EIChroM ideas

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Since our last newsletter I am pleased to report on several exciting developments at Eichrom. The company continues to grow thanks in no small part to strong sales of our analytical radiochemistry products. Our focus remains to work closely with customers to develop new applications for existing products as well as to develop new products for our users' changing needs.

In the process business we have successfully completed another pilot of our iron control system in a copper mine in Australia. Additionally, a number of new product lines for metals separations are under development. I am also pleased to announce a merger between Eichrom and **MICRA Scientific, Inc.**, a Northbrook, Illinois-based manufacturer of novel HPLC products.

MICRA was founded in 1994 and has commercialized a line of HPLC columns, based on highly uniform, non-porous silica (NPS®) microspheres. The patented NPS manufacturing process produces microspheres in the 1.5 to 3 micron size range that are uniform, reproducible and metal-free. This technology allows for faster, more efficient separations. MICRA also manufactures the SynChropak® line of porous silica HPLC columns.

In addition to HPLC, the NPS technology also has applicability in growing life sciences markets for use in cell separation applications and point of care diagnostics. Several new products are in development that combine NPS with chemistry from the Eichrom side of the company. These products show promise in a wide range of analytical applications.

We are excited about this merger because it expands Eichrom's analytical product offerings into a broader customer base. It also promotes a synergy of ideas between our scientific research staffs. As our research and production facilities are combined at the beginning of 1999, we expect to see the continued development of new products which take advantage of the technology bases of both companies. Many of these new products will form the basis of Eichrom's continued growth well into the next century.

— Bruce R. Grotefend
President

Eichrom announces a merger with MICRA Scientific, Inc. "We are excited about this merger because it expands Eichrom's analytical product offerings… [and] promotes a synergy of ideas between our scientific research staffs."

EIChroM INDUSTRIES, INC.

MICRA Scientific, Inc.

Tc-99... Faster Than Ever!

With Eichrom's New TEVA Disc

The use of Eichrom's TEVA Resin in the analysis of Tc-99 has become widespread.

In a recent issue of *Eichrom Ideas* (Volume 4 Number 3, October 1997) we published a review of the TEVA Resin, listing eight literature references on this application. The TEVA Resin method for Tc-99 offers significant advantages over other methods including ion exchange and solvent extraction. Still the elapsed time of analysis can be quite lengthy due to the time required to pass large sample volumes through the TEVA Resin column. Given the competitive demands for fast sample turnaround time and increased analyst productivity, we sought to take full advantage of TEVA Resin's kinetics while maintaining the same separation power.

Earlier this year, Eichrom introduced a more efficient format for the TEVA Resin. The TEVA Disc is a 47 mm glass fiber filter loaded with 0.5 gram silica-based TEVA Resin. Aqueous samples can be passed through this disc at much faster flow rates: up to 30 mL/min under gravity flow and up to 200 mL/min with vacuum assistance. This can reduce the sample processing time to **as low as five minutes for a one liter sample!**

Aqueous samples are treated with hydrogen peroxide to ensure oxidation to pertechnetate. Most aqueous samples can be loaded onto the disc without further treatment. After the sample and a 50 mL 0.01M nitric acid rinse have passed through the TEVA Disc, the disc itself is easily transferred to a scintillation counting vial. Add 10 mL UltimaGold LLT cocktail, cap the vial and vortex until the disc disintegrates, forming a colorless, translucent gel.

Tests conducted in our development laboratory show that up to six liters of simulated ground water matrix adjusted to pH 2 can be passed through the TEVA Disc with >85% Tc-99 recovery. These results are shown in Table 1.

Table 1. Percent of Tc-99 Recoveries in Large Volume Simulated Hanford Ground Water Samples

Volume	%Tc-99 Recovery	Detection Limit pCi/L
1 Liter	95.9	1.77
2 Liter	96.2	0.89
3 Liter	86.8	0.59
4 Liter	89.9	0.44
5 Liter	85.4	0.35
6 Liter	87.0	0.30

Table 2. LSC Parameters

Counter:	Packard Tri Carb 2550 TR/AB
Beta window:	26–350 keV
tSIE:	Typically 450–550
Counting efficiency:	90%
Count time:	30 minutes

Detection limits as low as 0.3 pCi/L can be achieved using a 30 minute count time. LSC operating parameters are provided in Table 2.

In addition to data developed internally, a current user of a TBP-based Tc-99 method asked to be one of the first to give the new TEVA Disc a try. The TBP method, as well as other anion exchange and solvent extraction methods, produces a lot of waste, in many cases mixed hazardous/radioactive waste. This customer had expressed interest in switching to TEVA Resin in the past but found the TBP method, despite its generation of mixed waste, to be faster.

However, with the opportunity to get rid of their mixed waste generation and actually speed up their Tc-99 method, the analysts in this laboratory were eager to evaluate our product. They validated the TEVA Disc on

well characterized ground water samples containing from 40 to 40,000 pCi/L Tc-99. Across six sample batches, the results obtained with the TEVA Disc compared extremely well with the results previously obtained using the TBP method. This data is provided in Table 3.

Additionally, three of these samples were tested to determine matrix spike recovery. Results from all three were in the mid-ninety percent range.

Sample	Number of Replicates	TEVA pCi/L	TBP pCi/L	Relative % Difference
1	4	42434	42506	-0.2
2	2	43.5	41.8	+4.1
3	4	419.8	406	+3.4
4	1	51	48	+6.1
5	1	78	80	-2.5
6	1	56	50	+11.3

Table 3. Comparison of TBP vs. TEVA Disc Methods

The customer is pleased with the results and expects reduced production time from four hours to two hours for a batch of twenty samples from their current TBP method. It is also expected that 100 mL TBP and 5 L of H_2SO_4 waste will be eliminated per batch of twenty samples.

Eichrom's TEVA Resin has been a standard in the analysis of Tc-99 for some time now. The introduction of a new disc format of this product allows our customers to increase the speed of analysis and simplify their methods for Tc-99 even further. We are very happy to make this new product introduction and we look forward to bringing you ongoing innovations in radiochemical methods.

Actin

Through the years, one analytical challenge we have faced is the analysis of actinides in larger soil samples.

In Europe and the Far East, soil samples as large as 100 grams are often required to achieve detection limits for actinides specified by regulatory agencies. The matrix components present in such large sample sizes can interfere significantly with the proper functioning of any chemical separation technique. For this reason the chemical separation methods for actinides in large soil samples have always been long and cumbersome, and the results obtained using them subject to significant uncertainty.

Now, however, a new Actinide Recovery Method has been developed by the Savannah River Site Central Laboratory to preconcentrate actinides in very large soil samples prior to isolation of individual actinides by a variety of separations schemes. This method has been applied to samples as large as 50 grams. It has also been used following both leaching (nitric acid/HCl) and NaOH fusion for soil digestion.

In the method, a spiked, digested soil sample is loaded onto a conditioned 2.8–3.0 mL column of Eichrom's Diphonix® Resin (Part No. DP-B500-M-H). The column is rinsed with a mixture of 0.5M HF/0.5M HCl followed by water. The resin is removed from the column and transferred to a microwave digestion vessel.

Concentrated nitric acid (10 mL) is added and the resin is digested in the microwave oven through a three-stage process, each involving a gradual ramp up to the indicated temperature:

1) **at a temperature of 190°C for 20 minutes,**
2) **at 220°C for 35 minutes, and**
3) **at 210°C for 15 minutes with addition of 30% hydrogen peroxide.**

After each stage the vessels are removed from the microwave and allowed to cool, and nitric acid fumes are vented. After the digestion, a clear, colorless solution results.

The Diphonix Resin digestate is wet ashed (H_2O_2 and HNO_3) and the residue is re-dissolved in an appropriate solution for one of a number of chemical separation schemes utilizing combinations of Eichrom's TEVA Resin, UTEVA Resin and TRU Resin. [Horwitz, et al., *Analytica Chimica Acta,* 310 (1995) 63–78.] Chemical recoveries of individual actinide spikes were measured.

ide Recovery Method

The recoveries for U, Th, and Pu are shown in Tables 1 through 3, respectively. Uranium and thorium recoveries are nearly quantitative, even to large sample sizes. In some experiments with plutonium spikes, digested soil samples were passed through two tandem Diphonix columns to measure the breakthrough of plutonium through the first column. In cases where this was carried out, the recoveries for the first and second columns are both shown in Table 3. The data shows a significant amount of plutonium break-through for samples larger than 10 grams.

Americium retention on Diphonix Resin is affected by aluminum present in the soil matrix. Hydrofluoric acid (HF) can be added to the Diphonix Resin load solution to complex the aluminum and prevent its interference with Am uptake by the column. Table 4 shows the effect of varying amounts of HF on Americium recoveries from 10 gram soil samples.

Nearly quantitative neptunium spike recoveries were measured for 20 gram soil samples.

Table 1. Uranium Spike Recoveries

Sample Size	Average Chemical Recovery
5 gram	91%
50 gram	90%
60 gram	93%

Table 2. Thorium Spike Recoveries

Sample Size	Average Chemical Recovery
20 gram	96%
30 gram	86%
50 gram	87%

Table 3. Plutonium Spike Recoveries

Sample Size	Average Chemical Recovery 1st Cartridge	2nd Cartridge	Total
10 gram	104%	NA	104%
15 gram	69%	22%	91%
20 gram	58%	20%	78%

Table 4. Americium Recoveries: Optimization of HF 10 gram Sample

HF added	Average Chemical Recovery
3.0 mmol	60%
3.7 mmol	88%

Intercomparison samples from the EML QAP program were analyzed using this method. 10 gram aliquots were leached (nitric acid/HCl) or fused (NaOH) and run through the method as described above. Results for Pu and Am are summarized in Table 5 and Table 6 below.

Table 5. Results of Leached EML/QAP Samples

EML QAP	Aver. Chem. Recovery	Pu-238 (dpm/g)	Pu-239 (dpm/g)
9509 cert. value	NA	1.05	0.310
Results (n=3)	76%	1.06 (+1%)	0.313 (+1%)
9403 cert. value	NA	0.672	0.216
Results (n=3)	89%	0.653 (-2.8%)	0.211 (-2.4%)

Table 6. Results of Fused EML/QAP Samples

QAP-EML	Pu Chem. Recovery	Pu-238 (dpm/g)	Pu-239 (dpm/g)	Am Chem. Recovery	Am-241 (dpm/g)
9609 value	NA	0.080	1.47	NA	0.81
Results (n=3)	80%	0.061 (-10%)	1.42 (+11%)	80%	0.90 (+11%)

Development of the method is ongoing. In particular, the effect of different soil types and larger sample sizes is being investigated. If you are interested in more detailed information about the new Actinide Recovery Method, please contact your Eichrom representative.

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Frequently Asked Questions

1 Why do Ni Resin pre-packed columns have a six-month shelf life?

Recent Eichrom studies indicate that the flow rate of pre-packed Ni Resin columns decrease with time. After six months some columns flow at an unacceptably slow rate. Because of this we recommend that Ni Resin columns be used within six months of packing. All columns shipped after September 1, 1998, are labeled: "Best if used before: <date>."

In order to maximize the shelf life of packed columns in the customer's hands, we will now pack columns to order rather than stocking inventory. You can expect a 2–3 week delivery time for pre-packed columns.

2 Does bulk Ni Resin in bottles have the same six-month shelf life?

No, this flow rate decrease has not been observed for bulk resin stored dry.

3 Are chemical recoveries affected by the decreasing flow rates?

No, extensive analysis of a number of lots of Ni Resin columns showed that recoveries are unaffected regardless of flow rate observed.

4 What flow rates can I expect?

As with all resins, flow rates vary with the matrix and geometry of column packing. Flow rates with Eichrom's Ni Resin also vary with the amount of elemental nickel present. With clean solutions containing 1.7 mg nickel in the load, Eichrom typically observes flow rates in pre-packed columns as listed in the table below.

Typical Flow Rates Using Eichrom's Nickel Resin Column

Fraction	Flow rate (mL/min.)
Pre-rinse	0.23 – 0.33
Load	0.19 – 0.32
Rinse	0.15 – 0.19
Strip	0.38 – 0.50

5 Can I still use columns which I've had for more than six months?

Yes, you can still use columns which are more than six months old. Flow rates may be improved by inverting the columns overnight prior to use. This loosens the resin bed and often increases flow rates observed.

Did You Know...

Nuclear power plant samples high in activation and fission products can be troublesome for Tc-99 analysis using TEVA Resin. One customer reports that by loading Tc-99 onto the TEVA Resin from 2M nitric acid, she minimizes interference from Cs and Co. She warns, however, that at the high nitric acid concentration, any tetravalent actinides in the sample may be retained by the resin. If so, a pre-treatment to remove the actinides, such as an iron hydroxide precipitation, should be performed.

In This Issue...

- Faster Tc-99 Using TEVA Disc
- Actinide Recovery Method
- Eichrom Announces Merger with MICRA

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Announcements

USERS' MEETINGS

COMING UP IN 1998

UK Users' Meeting:
Manchester, Friday, December 4
(English language only)

French Users' Meeting:
Paris, Monday, December 7
(Simultaneous French/English translation provided)

German Users' Meeting:
Kassel, Thursday, December 10
(German language only)

Eichrom Workshop at the Bioassay Conference:
Albuquerque, Monday, November 16, 8:00 am
(English language only)