TK227 Resin - Uses for preconcentration and measurement of Sr-90 via Y-90

Developments of alternative supports

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Raddec-Triskem Technical Workshop

Portsmouth – 11/09/2026

TRISKEM
Expertise in Separation Chemistry

Summary

- Context of TK227 Resin developpment
- Characterisation of TK227 Resin
- Applications
- Developments of alternative supports
- Conclusions

Summary

- **Context of TK227 Resin development**
- Characterisation of TK227 Resin
- Applications
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Context of TK227 Resin development

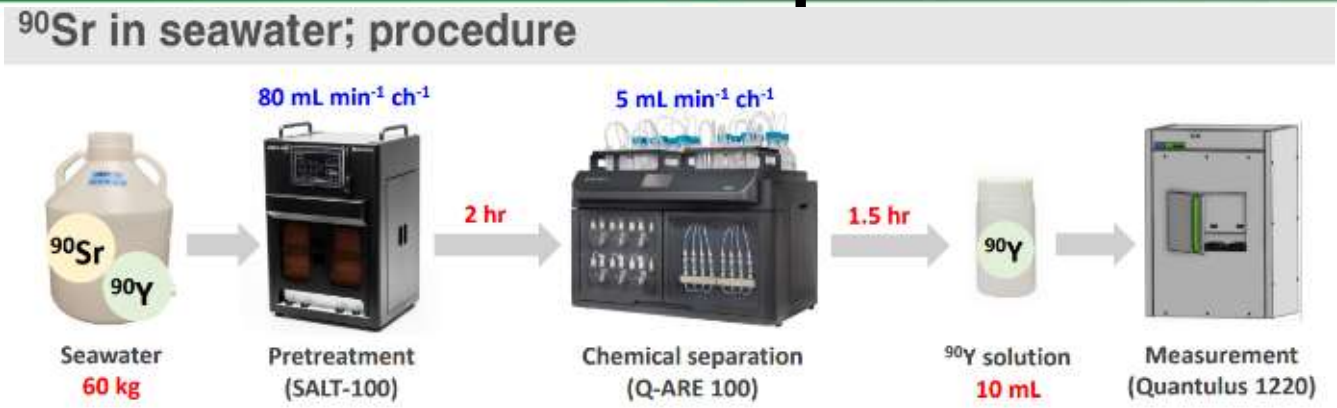
Measurement of Sr-90 via Y-90 in seawater samples by DGA,N Resin [1][2]

- Y-90 at equilibrium with Sr-90
- No need to wait 14 days for the (re-)measure @equilibium after Y-90 ingrowth
  - Rapid methods developped to assay ^{90}Sr at very low levels e.g. Method by Maxwell et al.[1] – 1-40L
  - Method by Kim et al.[2] - 1-50L/60kg

[1] Maxwell, S.L., Culligan, B.K., Hutchison, J.B. *et al.* Rapid determination of ^{90}Sr in seawater samples. *J Radioanal Nucl Chem* **303**, 709–717 (2015). https://doi.org/10.1007/s10967-014-3391-8

[2] Hyuncheol Kim and Gahyun Kim: "A simple and straightforward technique for analyzing radionuclides in sea water" presented at the Raddec/TrisKem Workshop (as part of the 2024 LSC conference) on April 18 in Portsmouth (UK). Available online: https://www.triskem-international.com/scripts/files/662fa5023ef284.68970177/raddec_tki-ws-2024_6_kim.pdf

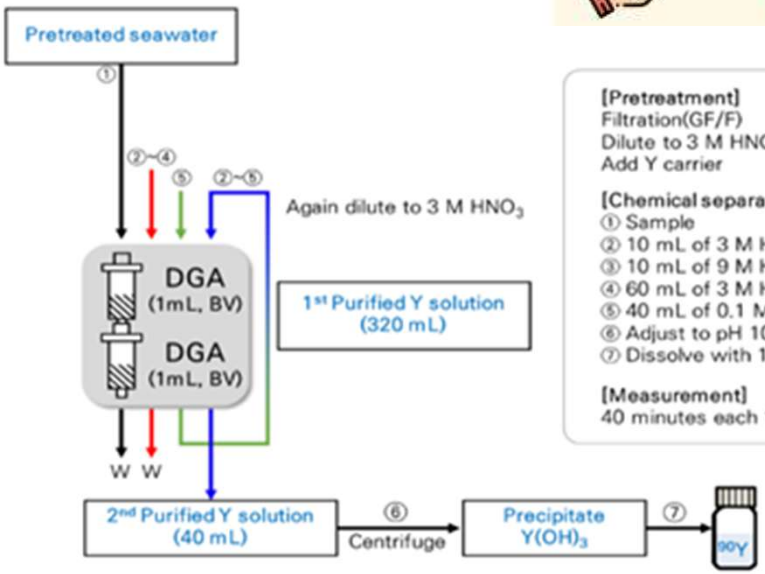
Context of TK227 Resin development



- **Kim et al. [2][3][4]**
- **Gahyun et al. [5]**

Elapsed time for analyzing the 60 kg of seawater?

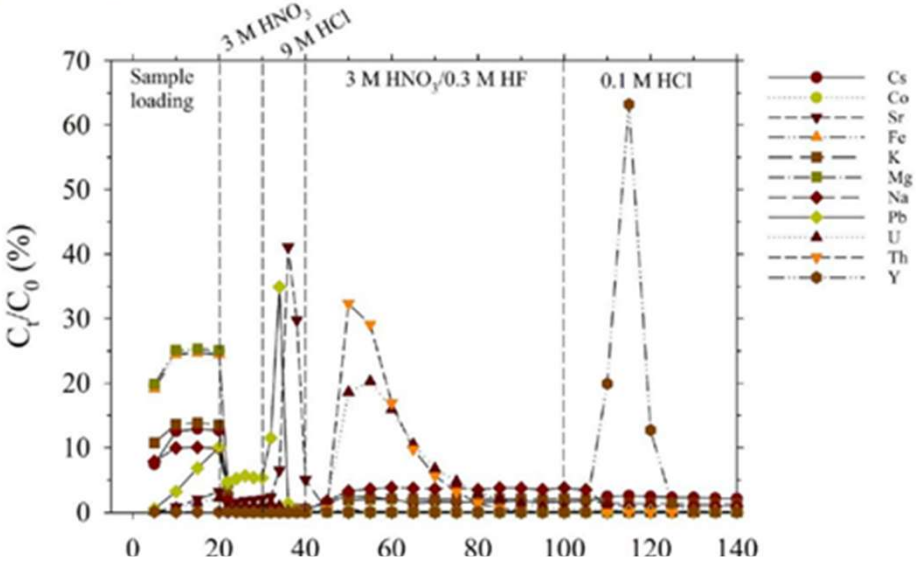
4 hr excepting the measurement time



[Pretreatment]
Filtration(GF/F)
Dilute to 3 M HNO_3 using Conc. HNO_3
Add Y carrier

[Chemical separation]
① Sample
② 10 mL of 3 M HNO_3
③ 10 mL of 9 M HCl
④ 60 mL of 3 M HNO_3/0.3 M HF
⑤ 40 mL of 0.1 M HCl
⑥ Adjust to pH 10 using NaOH
⑦ Dissolve with 10 mL of 0.1 M HNO_3

[Measurement]
40 minutes each 10 times by LSC



[2] Hyuncheol Kim and Gahyun Kim: "A simple and straightforward technique for analyzing radionuclides in sea water" presented at the Raddec/TrisKem Workshop (as part of the 2024 LSC conference) on April 18 in Portsmouth (UK). Available online: https://www.triskem-international.com/scripts/files/662fa5023ef284.68970177/raddec_tki-ws-2024_6_kim.pdf
[3] Video KAERI Method: "Simple and Straightforward technique for Sr-90 analysis in seawater". Available online: https://www.youtube.com/watch?v=i2R2b98oTCU
[4] Hyuncheol Kim, Yoo Gyum Kang, Yong-Jin Lee et al.: "Automated extraction chromatographic radionuclide separation system for analysis of 90Sr in sea water", Talanta 217 (2020) 121055. https://doi.org/10.1016/j.talanta.2020.121055
[5] Gahyun Kim, Sang-Do Choi, Jong-Myoung Lim, Hyuncheol Kim, Strontium-90 levels in seawater southeast of Jeju Island during 2021–2023, Marine Pollution Bulletin, Volume 193, 2023, 115258. https://doi.org/10.1016/j.marpolbul.2023.115258

Context of TK227 Resin development

BUT acidification of 50L seawater @ 3M HNO_3 requires 9,5-10L of cc HNO_3

- Modification of DGA,N Resin to get Y-90 @ lower HNO_3 concentrations

Initial 3M HNO_3 – 50L	9,5L of cc HNO_3
1M HNO_3	3,2L
0,5M HNO_3	1,6L
0,1M HNO_3	320mL

- Ideally, 0,1M HNO_3 would be the best result.

Context of TK227 Resin development

How to incease retention of Y (trivalent elements) @lower HNO_3 concentrations

- **use of ionic liquid**

- ✓ **All work presented about TK227 Resin was performed by I. Dovhyi**

Summary

- *Context of TK227 Resin development*
- **Characterisation of TK227 Resin**
- Applications
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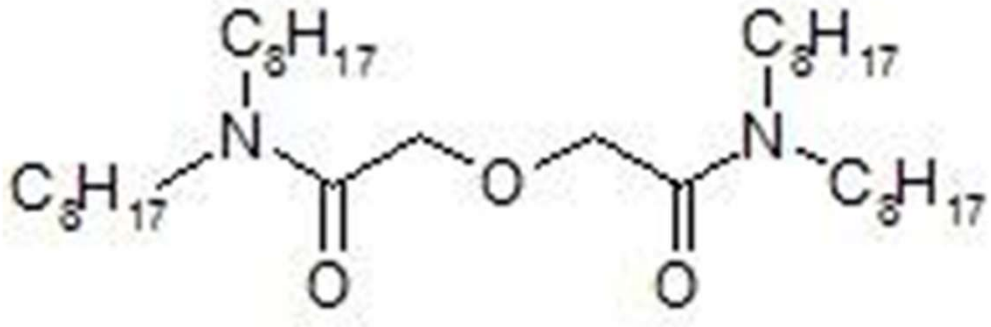
Characterisation of TK227 Resin

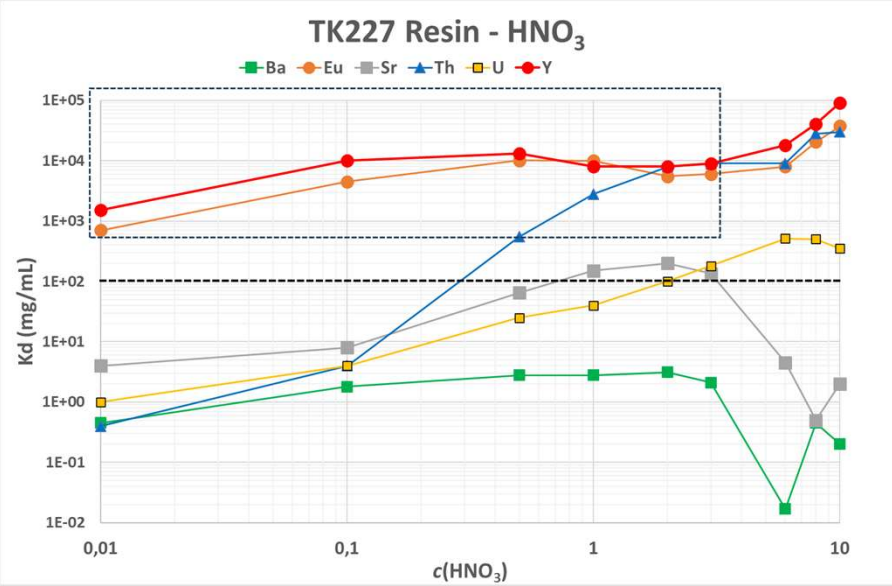
TK227 resin is comprised of

- Inert support
- TO-DGA extractant
- Ionic liquid
- Long chain alcohol

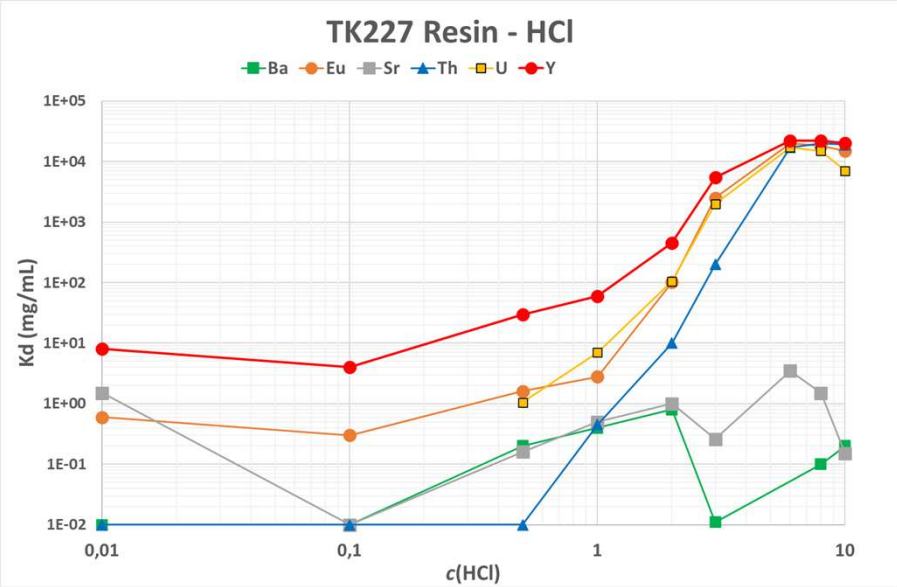


Characterisation of TK227 Resin

Kd values of elements of interest in HNO_3 medium on TK227 Resin



Kd values of elements of interest in HCl medium on TK227 Resin

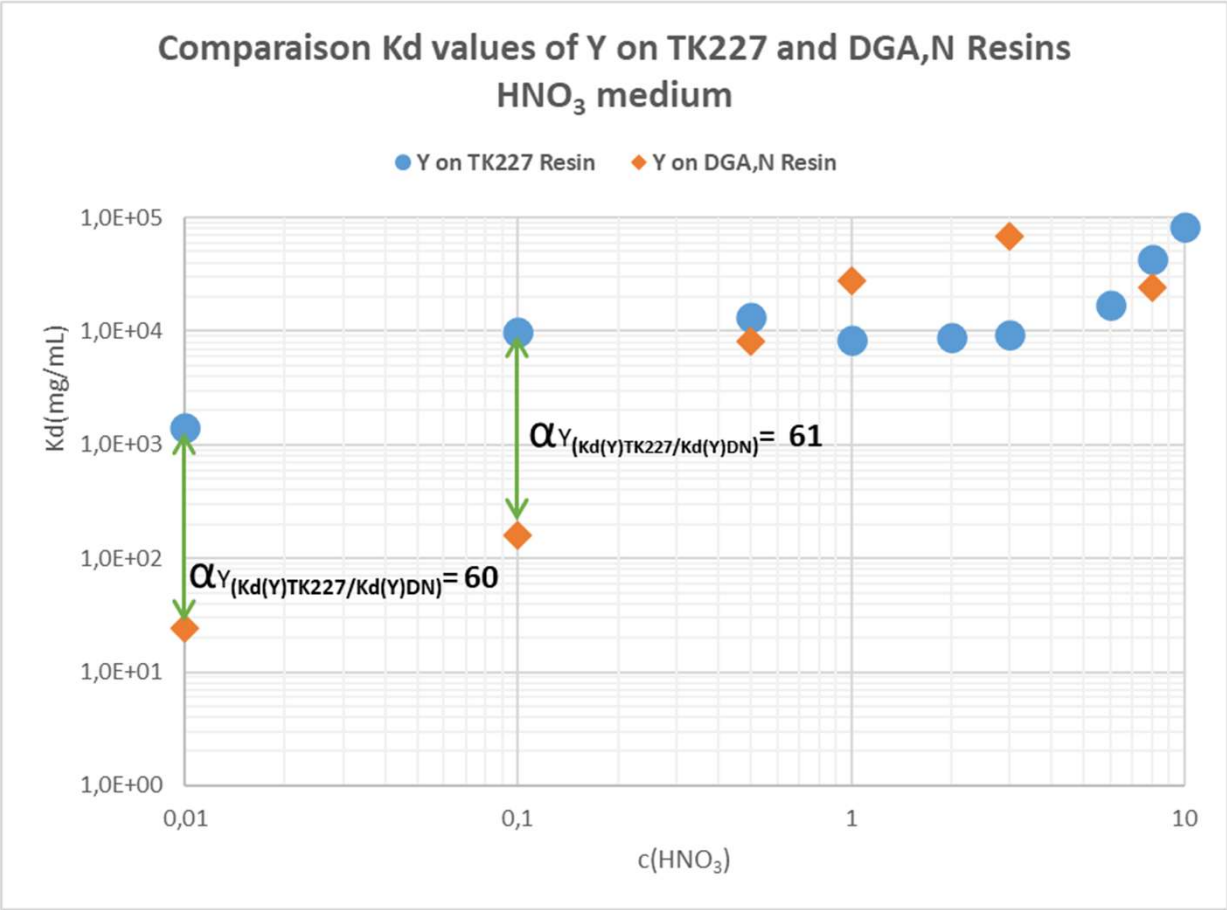


- Strong uptake of Y on the HNO_3 concentrations tested (same for Eu)
- Uptake of Sr between 1 and 3M HNO_3
- Strong Th uptake above 0,5M HNO_3
- Uptake of U above 2M HNO_3

In HCl,

- No retention below 0,5M HCl
- Retention increase for Y, Eu, Th and U at high HCl concentration

Characterisation of TK227 Resin



- ✓ Developped TK227 allows for higher retention of Y at low HNO_3 concentrations
- ✓ Y-carrier needed (minimum of 1mg).

Summary

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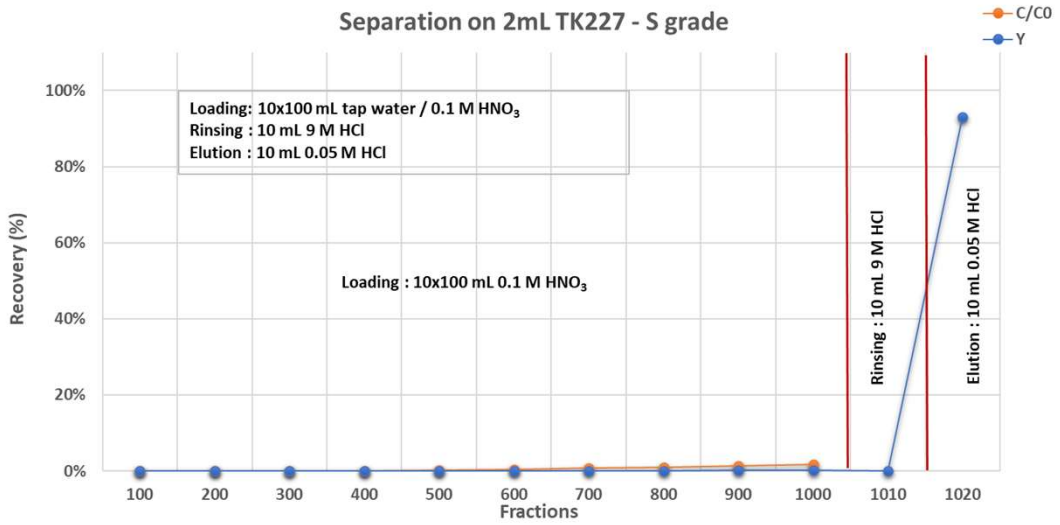
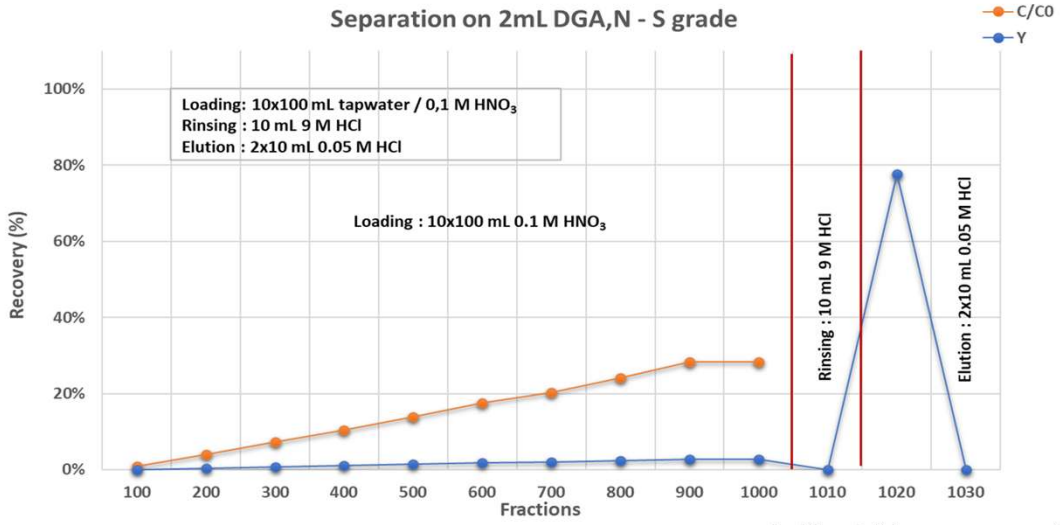
Applications:
Y breakthrough studies - tapwater

1L samples acidified @ pH 1, Test of Y breakthrough on 2mL cartridges of both TK227 and DGA,N Resins (Fractions measured by ICP-MS)





TK227 Resin:

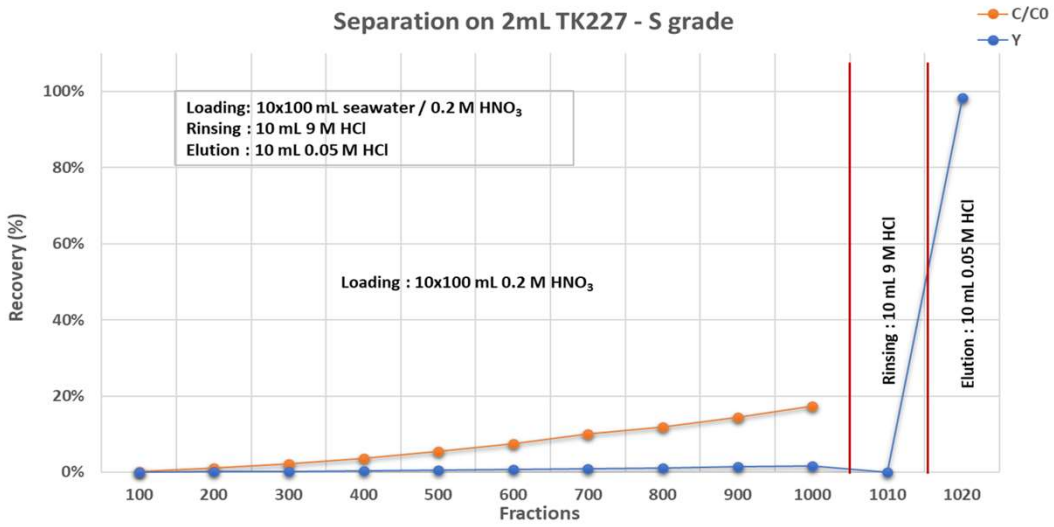
- Slight Y breakthrough observable starting at 700mL but remain quite low.
- Elution of Y in 10-20mL

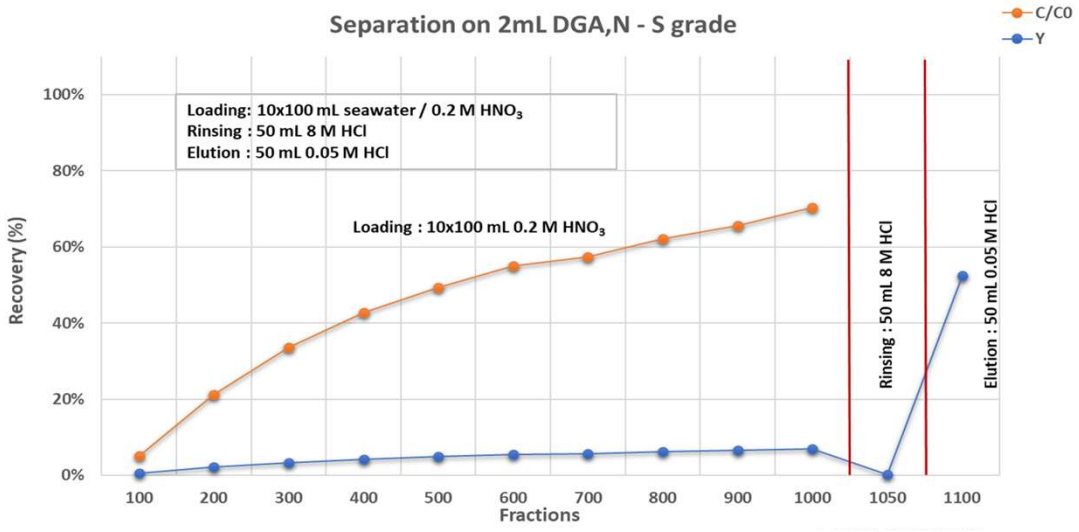
DGA,N Resin:

- Y breakthrough starting immediately with loading at 0,1M HNO_3
- Elution of remaining Y in 10-20mL

Applications:
Y breakthrough studies – seawater

1L samples acidified @ 0,2M HNO_3, Test of Y breakthrough on 2mL cartridge of both TK227 and DGA,N Resins (Fractions measured by ICP-MS)





TK227 Resin:

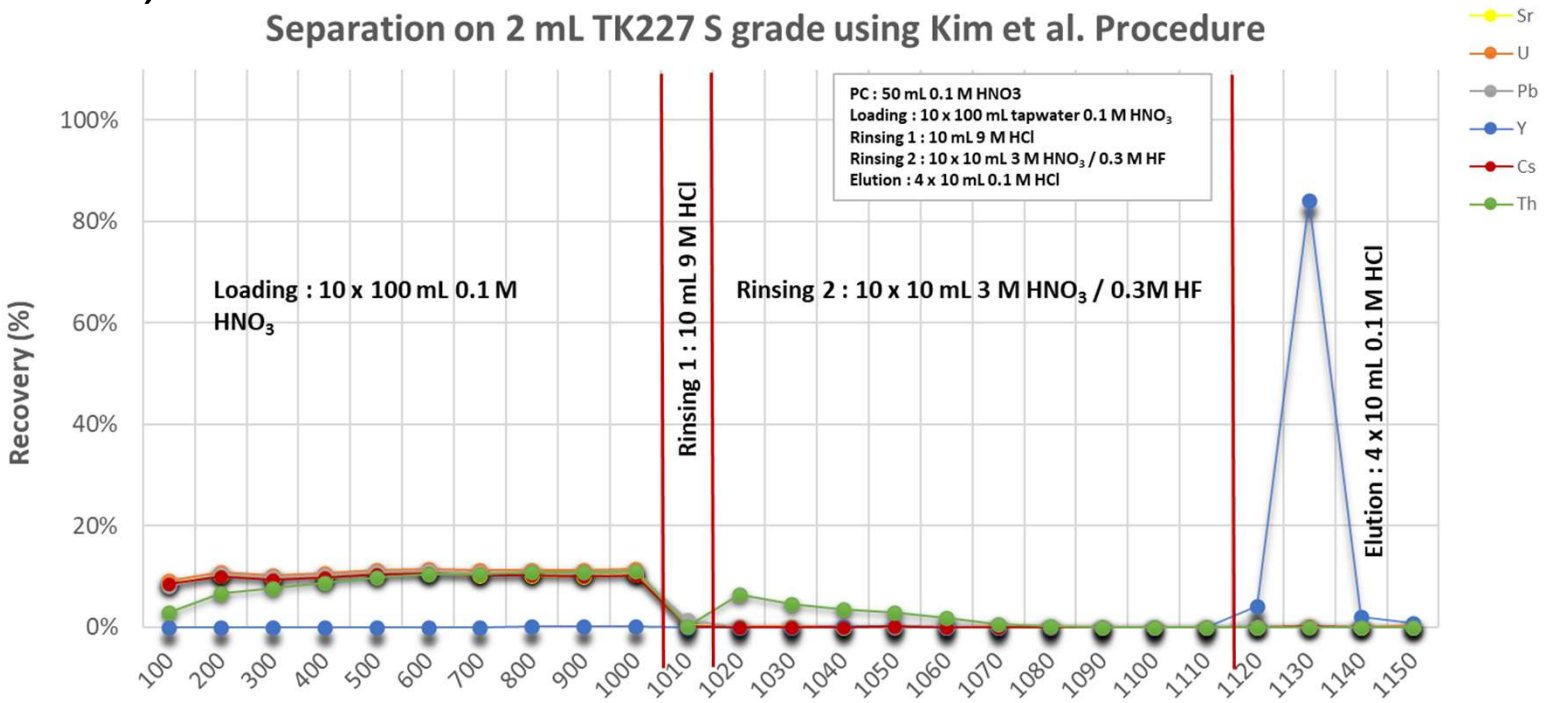
- Y breakthrough observable starting at 300mL. Remain <20%.
- Elution of remaining Y in 10-20mL
- Better Y retention using higher HNO_3 conc. (e.g. 0,5M HNO_3)

DGA,N Resin:

- Y breakthrough starting immediately with loading at 0,2M HNO_3
- Elution of remaining Y in 50mL

Applications:
Elution studies on tapwater sample

1L Tapwater sample acidified @ pH 1, Elution study on 1x2mL cartridge TK227 using Kim et al. Conditions [1] (Fractions measured by ICP-MS)

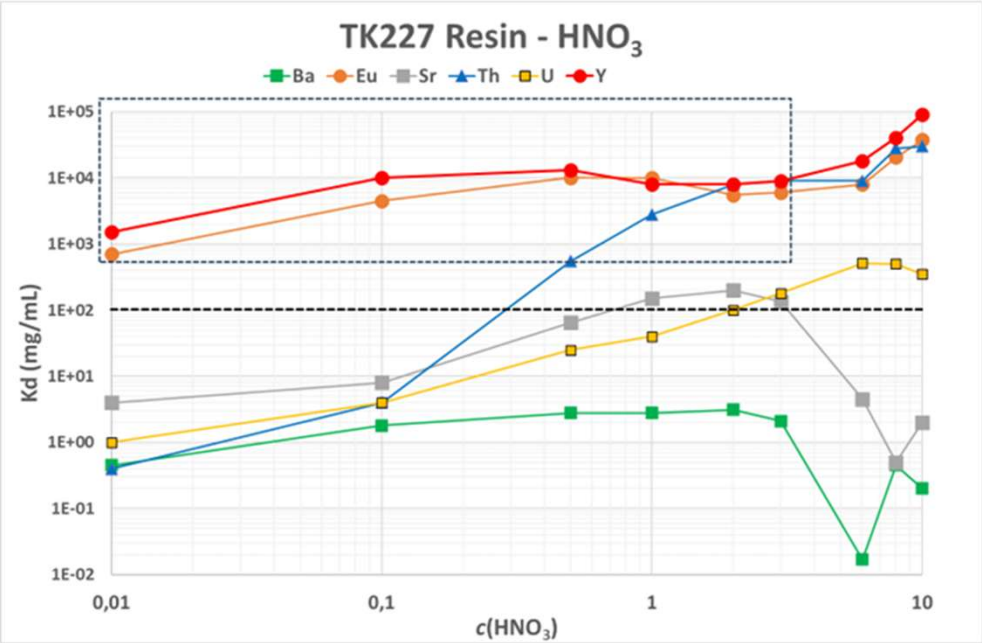


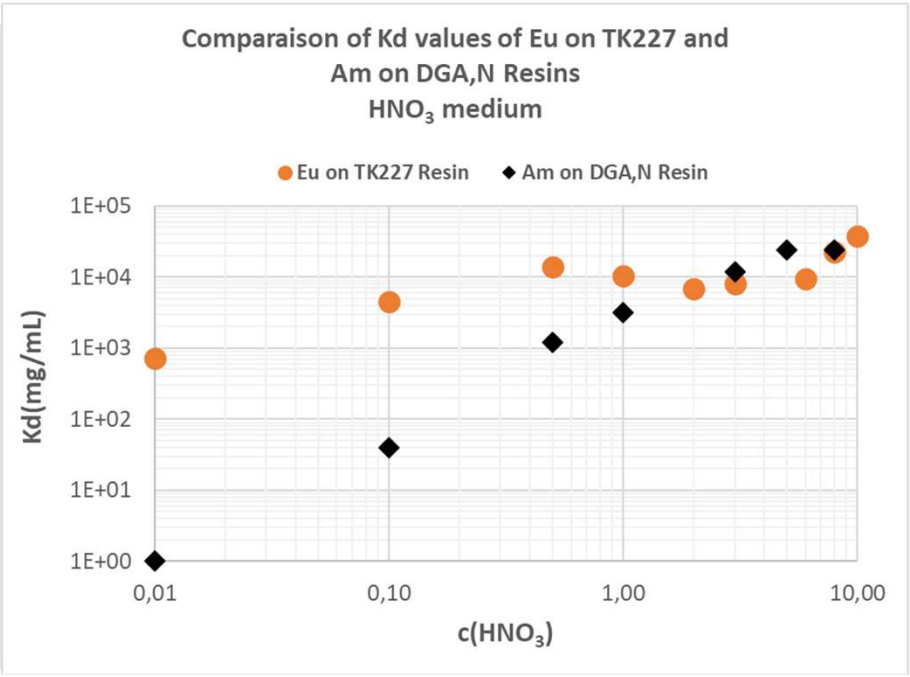
- Th, U, Sr, Pb,Cs elute during Loading and Rinsing 2
- Quantitative recovery of Y in 40mL 0,1M HCl
  - Only one preconcentration/purification step

[1] Hyuncheol Kim and Gahyun Kim: "A simple and straightforward technique for analyzing radionuclides in sea water" presented at the Raddec/TrisKem Workshop (as part of the 2024 LSC conference) on April 18 in Portsmouth (UK). Available online: https://www.triskem-international.com/scripts/files/662fa5023ef284.68970177/raddec_tki-ws-2024_6_kim.pdf

Applications:
Use for Am separation @ low pH

- Eu (proxi for Am) with high uptake between 0,01 and 0,1M HNO_3





Method development to be tested to preconcentrate/purify Am from low HNO_3 concentration

Load @ 0,1M HNO_3; Rinse with 0,1M HNO_3; Hypothesis on rinses: *Rinses with 8-9M HCl then 3M HNO_3/0,3M HF*; Elution Am with 0,1-0,25M HCl

To be used in combination with TK200 Resin for (U,Th, Pu) separation as TK200/TK227 (see Inés Llopart presentation)

Summary

- *Context of TK227 Resin development*
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Development of alternative supports: impregnation of different types of membranes

Resins are not always the optimum format of use depending on final application, sample nature, …

Kinetics of uptake are being quite rapid for most resins

=> Transfer of the impregnation technique from resins to alternative supports

- tests sticks (see presentation of Alex Tribolet) => DTM screening to decide for further chemistry or not

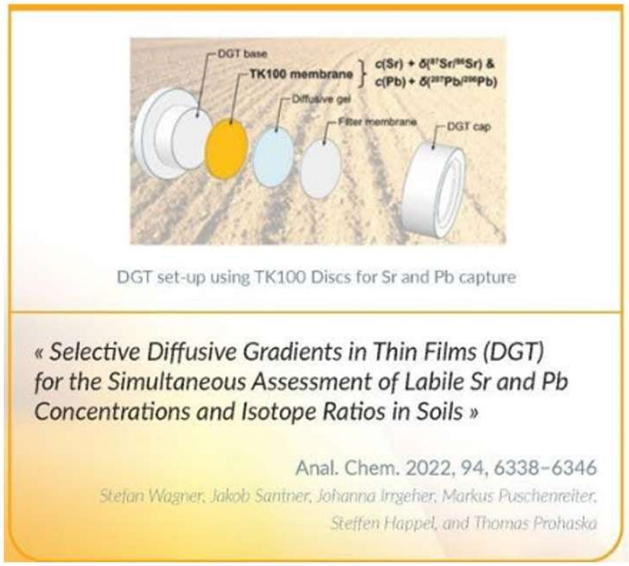
- Filtering membranes,
- Impregnated Thin Layer Chromatography

Development of alternative supports: impregnated discs already in use

Transposition of different resins extractive system onto discs to simplify preconcentration/purification/detection-counting

- Filtration or passive sampling on 25mm or 47mm diameter discs
- Already in-use/published: TK100, TK201 and TK-GA

TK100 Discs for passive uptake of Sr and Pb in soils [1][2]



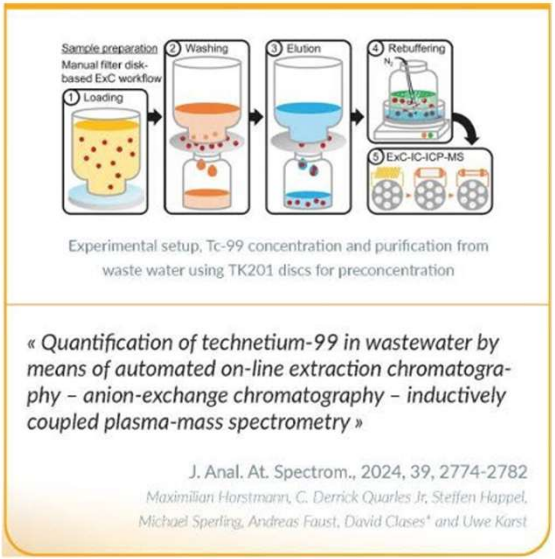
DGT set-up using TK100 Discs for Sr and Pb capture

« Selective Diffusive Gradients in Thin Films (DGT) for the Simultaneous Assessment of Labile Sr and Pb Concentrations and Isotope Ratios in Soils »

Anal. Chem. 2022, 94, 6338−6346

Stefan Wagner, Jakob Santner, Johanna Irrgeher, Markus Puschenreiter, Steffen Happel, and Thomas Prohaska

TK201 discs for Tc-99 preconcentration /determination in waste water [3]



Experimental setup, Tc-99 concentration and purification from waste water using TK201 discs for preconcentration

« Quantification of technetium-99 in wastewater by means of automated on-line extraction chromatography – anion-exchange chromatography – inductively coupled plasma-mass spectrometry »

J. Anal. At. Spectrom., 2024, 39, 2774-2782

Maximilian Horstmann, C. Derrick Quarles Jr, Steffen Happel, Michael Sperling, Andreas Faust, David Clases* and Uwe Karst

[1] S. Wagner, J. Santner, J. Irrgeher et al. Selective Diffusive Gradients in Thin Films (DGT) for the Simultaneous Assessment of Labile Sr and Pb Concentrations and Isotope Ratios in Soils. Anal Chem, 2022, 94, 16, 6338-6346, https://doi.org/10.1021/acs.analchem.2c00546

[2] S. Wagner, J. Santner, M; Puschenreiter, et al. Prediction of Radiogenic Sr and Pb Isotope Signatures in Plants using Diffusive Gradients in Thin Films. Analytical and Bioanalytical Chemistry, 2026, 418, 1541–1555, https://doi.org/10.1007/s00216-026-06315-6

[3] M. Horstmann , C. Derrick Quarles Jr , S. Happel et al. : Quantification of technetium-99 in wastewater by means of automated on-line extraction chromatography – anion-exchange chromatography – inductively coupled plasma-mass spectrometry. J. Anal. At. Spectrom., 2024, 39, 2774-2782, https://doi.org/10.1039/D4JA00270A

Development of alternative supports: impregnated discs already in use

Transposition of different resins extractive system onto discs to simplify preconcentration/purification/detection-counting

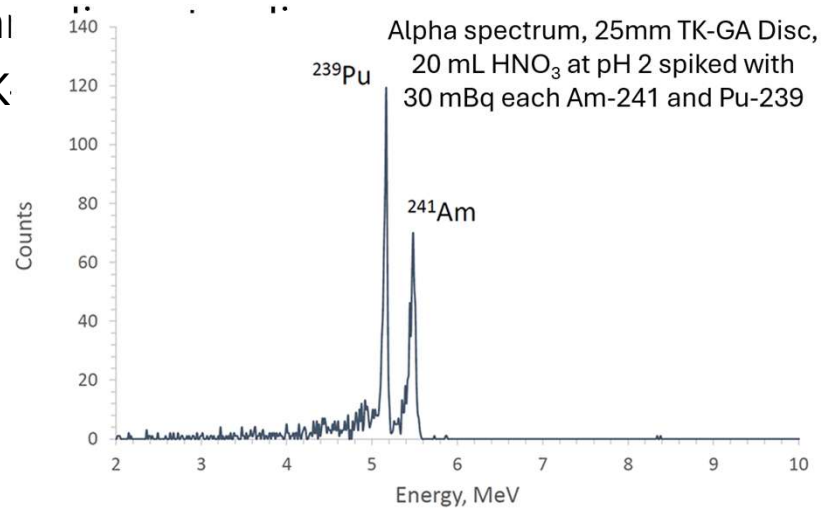
- Filtration or passive sampling on 25mm or 47m
- Already in-use/published: TK100, TK201 and TK

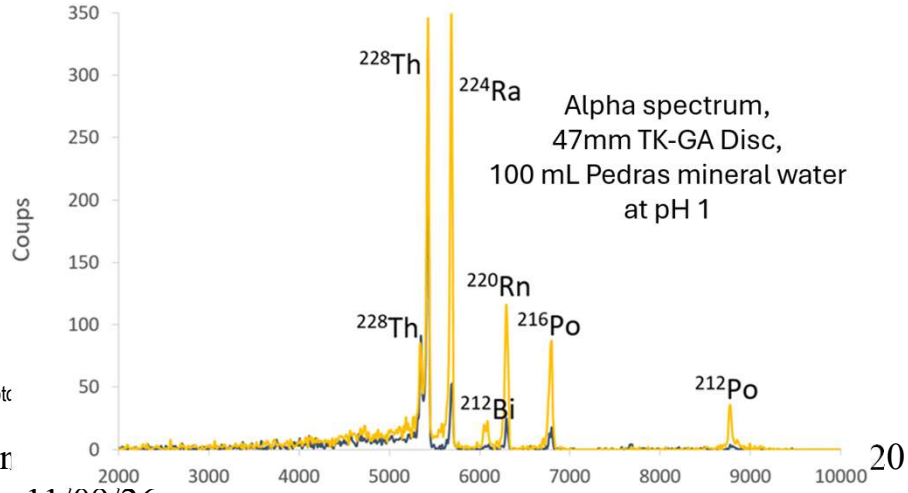
Typical applications of TK-GA Discs [4]:

- pH 1 – 2 (for some actinides up to 3M HNO_3)
- Disc preconditioning: 20% EtOH:H_2O, H_2O, then loading medium
- Sample Loading, flow rate: 1 - 10mL/min (Lower flow rates favors better resolution)
  - 25 mm Discs => up to 100mL samples
  - 47 mm Discs => up to 1L (higher volumes possible depending on extractant system)
- Rinsing with H_2O and 20% EtOH: H_2O
- Drying and glueing onto steel disc => alpha spec

Optional: subsequent Alpha/Beta by LSC

=> 100mL sample processed in 1,5h prior to alpha spec





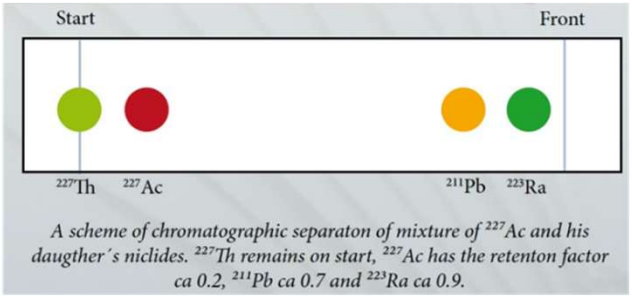
[4] C. Bailly et al. Selective impregnated Membrane Filters for Direct Alpha Spectrometric Analysis of Actinides; Applied Radiation and Isotc

Development of alternative supports:

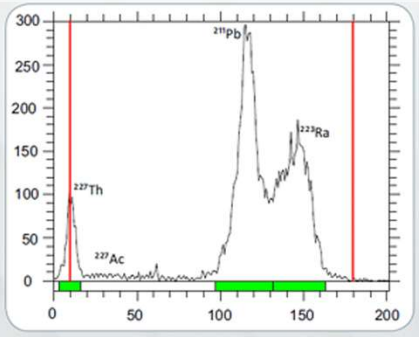
Impregnated TLC (iTLC) supports for Fast Radionuclidic purity QC Checks

DGA Sheets by Kozempel et al. (CTUP) [5][6]

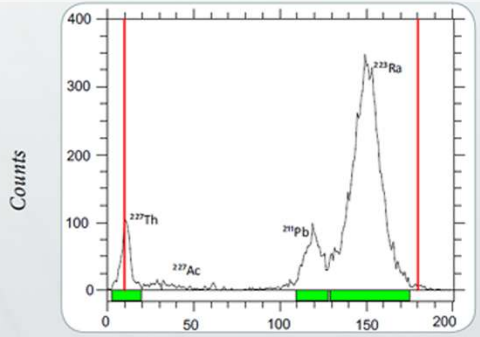
QC of radionuclides and generator eluents (e.g.: Ra-223, Ac-225/Bi-213, Pb-212,...)



A scheme of chromatographic separaton of mixture of ^{227}Ac and his daugther´s niclides. ^{227}Th remains on start, ^{227}Ac has the retenton factor ca 0.2, ^{211}Pb ca 0.7 and ^{223}Ra ca 0.9.



Radiochromatogram measured immediately after separaton. Low abundant radiatons of ^{227}Ac were not detected.



Radiochromatogram measured one hour afer separaton. Decay and ingrowth of ^{211}Pb is clearly visible.

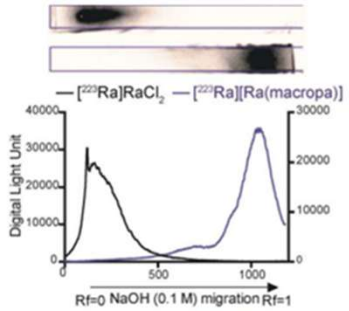


Fig. 1 Formation of [^{223}Ra][Ra(macropa)] under migration of [^{223}Ra]RaCl$_2$ (top) and [^{223}Ra][Ra(macropa)] (bottom) on DGA-coated chromatographic strips. [5]

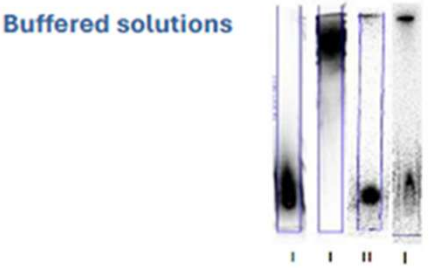


Fig. 2 TLC autoradiographic readings of [^{223}Ra][Ra(macropa)] (10 mM), DOTA (10 mM) and EDTA (5mM) migrated on DGA-coated TLC using a NaOH (0.1 M) mobile phase.
I. [^{223}Ra]RaCl$_2$ migration (Rf≈0)
II. [^{223}Ra][Ra(macropa)] (Rf ≈ 1)
III. ^{223}Ra tentatively chelated with DOTA showing unsuccessful radiolabeling (85% remained unchelated)
IV. ^{223}Ra partially chelated with EDTA (65% remained unchelated).[5]

Serum

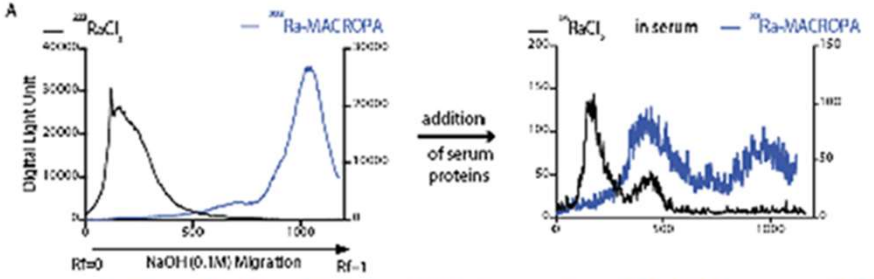


Fig. 3 TLC profiles of pure [$^{223/224}$Ra]RaCl$_2$ and labeled macropa in PBS (left) and in human serum (right). [^{223}Ra]RaCl$_2$ (black trace) and [^{223}Ra][Ra(macropa)] (blue trace) mixed in serum were poorly separated, questioning the suitability of the DGA sheets to detect Ra decomplexation in serum. Weak ionic interactions of [^{223}Ra]Ra^{2+} or [^{223}Ra][Ra(macropa)] with proteins may occur and result in the inability of TLC to clearly separate each species. [5]

[5] D. S. Abou, N. A. Thiele, et al.: Towards the stable chelation of radium for biomedical applications with an 18-membered macrocyclic ligand. Chem. Sci., 2021, 12, 3733-3742, https://doi.org/10.1039/D0SC06867E
[6] J. Svedjehed et al.: "New extractant impregnated iTLC-SG paper facilitates improved TLC analysis for Cu radiolabelled peptides", poster presented at TERACHEM 2022, 14 – 17 September 2022, Bressanone (Italy). Poster available on https://www.triskem-international.com/

Development of alternative supports:

Impregnated TLC supports for Fast Radionuclidic purity QC Checks

CU Sheets by Svedjehed et al. [6]:

QC of Cu radiolabeled peptides (labeled vs free Cu): Both iTLC paper (SG and SG-CU) developed in less than 10 min. while Whatman paper took 25 – 30 min.

On-going studies on iTLC sheets : Zr, TK201, … // Possible use of 2D TLC for radionuclide screening

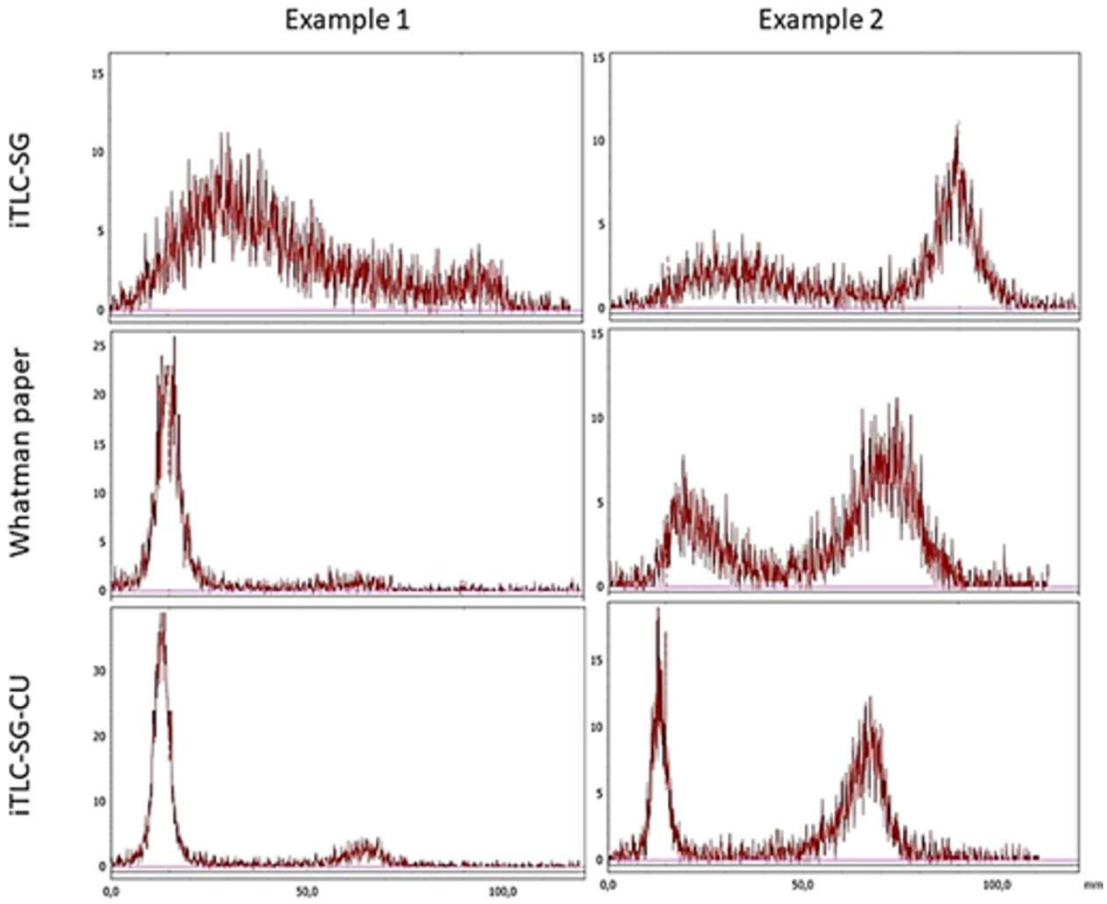


Figure 1. TLC scans of [^{61}Cu]Cu-NOTA-octreotide spotted on: top, iTLC-SG; middle, Whatman paper; bottom extractant-impregnated iTLC-SG. Example 1 notes elevated levels of unlabelled ^{61}Cu, while example 2 notes comparable levels of labelled to unlabelled ^{61}Cu.

[6] J. Svedjehed et al.: "New extractant impregnated iTLC-SG paper facilitates improved TLC analysis for Cu radiolabelled peptides", poster presented at TERACHEM 2022, 14 – 17 September 2022, Bressanone (Italy). Poster available on https://www.triskem-international.com/

Extension to New Supports:
impregnated filtering membranes

For impregnated membranes under developments (e.g. TK227):

- **See Inés Llopart presentation on applications**

On-going studies on extractive membranes :

- TK221 and TK200 for Actinides rapid screening
- new application using TK200 for Po in Ac-225
- TK101 for Ra and TK102 for Sr
- …

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Conclusions

The development of TK227 Resin has shown

- ✓ Y loading at 0,1-0,2M HNO_3 is possible
- ✓ Y is well separated from other elements
- ✓ Y needs to be carried
- ➢ It needs further testing (e.g.@ 0,5M-1M HNO_3) to avoid Y breakthrough in 1L up to 50L
- ➢ On-going methods developments for preconcentration/purification on resins and alternative supports

Thank you for your attention!

support@triskem.fr

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