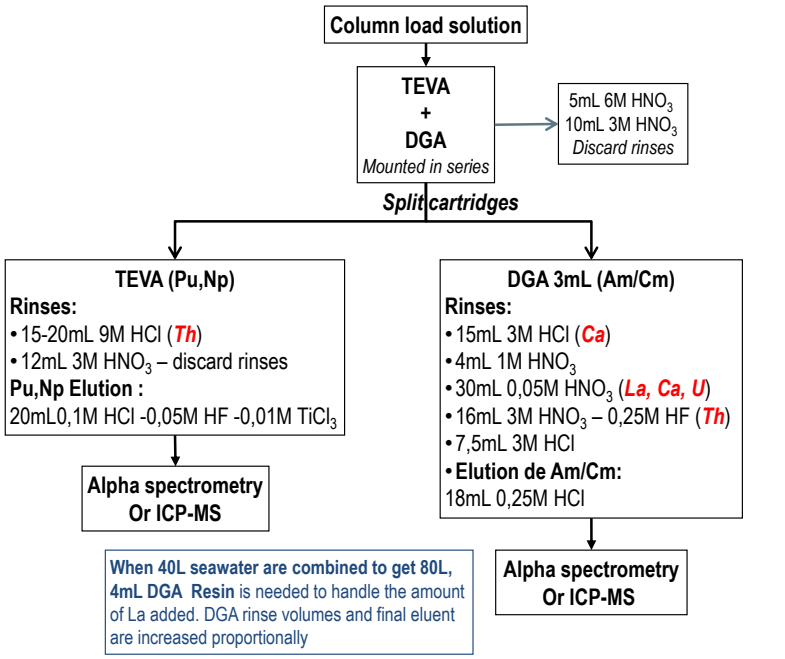


Figure 2: Separation scheme for actinides in 80L seawater sample [1].

**For Pu determination by ICP-MS an additional purification of the Pu fraction is necessary to further reduce U. By using an UTEVA/DGA protocol, U removal will be up to 10E6 - 10E7, using DGA only up to 10E5 - 10E6.*

Two new rapid methods for the determination of radiostrontium in seawater samples have been developed at the Savannah River National Laboratory (SRNL). One method allows for the determination of both ^{89}Sr and ^{90}Sr in up to 2L seawater samples. Details of the procedure are shown in figure 3. Two purified fractions are obtained, a Sr fraction (via SR Resin) and a Y fraction (via DGA Resin). ^{90}Sr can thus be determined in two different ways: by LSC measurement of the Sr fraction, or via ^{90}Y through Cerenkov or LS counting of the Y fraction. This approach also allows for the determination of ^{89}Sr and ^{90}Sr through independent measurements, especially interesting in case of very high activity ratios. ^{89}Sr can be determined by Cerenkov counting of the Sr fraction, whereas ^{90}Sr can directly be determined via ^{90}Y as discussed before.

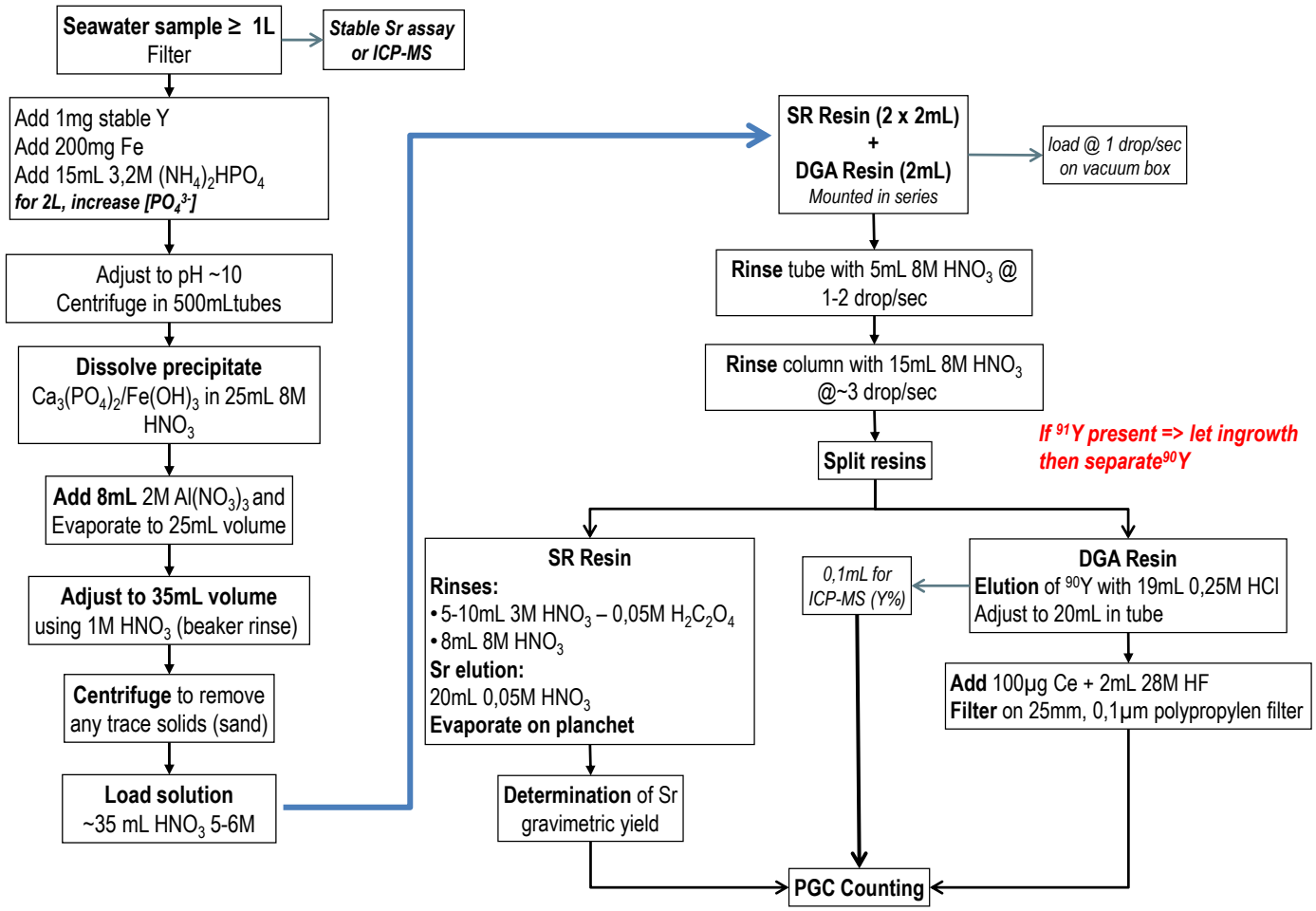


Figure 3: Separation scheme of strontium in seawater samples [3].

^{90}Sr results obtained for 1 to 10L seawater samples via ^{90}Sr and ^{90}Y with both methods, are given in Table 1. Chemical yields were determined by ICP-MS using stable Sr or Y.

The second method, a rapid method for ^{90}Sr in large seawater allows assaying ^{90}Sr at very low levels. The new method employs novel pre-concentration steps to collect ^{90}Y quickly from up to 10L seawater to achieve a very low minimum detectable activity (MDA) [3]. An MDA for ^{90}Sr of 0.61 mBq L^{-1} may be obtained with this method using a 10 L seawater sample aliquot, counting 1000 minutes with gas flow proportional counting. Following the pre-concentration of ^{90}Y from the seawater, DGA Resin is used to rapidly separate ^{90}Y from seawater samples and remove additional beta interferences. Gas flow proportional counting, liquid scintillation or Cerenkov counting may be used to count the purified ^{90}Y and determine ^{90}Sr present in the seawater sample. This new sample preparation method takes < 8 hours.

AGENDA

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

° **TrisKem Users' Group Meeting**, 16/09/2014, **Bath (UK)** *http://www.triskem-international.com/pageLibre0001440d.asp*

° **ERA12 - Nuclear & Environmental Radiochemical Analysis**, 17-19/09/2014, Bath (UK) *http://www.rsc.org/Membership/Networking/InterestGroups/Radiochemistry/ERA12/?CFID=636621&CFTOKEN=66f83f173b853bb4-BD0FEFBC-063C-0FE8-99DFE8BF86289728*

° **ISTR 2014 - The 8th International Symposium on Technetium and Rhenium: Science and Utilization,** 29/09-03/10/2014, Pornichet La Baule (FRANCE) *http://istr2014.sciencesconf.org/*

° **TrisKem Users's Group Meeting**, 6-7/10/2014, **Moscow (RUSSIA)** – more information soon on our website or contact Tatiana Semenova at ***tsemenova@triskem.fr***.

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

Volume sample (L)	Spiked activity ^{90}Sr (mBq.L^{-1})	Chemical yield Sr (%) *SR Resin use*	Chemical yield Y (%) *DGA Resin use*	Number of replicates	Bias (%)
1	148	88.8 +/- 5.3 *(1SD)*	na	11	1.2
		na	95.0 +/- 1.6 *(1SD)*		3.1
2	148	81.9 +/- 4.1 *(1SD)*	na	4	4.2
		na	89.1 +/- 2.8 *(1SD)*		6.6
4*	740	na	91.6 +/- 2.6	1	-2.0
4*	74	na	88.7 +/- 2.5	1	0.0
10*	74	na	93.0 +/- 2.4 *(1SD)*	3	-2.7

Table 1: Results obtained for 2 hours counting; * values obtained using DGA Resin only.

Results indicate that ^{90}Sr can be determined accurately, and with high yield, through both fractions.
Samples of 4 up to 10L, in more recent tests even up to 40L, were tested with this scheme. The chemical yield determined via stable Y is 91.9% +/- 2.5%. The average bias is -2.0%.

Bibliography

[1] Maxwell, S.L., et al., Rapid determination of actinides in seawater samples. J Radioanal Nucl Chem (2014), doi: 10.1007/s10967-014-3079-0

[2] Maxwell, S.L., et al., Rapid Determination of 237Np and Plutonium Isotopes in Urine By Inductively-Coupled Plasma Mass Spectrometry and Alpha Spectrometry. Health Physics (2011) - Vol 101 - Issue 2 - pp 180-186

[3] Maxwell, S.L., et al., Rapid determination of radiostrontium in seawater samples. J Radioanal Nucl Chem (2013), doi: 10.1007/s10967-013-2430-1

InBrief: TrisKem Users' Group Meeting, Bath

We are pleased to invite you to our Users' Group Meeting taking place on the **16th of September** in Bath (UK).

During the meeting amongst others the following topics will be discussed:

- Rapid methods for the determination of Actinides and Sr

- Determination of radionuclides in large environmental samples

- Determination of long-lived radionuclides in nuclear waste and decommissioning samples

- New developments

You are very welcome to present your work !

If you wish to participate, could you be so kind to fill the registration form available at http://www.triskem-international.com/iso_album/registration_form_ugm_bath.pdf and send it by e-mail to **tsemenova@triskem.fr**, fax +33(2) 99 05 07 27 or postal mail to TrisKem International, Parc Lormandière, Bat.C – Campus de Ker Lann, 35170 Bruz, France until the 1st of September 2014.

If you wish to present your work, could you be so kind to fill the registration form enclosed and send it by e-mail to **abombard@triskem.fr**

We are looking forward to meeting and discussing with you !

http://www.triskem-international.com/pageLibre0001440d.asp

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