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## ● NEW PRODUCTS

## ● EDITO

# CL RESIN

As 2010 is almost over, the whole team of TriKem International wishes you all the best for 2011.

The CL Resin is one of the first products originating from TrisKem R&D efforts. It is used for the separation of chloride and iodide and is based on an extraction system that is selective for platinum group metals, gold and silver in acidic conditions. For  $Cl^-/I^-$  separation  $H_2SO_4$  is the best suited acid (table 1). The selectivity for halides is introduced by loading the resin with silver.

Since the beginning TrisKem has given high importance to its R&D department. Therefore, we are happy to inform you of the addition of 2 new resins resulting from our R&D program in the course of the first 2011 semester to our catalogue. One is dedicated to the separation and isolation of Cl-36 and I-129 and is named CL Resin. The second resin is dedicated to the separation and isolation of Cu, especially Cu-64 and Cu-67, and is named CU Resin.

*Table 1 :  $D_w$  values CL Resin of selected cations in sulphuric acid (data taken from [1]).*

Analyte	Extraction condition	$D_w, mL.g^{-1}$
Ag	1M $H_2SO_4$	650000
Ag	Sulfuric acid, pH 3	600000
Ag	Sulfuric acid, pH 5	350000
Cd	1M $H_2SO_4$	<1
Ce	1M $H_2SO_4$	4
Co	1M $H_2SO_4$	<1
Cu	1M $H_2SO_4$	<1
Fe	1M $H_2SO_4$	<1
Mn	1M $H_2SO_4$	<1
Ni	1M $H_2SO_4$	<1
Pd	1M $H_2SO_4$	87000
Zn	1M $H_2SO_4$	25

In this issue, we are presenting some characteristics of the CL Resin. You'll find more information on the technical sheets and associated methods available for download on our website [www.triskem-international.com](http://www.triskem-international.com).

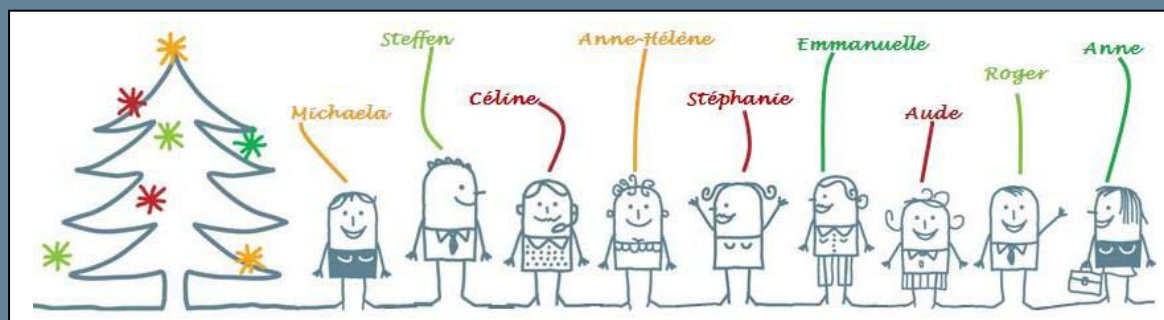
The loading of the resin with silver cations allows good selectivity for anions, especially halides, forming sparingly or insoluble Ag complexes.  $D_w$  values for chloride and iodide on the silver loaded CL Resin in 1M  $H_2SO_4$  were determined to be 1600 and 1980 respectively. Both are thus well retained under those conditions. The CL Resin used for the  $D_w$  experiments was loaded with 20 mg  $Ag^+$  per g of CL Resin prior to...

We'll be glad to meet you at the different conferences and meetings of 2011 (see page 4 for details of events).

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Aude Bombard  
Product Manager

Please note that TrisKem will be closed from 24th December to the 2nd January included.



We wish you a merry Christmas, a good start into the New Year and a happy and successful year 2011!

TRISKEM INFOS

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## News about Cartridges

In the course of the first semester of 2011, we'll start providing you with 2mL cartridges of our production.

The cartridge Luer-Lock connections remain the same (to ensure compatibility with the vacuum box and its associated accessories), as well as cartridge geometry and raw materials used for production. However the design is changing (see Photos 1 and 2 below): the main difference is the new colour-coded ring allowing for easier identification and hermetic closure of the cartridge.



**Photo 1:** new 2mL cartridge design for TRU resin with blue color code ring.



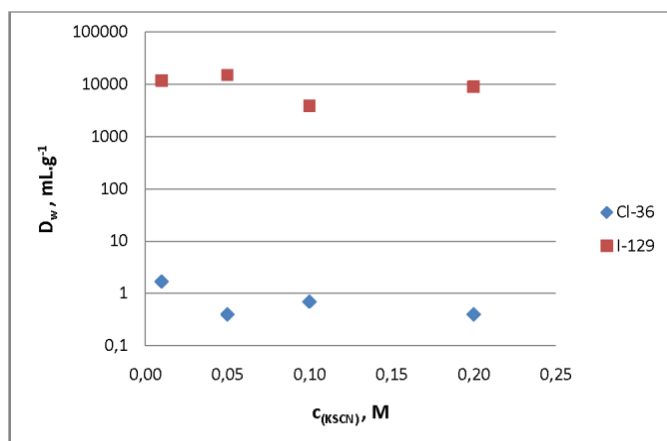
**Photo 2:** new 2mL cartridge design for DGA,N resin with grey color code ring.

**For more information, do not hesitate to contact us and/or to download technical data sheet from our website [www.triskem-international.com](http://www.triskem-international.com)**

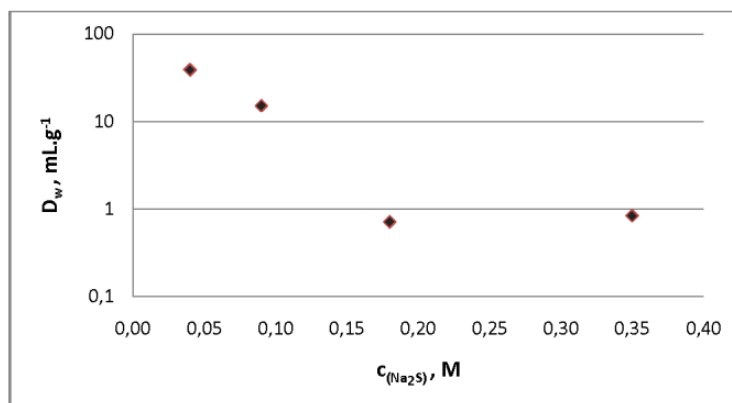


...the extraction experiments which corresponds to a typical working capacity. The capacities for chloride and iodide of the silver loaded resin under these conditions are about 25 mg iodide per g of  $\text{Ag}^+$  loaded CL Resin and about 6.5 mg chloride per g of  $\text{Ag}^+$  loaded CL Resin. Higher capacities for halides can be obtained by increasing the silver load of the CL Resin.

In order to evaluate best suited conditions for the separation of chloride and iodide  $D_w$  values of chloride and iodide were determined on silver loaded CL Resin in varying  $\text{SCN}^-$  and  $\text{S}^{2-}$  concentrations: Fig. 1 and 2 show the obtained results.



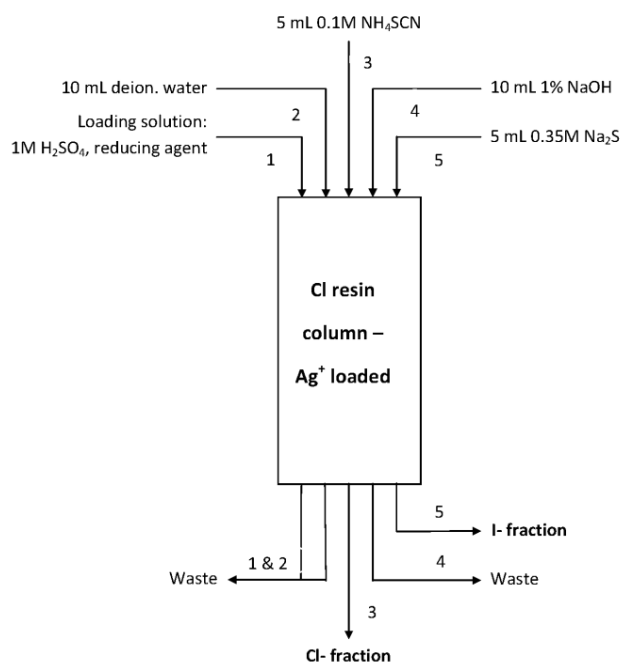
**Figure 1 :**  $D_w$  of Cl and I on  $\text{Ag}^+$  loaded CL Resin at pH 7 and varying  $\text{SCN}^-$  concentrations [1].



**Figure 2:**  $D_w$  of I on  $\text{Ag}^+$  loaded CL Resin at pH 7 and varying  $\text{Na}_2\text{S}$  concentrations [1].

Chloride can be easily eluted from the resin using  $\text{SCN}^-$  solutions whereas iodide remains fixed. Iodide can then be eluted from the resin using an elevated concentration solution of  $\text{S}^{2-}$ . Based on this information, a method for the separation of chloride and iodide was developed and optimized by Zulauf et al. [1]; fig. 3 schematically shows this method. In order to assure that both chlorine and iodine are present as chloride and iodide, the sample might be loaded from a sulphuric acid solution containing 0.1M  $\text{SnSO}_4$  as reducing agent. This is especially important in case of chlorine since e.g. chlorate is not fixed on the resin, whereas iodate is extracted, as could be expected from silver salt solubility data.

The sample is preferably loaded onto the silver loaded CL Resin from 1M  $\text{H}_2\text{SO}_4$  (slightly acidic or even neutral conditions are also acceptable). During a first rinse (deionised water) matrix elements and potential interferences are removed from the



**Figure 3:** Scheme of optimized Cl / I separation method [1].

column. Chloride is then eluted in a small volume of  $\text{NH}_4\text{SCN}$  or  $\text{NaSCN}$ .

During method optimization it was shown that rinsing the column with a dilute alkaline solution before iodide elution lead to a strong increase of the iodide yield. Therefore, the CL Resin column is rinsed with 1%  $\text{NaOH}$  before iodide is finally eluted in a small volume of a  $\text{Na}_2\text{S}$  solution.

The small elution volumes used for elution allow for direct measurement of the obtained fractions by LSC. Some LSC cocktails reduce traces of  $\text{Ag}^+$  co-eluted from the column resulting in 'blackened' LSC samples; it is thus advisable to test your cocktail before use. ProSafe HC (produce by Meridian Biotechnologies Ltd.) was found to be a suitable cocktail.

In order to obtain additional information on the purity of the chloride and iodide fractions decontamination factors ( $D_f$ ) were determined by applying the optimized method to several multi-element solutions and solutions of radioactive standards, table 2 summarizes the results.

Mean chemical yields of the separation were found to be 97.0% ( $\pm 2.5\%$ ,  $k=1$ ,  $N=30$ ) for chloride and 91.7% ( $\pm 10.1\%$ ,  $k=1$ ,  $N=30$ ) for iodide [1]. These yields were then applied to the analysis of spiked tap water samples. Table 3 compares obtained and spiked activities, both agree very well. Mokili et al. also successfully applied this method to spiked effluent samples [2].

In addition to aqueous samples Zulauf et al. [3] also tested the separation method on spiked soil, concrete and membrane filter samples.

Analyte	$D_f$ in Cl <sup>-</sup> fraction	$D_f$ in I <sup>-</sup> fraction
Ba	>1000	>600
Cd	>6900	>7700
Co	>170	>1500
Cr	>29	>430
Cs	>200	>6200
Cu	>210	>190
Mn	>210	>370
Ni	>170	>320
Pb	>300	>720
Rb	>16	>2300
Sr	>180	>17000
U	>1900	>200
Zn	>32	>11
<sup>60</sup> Co	>320	>320
<sup>137</sup> Cs	>150	>150
<sup>90</sup> Sr/ <sup>90</sup> Y	>180	>160
<sup>36</sup> Cl	NA	>160
<sup>129</sup> I	>420	NA

**Table 2:** Decontamination factors  $D_f$  of various elements in chloride and iodide fractions.

	determined activities		added activities		Bias / %	$E_n$
	A(I-129) / Bq	$U_{A(I-129)}$ / Bq	A(I-129) / Bq	$U_{A(I-129)}$ / Bq		
I-129						
Repl. 1	8,24	1,98	8,22	1,31	0,3%	0,01
Repl. 2	8,17	1,97	8,22	1,31	-0,5%	0,02
Repl. 3	7,86	1,89	8,22	1,31	-4,4%	0,16
Cl-36						
Repl. 1	8,97	1,05	9,44	0,94	-5,1%	0,34
Repl. 2	9,11	1,06	9,44	0,94	-3,5%	0,23
Repl. 3	9,12	1,06	9,44	0,94	-3,5%	0,23

**Table 3:** Comparison determined vs. added activities, spiked tap water samples, 3 replicates, bias and  $E_n$ ,  $k=2$ .

In a first step, the leaching yields for chloride and iodide for the three matrices were determined to be > 90%. Then fresh sets of spiked samples were prepared by spiking the respective matrix with defined amounts of Cl-36 and I-129, followed by a drying step. The samples were then leached and separated as described before. Overall determined and reference activities agree very well (see technical data sheet CL Resin).





## IN BRIEF

You can find former issues of our newsletter on our website. If you would like to stop receiving the TrisKem Infos, please advise us by either contacting us at [contact@triskem.fr](mailto:contact@triskem.fr) or by phone to +33 (0)2 99 05 00 09.

## AGENDA

° Winter Plasma Zaragoza 2011 – 30/01-04/02/2011, Zaragoza (Spain)  
<http://www.winterplasmazaragoza2011.es/>

° International Symposium on isotopes in Hydrology, Marine Ecosystems, and Climate Changes Studies – 27/03-01/04/2011, MONACO  
<http://www-pub.iaea.org/mtcd/meetings/Announcements.asp?ConfID=38297>

° PROCORAD 2011 – 22-24/06/2011, Rhodes (Greece)  
[http://www.procorad.org/fr/avenir\\_reunion/](http://www.procorad.org/fr/avenir_reunion/)

° 19th International Symposium on Radiopharmaceutical Sciences – 28/08-02/09/2011, Amsterdam (Netherlands)  
<http://www.isrs2011.org/>

° 7th International Conference on Isotopes – 4-8/09/2011, Moscow (Russia)  
<http://www.isotop.ru/en/events/information-for-participants/information-for-participants-2/>

° 3rd International Nuclear Chemistry Congress – 18-23/09/2011, Palermo (Italy)  
<http://3rdincc.mi.infn.it/>

You will find the update on our participations to conferences on our website

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Warwick et al. [4] developed a method for the analysis of decommissioning samples (e.g. spent resin) based on the thermal decomposition of the sample using a 'Pyrolyser' furnace. Volatilized chlorine species are transported by a stream of moistened air into a bubbler containing a 6 mM Na<sub>2</sub>CO<sub>3</sub> solution where they are retained. The bubbler solution is then directly loaded onto silver loaded CL Resin. The authors found that, since the sample is not loaded from a highly acidic sample solution, an additional rinsing step is necessary ('modified wash') in order to improve C-14 decontamination. Table 4 shows the decontamination factors obtained using the developed and optimized method.

The overall yield of the method (pyrolyser step and column separation) was about 86% which allowed obtaining a detection limit of 20 mBq.g<sup>-1</sup> ( $m_{\text{sample}}=1\text{g}$ ,  $\epsilon_{\text{LSC}}=98\%$ ,  $t_{\text{counting}}=180\text{ min}$ ). The method was successfully applied to the <sup>36</sup>Cl determination in a spent exchange resin.

	<sup>36</sup> Cl fraction	<sup>129</sup> I fraction
<sup>3</sup> HTO	> 500	> 2000
<sup>14</sup> C <sub>2</sub>	7	5000
<sup>14</sup> C modified wash	700	
<sup>35</sup> S modified wash	1500	1000
<sup>36</sup> Cl		> 2000
<sup>129</sup> I	1300	

Table 4 : Decontamination factors of Pyrolyser / CL Resin based method [4].

## Bibliography

- [1] A. Zulauf, S. Happel, M. B. Mokili, A. Bombard, H. Jungclas: Characterization of an extraction chromatographic resin for the separation and determination of <sup>36</sup>Cl and <sup>129</sup>I. *J. Radioanal Nucl Chem*, 286(2), 539-546 (DOI: 10.1007/s10967-010-0772-5).
- [2] A. Zulauf, S. Happel, M. B. Mokili, P. Warwick, A. Bombard, H. Jungclas: Determination of Cl-36 and I-129 by LSC after separation on an extraction chromatographic resin. Presentation at the LSC 2010 conference, 07/09/2010, Paris (France), available online: <http://www.nucleide.org/LSC2010/presentations/O-56.pdf>.
- [3] A. Zulauf, S. Happel: Characterisation of a Cl- and I- selective resin. Presentation at the TrisKem International users group meeting, 14/09/2010, Chester (UK); available online: [http://www.triskem-international.com/iso\\_album/ugm\\_chester\\_06\\_zulauf\\_happel\\_cl\\_resin.pdf](http://www.triskem-international.com/iso_album/ugm_chester_06_zulauf_happel_cl_resin.pdf).
- [4] P E Warwick, A Zulauf, S Happel, I W Croudace: Determination of <sup>36</sup>Cl in decommissioning samples using a Pyrolyser furnace and extraction chromatographic separations. Presentation at the 11th ERA Symposium, 16/09/2010, Chester (UK); available online: [http://www.triskem-international.com/iso\\_album/11\\_era\\_chester\\_warwick\\_determination\\_of\\_36cl\\_in\\_decommissioning\\_samples\\_using\\_a\\_pyrolyser.pdf](http://www.triskem-international.com/iso_album/11_era_chester_warwick_determination_of_36cl_in_decommissioning_samples_using_a_pyrolyser.pdf).

## Liquid Scintillation Cocktails Capacities

We do provide for a year now different liquid scintillation cocktails (see TKI N°3). Experiments have been performed by James Thomson (Meridian Biotechnologies Ltd.) to test the maximum accepted volume of standard aqueous solutions used for radionuclides stripping from our extraction chromatographic resins by 4 of the main scintillation cocktails used.

Capacities @20° C	Gold Star	Gold Star LT2	ProSafe+	ProSafe HC+
0.1M citric acid	10.0 ml	0.75 - 10.0 ml	3.25 ml	7.5 ml
0.1M ammonium citrate	10.0 ml	1.25 - 7.5 ml	2.75 ml	5.5 ml
0.1M EDTA	10.0 ml	2.25 - 4.5 ml	3.40 ml	5.25 ml
0.05M HNO <sub>3</sub>	10.0 ml	10.0 ml	3.75 ml	10.0 ml
0.35M HNO <sub>3</sub>	10.0 ml	10.0 ml	5.75 ml	10.0 ml
2M HNO <sub>3</sub>	4.25 ml	2.75 ml	2.75 ml	4.5 ml
3M HNO <sub>3</sub>	3.25 ml	2.25 ml	2.25 ml	4.25 ml
4M HNO <sub>3</sub>	2.75 ml	2.25 ml	2.50 ml	4.0 ml

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