

# RadChem *Info*

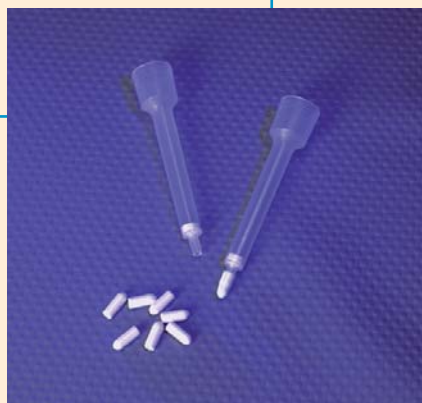
In Brief

Lead Resin

Agenda

## EICHROM ENVIRONMENT

N°11 - January 2008



## Editorial

All the Eichrom Environment team wishes you a happy new year and all our best wishes for 2008.

Year 2007 has been the year of change regarding our separation from Eichrom Technologies Inc.-US and the creation of Eichrom Environment with the implementation of the production unit in Rennes. We see 2008 as a year of services and products innovation to continuously meet your exigences and needs.

In this RadChem number we present the characteristics and properties of Lead Resin (Pb Resin).

We are inviting you to our next Users' Group Meeting next May in Madrid, Spain. You may already register for the meeting, please refer to instructions page 4.

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Eichrom Environment

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## EICHROM ENVIRONMENT

# Resins

## Pb resin

Lead Resin is composed of crown-ether (fig 1) diluted in isodecanol and coated on an inert support. Its composition is similar to the one of the Sr Resin with the exception of the diluent which is n-octanol in Sr Resin. Isodecanol, having a longer carbonated chain than n-octanol, is used to facilitate lead elution from the resin.

Lead affinity ( $k'_{Pb}$ ) for Pb Resin over the range  $10E-02$  to  $10E+01$  M  $HNO_3$  is varying from ~20 to 800 (fig 2). In HCl media,  $k'_{Pb}$  for Pb Resin varies from 20 to 100. The maximum retention is observed between  $5.10E-02$  to 2 M HCl. Below and above these HCl concentrations, Pb affinity for the resin is rapidly decreasing.

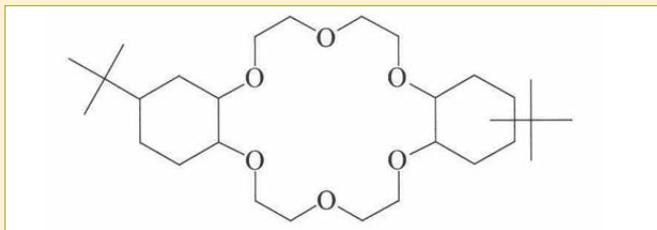


Figure 1 : 4,4'(5')-bis(tert-butylcyclohexano)-18-crown-6.

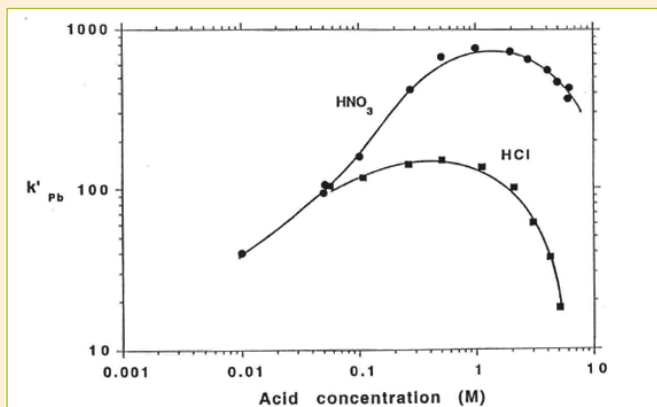


Figure 2 : Pb behaviour on Pb Resin in  $HNO_3$  and HCl media<sup>(1)</sup>.

Resin density	0.37 g/mL of resin
Vm (mobile phase) in mL/mL of resin	0,65 (Cs-137 elution)
Vs (stationary phase)	0,16 mL /mL of resin
Experimental capacity	29 mg Pb/ g of dry resin

Table 1 : Pb resin data<sup>(1)</sup>.

Monovalent and divalent ions show similar retention/elution profiles as those observed on Sr Resin (fig 3 and 4). Sodium and calcium do not interfere with the lead uptake on the Pb resin for concentrations from  $10E-02$  to 1 M (fig 5). The presence of potassium up to 1M still allows a lead uptake with  $k'_{Pb} \sim 80$ . In 0.1M  $HNO_3$ , most ions are not retained on the resin. Only Pd is partly fixed. This latter is completely eluted with 40

free column volume rinsing.

Elution of lead can be performed with different solutions as 0.1M ammonium citrate, 0.1M ammonium oxalate or 0.1M glycine<sup>(1)</sup>.

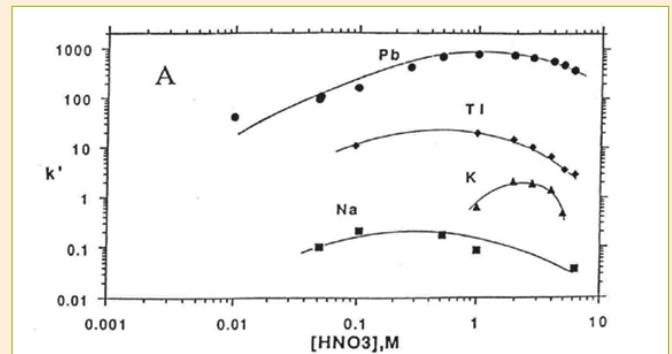


Figure 3 : Affinity of monovalent ions for the Pb Resin in  $HNO_3$  media<sup>(1)</sup>.

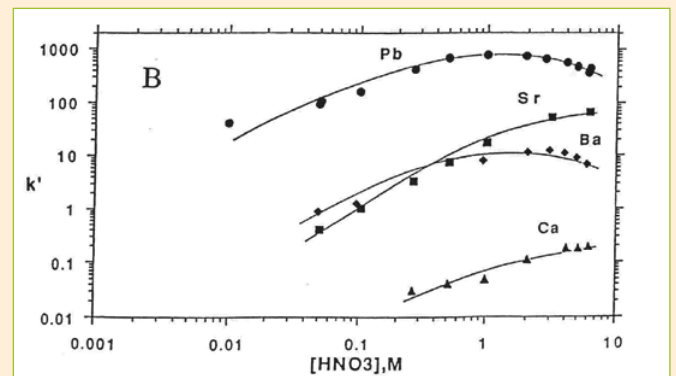


Figure 4 : Affinity of divalent ions for the Pb Resin in  $HNO_3$  media<sup>(1)</sup>.

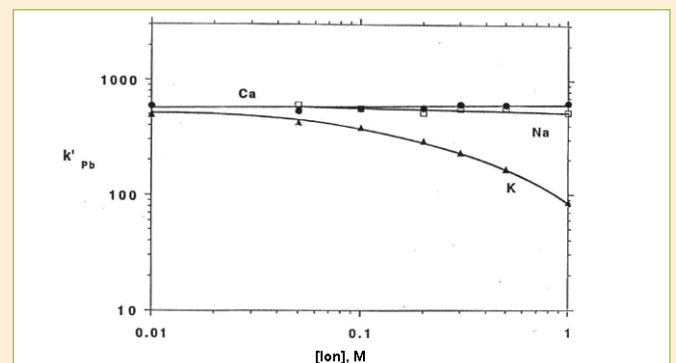


Figure 5 : Behaviour of interfering ions on Pb Resin in  $HNO_3$  media<sup>(1)</sup>.

Pb Resin is used in different types of applications. The main one of these applications is the separation and measurement of Pb-210 in water samples. Due to the radiotoxicity of both Pb-210 and its daughter Po-210, the World Health Organisation is recommending the determination of the activities of Pb-210 and Po-210 in drinking water samples in order to obtain more realistic values of the Total Indicative Dose (TID). Some countries (e.g. France) are already following this recommendation.

Pb-210, a fortiori Po-210, are naturally present in the environment as progeny of U-238 series (fig 6).

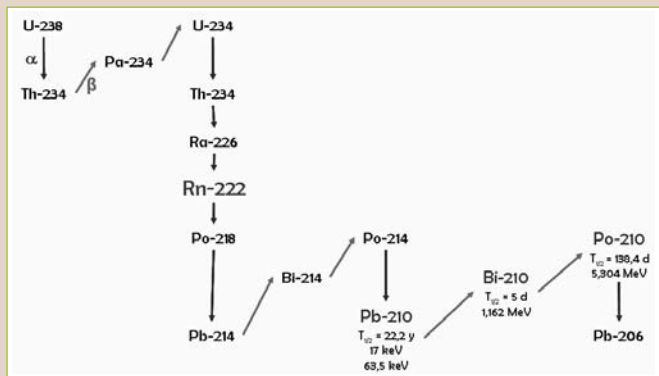


Figure 6 : U-238 series<sup>(2)</sup>.

A method for Po-210/Pb-210 separation was presented in November 2006 at the Users' group meeting in Bratislava. Results are presented figure 7. The sample is dissolved in 2M HCl and is loaded onto a resin preconditioned with 2M HCl. Under these conditions Bi-210, other than Pb and Po, does not fix on the resin. Then Po-210 can be eluted with 0.1M HNO<sub>3</sub>. Pb-210 can finally be eluted with a 0.1M citric acid solution<sup>(2)</sup>.

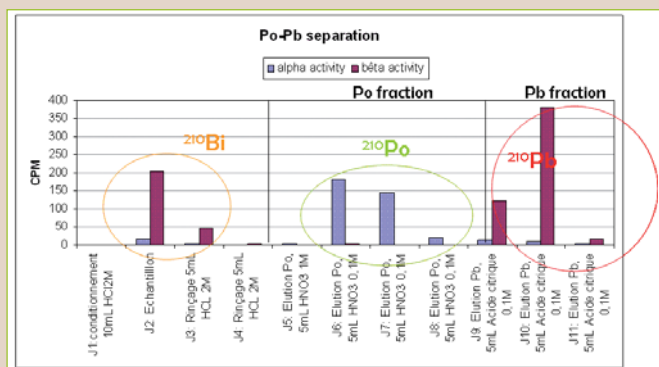


Figure 7 : Separation scheme of Po-210/Pb-210<sup>(3)</sup>.

Eichrom procedures OTW01 et OTS01, respectively describe the separation of lead in water and soil samples, and its measurement by gas proportional counting of the Pb-210 daughter Bi-210. In case of soil samples, Pb is in a first step solubilised departing from a 2g sample. The sample size is 100 mL 0.1M for soil

samples and 500mL up to 1L 0.1M HNO<sub>3</sub> for water samples. The separation procedure is then independent of the initial nature of the sample :

1/ lead is preconcentrated either by ferric hydroxide coprecipitation or on cationic exchange resin. In this later case, Pb is eluted from the cationic resin with 8M HNO<sub>3</sub> solution.

2/ if the ferric hydroxide coprecipitation was chosen, then the residue is centrifugated and dissolved in 1M HNO<sub>3</sub>. In the case of the cationic resin use, the eluate is evaporated then dissolved in 1M HNO<sub>3</sub>.

3/ the sample is loaded on Pb resin in 1M HNO<sub>3</sub> media. At this concentration, bismuth-210, daughter of lead-210 is not fixed on the resin. Polonium-210 is eluted with 0.1M HNO<sub>3</sub>. Lead can then be eluted with deionised water.

4/ the measurement of lead-210 is done by gas proportional counting of bismuth-210 after its ingrowth to equilibrium.

Pb Resin is also used for the measurement of lead in iron and steel by flow injection coupled to flame atomic absorption spectrometry<sup>(3)</sup>. Pb resin is preconditioned in 1M HNO<sub>3</sub> for 3 minutes with a 3mL/min flow rate. The sample (about 30 mL) of iron or steel in 1M HNO<sub>3</sub> media is introduced in flow injection device. The resin is rinsed for 2 minutes with 1M HNO<sub>3</sub> at 3mL/min. Lead is finally eluted with a solution of 0.1M oxalate ammonium during 3 minutes with a flow rate of 4mL/min, in the opposite direction to the sample flow. The lead solution is directly introduced in the nebuliser of the spectrophotometer. The results obtained by the authors analysing certified reference materials agreed well with the corresponding reference values.

### Bibliography

- (1) Horwitz E.P., Dietz M.L., Rhoads S., Felinto C., Gale N.H., Houghton J.; Analytica Chimica Acta, Vol.292, p263-273 (1994) ; Eichrom reference HP194.
- (2) Happel S., Le Berre M., Johanson L., Bombard A. ; Validation of an improved method for the separation and measurement of Pb-210 and Po-210; Users' Group Meeting, Bratislava - Slovakia, 10th November 2006.
- (3) Seki T., Takigawa H., Hirano Y, Ishibashi Y. ; Analytical Sciences, Vol.16, p513-516 (2000) ; Eichrom reference ST001.

Do not hesitate to contact us for more information.

# In Brief

This year, the Users' Group Meeting will be held in Madrid - Spain, on the 9th of May. Meeting location and a list of surrounding hotels will be provided later on.

Meeting language will be English, with an English-Spanish translator. You might already register to the meeting by sending back by e-mail ([contact@e-environment.fr](mailto:contact@e-environment.fr)) or by fax (+33 (0)2 99 05 07 27) the form page 4.

# Agenda

## REGISTRATION TO THE USERS' GROUP MEETING IN MADRID - SPAIN

9th May 2008

(Fill in capital letters)

Last Name : .....

First Name : .....

Company/Organisation : .....

Address : .....

.....

ZIP Code : ..... City : ..... Country : .....

Phone : ..... Fax : ..... e-mail : .....

4

I would like to do a presentation entitled :

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I'll be accompanied by :

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Signature ..... Date .....

For the lunch, please indicate if necessary your wishes (vegetarian, allergy...) : .....

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### IMPORTANT :

Deadline for registration and sending of abstract : 20 March 2008

Sending of the presentation : 21 April 2008