

Determination of Actinides and Strontium
in large soil samples

PAGE 1

Tips and tricks: Cartridges accessories,

PAGE 2

Agenda

PAGE 4



● **Determination of Actinides and Strontium in large soil samples**

This issue of the TKI is dedicated to procedures used for separation of actinides and strontium from large (>30g) soil/sediment samples. A crucial, and usually very time-consuming, step within the analysis of large samples is the treatment of the sample before the actual separation of the analytes. An efficient solubilisation of radionuclides prior to their subsequent separation and measurement is key for accurate analysis, as is their pre-concentration and the removal of matrix elements.

Horwitz et al.^[1] have proposed a procedure for the preconcentration and sequential separation of Pu and Am from 100g soil/sediment sample. DGA,N resin is used for the preconcentration step as it strongly retains actinides, especially Am, even in presence of large quantities of iron, while the greatest part of the matrix is eliminated. This procedure is comprised of 4 steps:

- 1/ Drying and ashing of the soil sample;
- 2/ Leaching of the mineralized ash with 6M HCl followed by preconcentration of Am and Pu on DGA,N resin at elevated flow rates.
- 3/ Am and Pu are eluted from DGA,N resin and separated using combined TEVA resin and DGA,N resin. The former allows for Pu and Th separation. The later allows for retention of Am and lanthanides;
- 4/ Purification of Am from lanthanides on TEVA resin in SCN⁻ medium.

The procedure reported by Horwitz et al. allows obtaining results in about 5h (excluding alpha spectrometry); however obtained chemical recoveries were in general not very elevated.

Tait et al.^[2] have modified Horwitz' procedure to use it as a routine method for monitoring Am and Pu (Figure 1). They used a microwave supported acid extraction of the soil sample instead of standard leaching. (NEXT PAGE 2)

● **DEAR CUSTOMERS**

We would like to use the end of the year to thank you for your confidence and your support. Thanks to your demands and suggestions we were able to integrate new products and applications into our catalogue. To continue this fruitful collaboration, please do not hesitate to contact us in case of any specific needs.

For any technical questions please get in touch with our technical support, who is at your disposal via e-mail contact@triskem.fr or phone +33 2 99 05 00 09.

If you have developed an application, a resin or another interesting product, please do not hesitate to contact us, we would be very pleased to test your development and to evaluate its market potential.

We dedicate this edition of our Triskem Info to the separation of strontium and actinides from large soil samples, as we have received several requests concerning these subjects within the past months. If you wish to read about a particular topic please let us know.

I wish you and your families a Merry Christmas and a Happy New Year 2014.

Michaela Langer
TRISKEM CEO

TRISKEM INFOS

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

N°11

December

2013

SOIL
SAMPLING
ANALYSIS
PROCEDURE



Tips and Tricks

- Vacuum box:**

AC-1000-TUBE-PE replaces
AC-1000-IT



Figure 2: AC-1000-TUBE-PE



Figure 3: AC-1000-TUBE-PE in AC-1000-OT for set-up on vacuum box

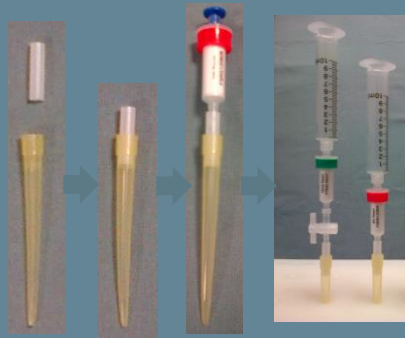


Figure 4: same set-up of AC-1000-TUBE-PE as former AC-1000-IT

- AMP-PAN resin:**

AMP-PAN resin is now available upon request as 2mL, 5mL, 8mL and 10mL pre-packed columns in 0.01M HNO₃.

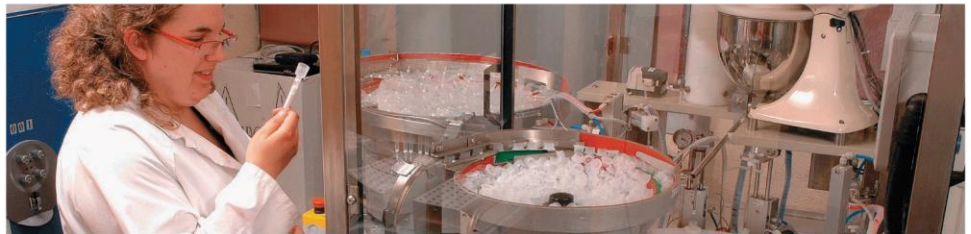
Join us on

[LinkedIn](#)



and

[Environmental Expert](#)



The authors introduced a second TEVA resin step (Fig.1) in order to retain any residual Th that could breakthrough from the first TEVA resin for the case of Th rich soil/sediment samples.

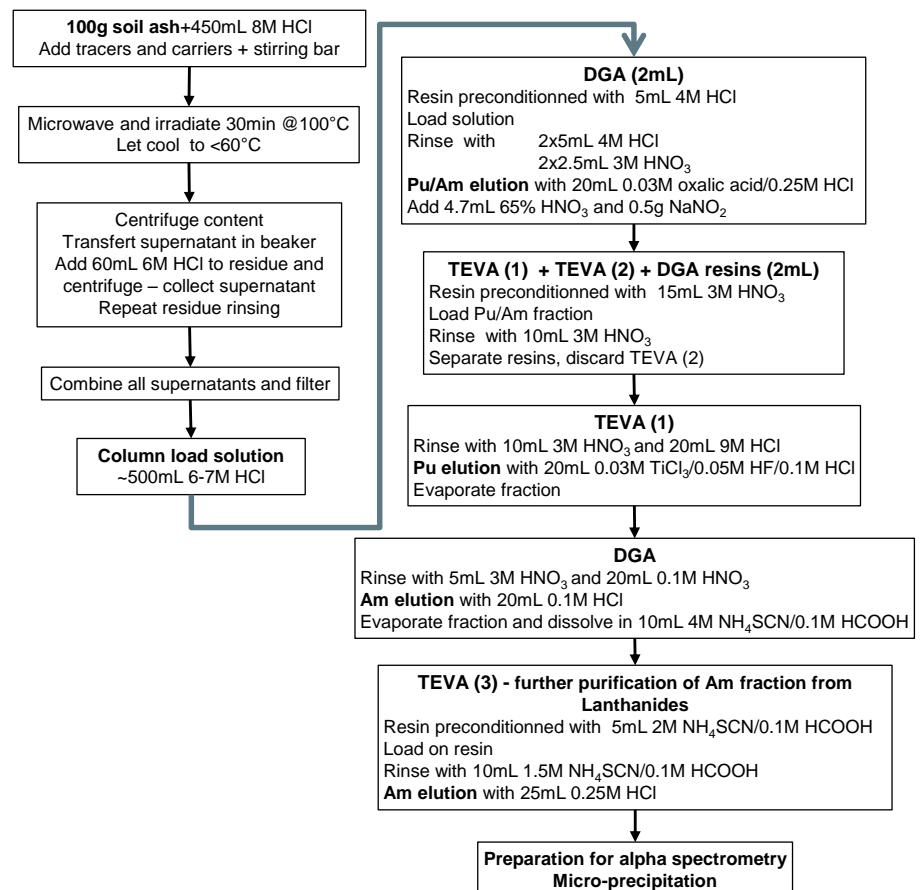


Figure 1: Separation scheme for the separation and determination of Pu, Am from 100g soil/sediment samples^[1].

The results obtained with the modified method are in good agreement with the PTB reference value (Table 1). Overall the authors obtained chemical yields of 87% (s=11%) for Pu and 72% (s=14%).

	Number of values in proficiency test free of outliers	PTB Reference values / Bq.kg ⁻¹ of dry mass	Values with modified method / Bq.kg ⁻¹ of dry mass	Mean of Lab means / Bq.kg ⁻¹ of dry mass
²³⁸ Pu	12	19.6 (s=1.3)	18.6 +/- 1	20 (s=1.4)
^{239/240} Pu	20	1.1 (s=0.1)	1.3 +/- 1	1.2 (s=0.3)
²⁴¹ Am	24	154 (s=5)	134 +/- 5	133 (s=12)

Table 1: Results of analysis with modified method for Pu in soils and ²⁴¹Am in river sediments^[2].

Authors also carried out experiments on soils from moor pasture (10 analyses) and high organic content moor (4 analyses). Recoveries in ²⁴²Pu were 69% (s=14%) and 66% (s=11%) respectively for moor pasture and rich organic content moor. Recovery in ²⁴³Am for both moors was 69% (s=8% and 14% respectively).

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

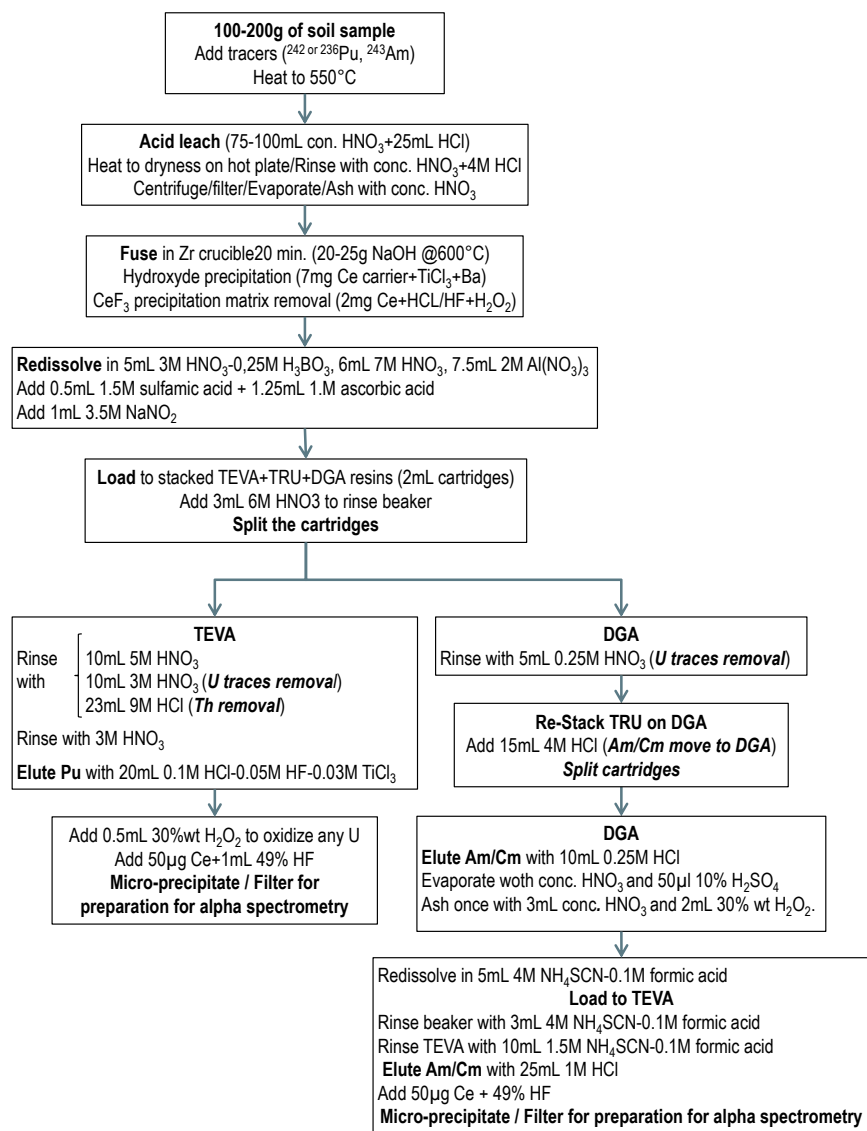
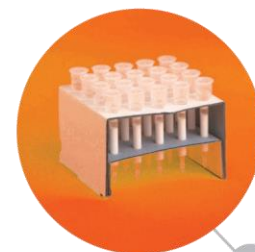


Figure 5: Separation scheme for the separation and determination of Pu, Am from 100 - 200g soil/sediment samples^[4].

⁹⁰ Sr Reference (mBq·g ⁻¹)	Number of analysis	Mean ⁹⁰ Sr measured (mBq·g ⁻¹)	Mean Sr carrier (%)
59.2	7	57.8 +/- 1.7	89.3 +/- 4.7
11.84	7	11.5 +/- 0.7	89.6 +/- 2.7
5.92	7	5.95 +/- 0.22	94.0 +/- 2.6

Table 2: Results obtained on ⁹⁰Sr with procedure for ≥ 50g soil samples; values were corrected for native content in soil of 1.35mBq ⁹⁰Sr·g⁻¹ [5].

A similar method is being tested by Maya Jaeggi from PSI (Switzerland) for 30g samples using anion exchange and UTEVA after the first TEVA resin cartridge^[3]. Reported yields are high, samples with extremely high Ca content show somewhat lower yields though. She further discussed the integration of Sr into the separation scheme.

A different approach is suggested by Maxwell et al.^[4] for soil samples up to 200g. Matrix removal is reached through two subsequent co-precipitation steps (Fe(OH)₃ and CeF₃). The CeF₃ co-precipitation is performed under oxidative conditions. It thus not only greatly reduces the amount of matrix elements in the sample but also removes silicate interference. It also enhances U decontamination as U(VI) co-precipitates under the given conditions. The separation is performed using stacked TEVA/TRU/DGA resins followed by an Am purification from lanthanides using SCN⁻ / TEVA resin, as shown in Figure 5. The authors report yields in the order of 80 – 90% for Pu and Am and detection yields down to 1 mBq/kg (16h counting time).

Maxwell et al.^[5] also updated the procedure used at the Savannah River Site Laboratory for the determination of ^{89,90}Sr in 50g or more soil samples. The iron hydroxide precipitation uses iron naturally present in the samples. The fluoride precipitation allows for the removal of silicates amongst other matrix components which later facilitate flow rate through the resin. Another advantage of the method is that the amount of Sr resin used is lower than e.g. suggested in the ISO 18589-5:2000 norm (2.8g). This method was tested with soil samples spiked with different activities; results are summarized in Table 2.



AGENDA

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

° **International Conference on Radiopharmacy and Radiopharmaceuticals,**

27-28/03/2014, Madrid (Spain)
<http://www.waset.org/flyers/2014/03/madrid/ICRR>

° **17th Radiochemical Conference,**
 11-16/05/14, Marianske Lazne (Czech Republic) <http://www.radchem.cz/>

° **TrisKem Users' Group Meeting,**
 20/05/2014, location to be announced (France) <http://www.triskem-international.com>

° **TrisKem Users' Group Meeting,**
 22/05/2014, Manchester (UK) <http://www.triskem-international.com>

° **PROCORAD**
 18-20/06/14, Cherbourg (France) <http://www.procorad.org/en/annual-meeting/Next-Meeting>

° **ILSEPT: 2nd International Conference on Ionic Liquids in Separation and Purification Technology,** 28/06-07/02/2014, Toronto (Canada) <http://www.ilsept.com/>

° **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>

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



The results indicate good agreement between reference and measured values, with Sr chemical yield greater than 84%. This also shows that the pre-concentration steps are efficient to remove soil matrix prior to Sr separation. The authors reported a time of analysis of less than 16h (using resin in cartridges).

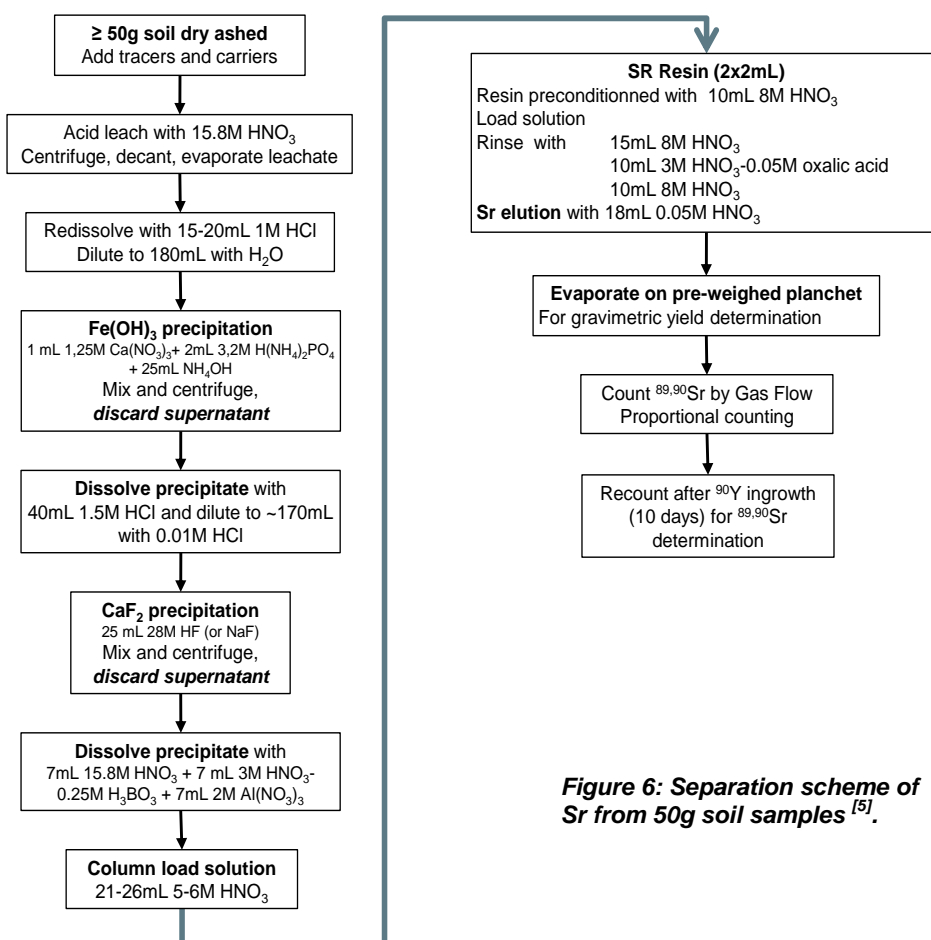


Figure 6: Separation scheme of Sr from 50g soil samples [5].

Bibliography

- [1] Horwitz E.P., Thakkar A.H., McAlister D.R., "A Rapid Method for the Preconcentration of Non-Refractory Am and Pu from 100g Soil Samples". 10th International Symposium on Environmental Radiochemical Analysis, Oxford – UK – September 2006, pp 77 - 85
- [2] Tait D. and Koch B., "Further Development of a Fast Method for Determining plutonium and Americium in Soils in Germany", 11th International Symposium on Environmental Radiochemical Analysis, Chester –UK –September 2010, pp 9-20
- [3] Jaeggi M.: "DGA als Vorkonzentration von Aktiniden zur späteren Trennung und Bestimmung von ^{238,239,240}Pu, ²⁴¹Am und ⁹⁰Sr in Boden- und Sedimentproben", German Users Group Meeting, 12.11.12, Munich - Germany
- [4] Maxwell S.L., "Rapid Method for Plutonium, Americium and Curium in Very Large Soil Samples", Journal of Radioanalytical and Nuclear Chemistry, February 2008, 275(2): pp 395-402
- [5] Maxwell S.L., Culligan B.K., Shaw P.J., "Rapid Determination of Radiostrontium in Large Soil Samples". J. Radioanal. Nuc. Chem. 295(2): pp 965-971(2013)

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