RadChem Info

In Brief

Nickel Resin

Agenda

N°10 • September 2007

EICHROM ENVIRONMENT

Editorial

We are proud to announce that we obtained our ISO 9001:2000 certification last July for the design, production, sale and trade of laboratory consumables including:

> chromatographic extraction and ion-exchange resins for the analysis of radioactive and non-radioactive elements;
> related equipment.

Acopy of the certificate can be obtained either by:

> following the link www.afaq.org, under the identification number : 295881184752.
> contacting us either at contact@e-environment.fr or by phone +33 (0)2 99 05 00 09.

September is also for us the starting point for the sales of SR, TEVA, TRU and UTEVA Resins produced in France.

The technical part of this issue is dedicated to the properties and characteristics of the Nickel Resin.

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Product Manager

Eichrom Environment

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Nickel Resin

The Nickel Resin is dedicated to the separation of nickel from other elements. The Nickel Resin consists in dimethylglyoxime extractant coated on an inert suppot. Unlike our other extraction chromatographic resins, it is based on an on-column precipitation of nickel with dimethylglyoxime (abbreviated DMG, figure 1) at pH 8-9.

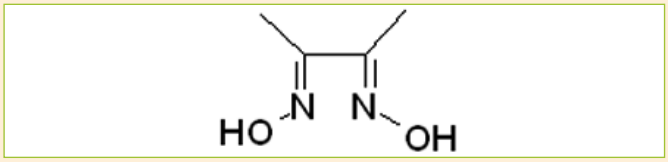


Figure 1 : DMG.

During the precipitation reaction two molecules of dimethylglyoxime react with Ni^{2+} [1] as follows :

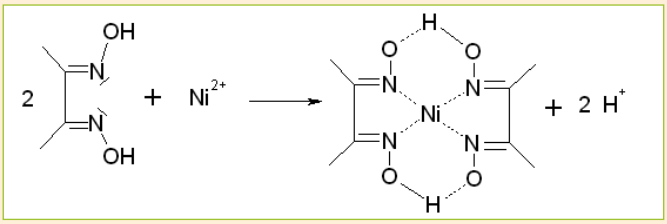


Figure 2 : Precipitation reaction of nickel cation with 2 molecules of diméthylglyoxime, $Ni(DMG)_2$.

Resin density	0,25 g/mL of resin
Experimental capacity	4 mg Ni/ g of dry resin

Table 1 : Data about Ni Resin [2].

The nickel-dimethylglyoxime complex, $Ni(DMG)_2$, is insoluble in water and difficult to "destroy". Its stability constant, log K_1 is 14,6 [3].

The general outline of nickel separation procedure is as follows [4].The sample is traced with a nickel carrier solution (Ni-carrier must not exceed 3 mg Ni/g of dry resin or 2mg Ni/2mL Ni columns). The sample is then evaporated to near dryness and converted to chloride by repeated evaporations with concentrated HCl. The obtained residue is dissolved in 1M HCl. A solution of 0.2M ammonium citrate is added to the sample and the overall solution is adjusted to pH 8-9 with concentrated ammonium hydroxyde. The resin is also preconditioned with a 0.2M ammonium citrate solution adjusted to pH 8-9. When the sample is loaded on the resin, a red precipitate appears. The resin is rinse with the 0.2M ammonium citrate adjusted at pH 8-9. The $Ni(DMG)_2$ complex is dissolved with a 3M HNO_3 solution, allowing its elution from the resin [2,4]. After $Ni(DMG)_2$ elution, the resin colour is changed back to white and the eluted solution is colourless. At this stage, most of the DMG initially coated onto the inert support has been eluted as the $Ni(DMG)_2$ complex, making the resin not suitable for re-use.

Ni-63 and Ni-59 can be measured directly by liquid scintillation or X-spectrometry respectively [2]. The $Ni(DMG)_2$ complex eluted can also be reprecipitated and then filtered. Ni-59 activity is measured by counting the precipitate on the filter [4]. For Ni-63 determination, the filter containing the precipitate is oxidized in a muffle furnace at 500°C. The nickel oxide residue, NiO, can then be dissolved in a minimum volume of *aqua regia*. The sample is converted to chloride form by successive evaporations to dryness of concentrated HCl. HCl is preferred to HNO_3 as $NiCl_2$ is not volatile compared to $Ni(NO_3)_2.6H_2O$, which ebullition point is 137°C. The nickel residue is then dissolved in a 0.1M HCl solution for its counting in liquid scintillation [4].

The preconditioning and conditioning of the Nickel Resin can also be done in tartrate solution. The presence of the citrate or tartrate ions prevents any co-precipitation of metals that would precipitate as insoluble hydroxides under the given pH values. High quantities of oxidizing agents can interfere by preventing the precipitation of nickel by *formation of soluble oxidised complex of $Ni(DMG)_2$* [1].

If the samples contain high quantity of iron, it is necessary to remove the iron before the precipitation of the nickel [4]. If the quantity of residue during the sample evaporation step is negligible or small, the iron removal can be performed on TRU Resin :

1/ Dissolve residue in 8M HNO_3
2/ Add Fe carrier (≤ 1.5mg Fe/g dry TRU Resin)
3/ Load solution on TRU resin
4/ Rinse TRU resin with 8M HNO_3

However, if the quantity of residue is large, the iron removal has to be performed on an anion exchange resin, e.g. 1x8 type :

1/ Dissolve residue in 12M HCl
2/ Add Fe carrier (≤ 1.5mg Fe/g resin)
3/ Load solution on the anion exchange resin
4/ Rinse the resin with 12M HCl

In both cases, the iron stays on the resin while the nickel is eluted. The eluates are repeatedly evaporated with concentrated HCl for the preparation of the sample before the loading onto the Nickel Resin.

A study on the decontamination factors of different radionuclides with respect to Ni was performed by D.F. Cahill and L. M. Peedin [5]. The results are given in table 2.

The same authors compared their standard Ni separation method with the method using Eichrom Nickel Resin, for the analysis of Ni-59/63. Their standard method was as follows :

1/ Addition of Ni carrier to the sample
2/ Acidification/evaporation
3/ Precipitation with $Fe(OH)_3$
4/ Centrifugation/filtration
5/ Adjustment of pH to 8-9
6/ Precipitation of $Ni(DMG)_2$
7/ Centrifugation/rinsing of the precipitate
8/ Dissolution of the precipitate
9/ 2nd precipitation of $Ni(DMG)_2$
10/ Centrifugation/rinsing of the precipitate

11/ Dissolution of $Ni(DMG)_2$
12/ Destruction of $Ni(DMG)_2$/ Conversion to NiO
13/ Dissolution and liquid scintillation counting

Comparisons between the two methods were performed for different types of samples analyzed in their laboratories. The results obtained are presented in table 3. They show that the two methods give similar results. However, in the case of the Nickel Resin separation, the manipulation time is 1 to 1,5 days against 2 to 2,5 days for their standard method.

Radionuclides	Decontamination factors
Cr-51	3,5E+02
Mn-54	8E+03
Fe-55	4E+02
Co-58	1E+03
Co-60	1,1E+03
Nb-95	1,3E+02
Cs-134	2,8E+03
Cs-137	3E+03

Table 2 : *Decontamination factors obtained on Nickel resin for different radionuclides compared to nickel [5].*

The Nickel Resin exists in 100-150 µm particle size. The 2 mL preconditioned columns of Ni Resin are delivered in a 0.15M ammonium citrate solution buffered at pH 9 with ammonium hydroxide.

The Eichrom procedure describing the nickel separation is NIW01. The associated bibliography is available on the following link : http://www.eichrom.com/ (follow « Radiochemistry » then « Bibliography »).

Sample type	Stadard method	Nickel resin
TL/HS tank	8,510E-02	8,810E-02
Lab waste tank	9,842E-02	9,213E-02
WECT tank	1,595E-01	1,543E-01
Ni-63 spike	1,876E+01	1,980E+01
Ni-59 spike	3,700E+02	3,396E+02
DAW Smears	1,547E+03	1,713E+03
Radwaste filter	2,738E+04	2,882E+04
RWCU Resin	6,771E+04	7,585E+04

Table 3 : *Comparison of Ni-63 results obtained for two nickel separation methods [5]. Activities are in Bq/unit.*

Bibliography

(1) Kirby L. J. ; *The Radiochemistry of Nickel*, November 1961, NAS-NS 3051.
(2) Rajkovich S., Cahill D., Peedin L., Wheland S., Lardy M., Eichrom Cincinnati Users' Seminar, OH - USA (1996); Référence Eichrom RS196.
(3) Furia T. E., *CRC Handbook of Food Additives; Chapter 6 - Sequestrants in Foods*, 2nd ed. (1972).
(4) Strebin R., Orr R., Kaye J., Fadeff S., *Nickel-59 and Nickel-63 Determination in Aqueous Samples*. Pacific Northwest Laboratory, Richland, WA - DOE Methods Compendium RP300; Référence Eichrom RP300.
(5) Cahill D. F., Peedin L. M., *A comparison of Standard and Extraction Chromatography Methods of Analysis for Nickel-59/63 and Tritium*; 41st Annual Conference on Bioassay, Analytical and Environmental Chemistry, Eichrom workshop, Boston, MA - USA, (1995); Référence Eichrom CD195.

Do not hesitate to contact us for more details

In Brief

2mL column rack project

Your experience feedback interests us

Our column rack (AC-103) was originally developed to be used with 50ml centrifuge tubes as reservoirs. Recently we got several remarks that it could be of interest to change its design. Therefore we would be interested in getting your experience feedback on the routine use of this reference in your labs. We would be very grateful if you would take a few minutes in order to fill the following questionnaire and/or send any comments either by fax (+33 2 99 05 07 27) or by e-mail to shappel@e-environment.fr. We thank you in advance for your answers.

1/ Are you satisfied with the current rack ❒ yes ❒ no
If not, why ? .....
2/ Is the height of the rack ❒ well suited ❒ too high ❒ too low
3/ Is the space for the collector containers (beakers, tubes, vials,...)
for the eluates under the rack well adapted ? ❒ yes ❒ no
4/ Do you use all the available positions at the same time? ❒ yes ❒ no
If not, why ? ❒ lower number of samples ❒ lack of space below the rack
❒ other : .....
5/ Do you think the labelling of each position could be useful? ❒ yes ❒ no

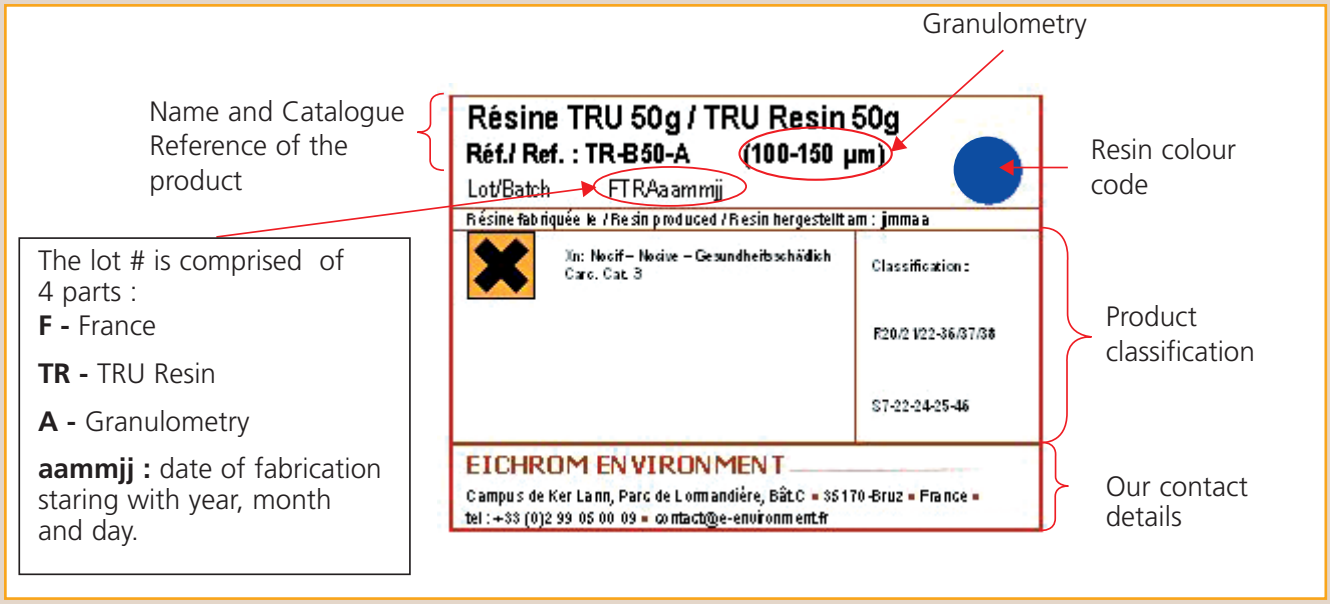
Agenda

Users' Group Meeting

The next Users' Group Meeting is foreseen for the first semester of 2008. We'll let you know in the next issues of the RadChem, the date and location of the meeting.

Labelling

The French production is signalled by a new label. An example of label for a bottle of 50g TRU Resin is given below :



The certificates of analysis are delivered with all of our bottles of resins.

2008 Radiochemistry conferences / congress

> 2nd INCC - 2nd International Nuclear Chemistry Congress, Cancun - Mexico, 13-18 April 2008
http://depa.fquim.unam.mx/2ndincc/

> LSC2008, Davos - Switzerland, 25-30 May 2008
http://lsc2008.web.psi.ch/

> PROCORAD - Teddington - UK, 18 - 20 June 2008
http://www.procorad.org/fr/avenir_reunion/

> NRC7 - Seventh International Conference on Nuclear and Radiochemistry, Budapest - Hungary, 24-29 August *2008http://www.nrc7.mke.org.hu/*

You also may address your requests and demands to contact@e-environment.fr

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