Slow Flow, Eichrom Fights Back With New Quality Controls

Gravity flow rate of our columns has always been an important performance consideration to our customers. A number of factors affect the actual flow rate that a user experiences. Among these are column dimension, solution type, temperature and volume of solution in the funnel over the column (head pressure). The particle size of the resin, however, remains one of the more important factors. We have known for some time that the commercially available resins suitable for use in manufacturing our extraction chromatographic materials are not uniform in particle size distribution and often contain unacceptable levels of very small particles (or fines). Either of these factors can contribute to slow flow or lot-to-lot variability in flow rates. For this reason, we imposed specific particle size requirements on our supplier of chromatographic supports to ensure that fines are removed and that the distribution of particles in each lot is consistent.

The particle sizing necessary to achieve our flow requirements was recently transferred to a firm outside the resin manufacturer. At that time we conducted a thorough evaluation of their process to ensure it resulted in material of adequate flow rate. Unfortunately, we did not institute a program of QC checks on each batch we received from this supplier. Early in 2000, we began to receive some customer comments about flow rate with our A grade products. We confirmed the flow rate problem through in-house studies and measured the particle size distribution of a number of older and newer lots of resins and polymer raw materials. It became apparent to us that the material we were receiving back from our toll processor was significantly smaller in particle size than what was specified and had been received during the evaluation process.

Through this particle size analysis of various lots of the chromatographic support, we identified one that was acceptable in terms of size distribution and flow rate. All subsequent production was shifted to this lot of material and slow flowing lots of finished product were removed from inventory. Additionally, a new toll processor was chosen to remove the fines from our chromatographic supports and, more importantly, a quality control procedure was written and incorporated into our ISO-9002 program to monitor the flow rates of all sized material received from the supplier.

The resolution of this issue is easily explained, but has required about six months to accomplish. Approximately 80% of inventory on our shelf was found to be slow flowing and needed to be replaced. Although we were able to make new resin to put back on the shelf, it took some time to restore our inventories to their normal level. Some of you may have noticed slower than usual delivery times in the late spring and early summer of this year as our production staff worked to restock our inventory with acceptable product.

The challenge of ensuring consistent flow rates is now behind us. We have implemented an incoming raw material flow rate evaluation program. This QC procedure ensures all incoming A grade chromatographic supports will have a gravity flow rate of 0.70 +/- 0.10 ml/min when packed in our columns, under specified test conditions. This program is in place and doing its job. We have passed several batches of chromatographic support and have already caught some slow flowing material, which was reprocessed to remove fines before subsequently being accepted.

Users of our products will still see flow rates that vary depending on the actual usage conditions (solution type, temperature, column dimension, etc.) We are confident, however, that with the measures we have recently taken, our A grade products will now be of more consistent particle size distribution. Please contact us if you have any questions about this issue or would like a copy of our new QC procedure for flow rates.

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ISO 9002 Reg. FA3024 UL

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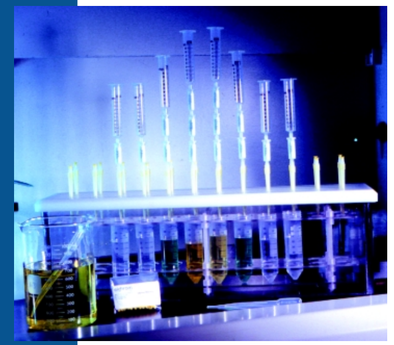
New Technology For Faster Analysis!

The Vacuum Box System



Eichrom continues its tradition of being your "time saver" by introducing its Vacuum Box System and extraction cartridge line. By employing vacuum in our already advanced separation methods, analysis time can easily be cut in half over traditional gravity flow Eichrom columns. Customer comments and research in our Radlab indicate the same consistently high yields for actinides and strontium as with our conventional columns. Chromatography is improved with the new cartridges that use our smaller 'S' grade resin beads. Another advantage of the cartridge design is the ability to stack two together for tandem separations. Many of our actinide procedures use a common load solution that passes through two resins, for example UTEVA and TRU Resin in our ACW03 method. Using the Luer connections, the cartridges can easily be stacked during sample loading and initial rinse steps and then be separated for final analyte stripping. For sample loading to our cartridges, a standard syringe barrel is recommended, although many reservoirs with a male Luer connection can be used. The cartridges are connected to the vacuum box by two disposable tips. The white inner tip provides an excellent seal with the cartridge and the yellow outer tip provides support. As with our columns, each cartridge is labeled with an identifying lot number. In addition to Sr, Ln, TEVA, TRU and UTEVA cartridges currently in stock, our other resins are available by special order in the cartridge format. Though inventory is limited on the Vacuum Box at this introductory stage, we are now accepting orders. Please inquire for an up to date delivery schedule when placing your order.

The Vacuum Box
Clear polycarbonate construction
24 sample capacity
Sample collection via inner 50 mL C tube rack
Inexpensive cartridge to box connection

The Extraction Cartridge
Standard 2 ml resin volume
50-100µ beads
Available in Sr, Ln, TEVA, TRU and UTEVA
Stackable for multiple analyte separation

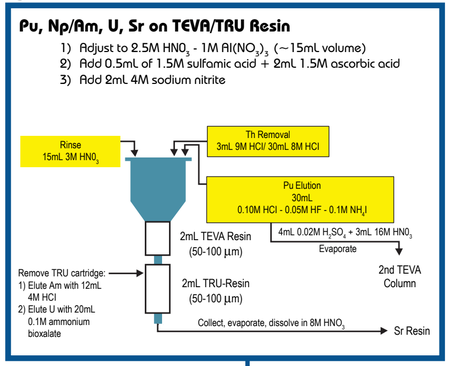
Eichrom and Westinghouse Savannah River Site Strive for Faster Bioassay Methods

The push for faster bioassay sample turnaround started long before Eichrom Technologies was formed, over ten years ago. Back in the 1980's, after a urine sample registered an extremely high activity many months after collection at Argonne National Laboratory, an existing committee consisting of Phil Horwitz, Don Nelson and their colleagues initiated research into faster bioassay methods. These new methods revolved around the creation of the same extraction chromatographic materials available from Eichrom today. This newsletter will chronicle some of the more recent developments in bioassay methods using extraction chromatography.

Over the years, the Savannah River Site has welcomed the benefits of Eichrom's extraction chromatographic materials. In what might be termed a true partnership, together we have harnessed this technology to improve their radiochemical methods used for environmental, process, waste and bioassay analyses. For urine, Savannah River Site has been using a variety of methods to tackle different combinations of U, Pu, Am, Np and Sr. Since 1996, when Pu, Am and Sr were called for together, a sequential procedure employing TRU Resin followed by Sr Resin gravity flow columns has been used. When Pu, Np and U analyses were called for, however, anion exchange resin has been used. These anion exchange resin based methods tended to generate a large volume of acid waste and at times would produce inconsistent recoveries resulting in reruns in order to hit required detection limits.

A research team headed by Sherrod Maxwell, III and David Fauth was tasked with a complete review of what is arguably the largest Bioassay Program in the United States, if not the world. David has many years of experience in Savannah River's Bioassay program while Sherrod gained much of his experience perfecting vacuum-assisted actinide methods for the high level waste and process activities at the site. Together they began combining their expertise, along with a healthy dose of input from Eichrom, to develop a new, faster, and more rugged series of bioassay methods.

Figure 1



Sherrod presented an overview of these new methods at our workshop series this year in the U.S. The method employs a single two-staged column consisting of TEVA Resin and TRU Resin to separate Pu and Np in one fraction along with U and Am in separate fractions. Strontium is later separated on Sr Resin. All column separations are carried out using vacuum-assisted flow. As with all of the talks given at our Users' Group Workshops, the complete presentations are available on Eichrom's web site, www.eichrom.com. Sherrod and Dave's work is also being published in Radioactivity and Radiochemistry, A Journal of Applied Radioactivity Measurements, Vol. 11, No. 3 and is entitled Rapid Column Extraction Methods for Urine.

The new method is designed to handle any combination of actinides and strontium and uses a 500 mL sample aliquot. After heating the acidified urine sample for 1.5 hours, the actinides and strontium are precipitated with calcium phosphate. The precipitate is wet ashed to destroy any remaining organic matter. Once the dried sample turns completely white it is ready to be taken up in the column load solution. First the sample is brought up in 3 M nitric acid-1.25 M aluminum nitrate. Oxidation states are then fixed by making the solution 0.05 M sulfamic acid and 0.2 M ascorbic acid before making the solution 0.4 M sodium nitrite. The method is unique in that iron (II) is not added to the valence adjustment chemistry. At this point the Pu and Np are in the (IV) state. Since urine does not typically contain significant amounts of iron, this load solution is ideal for Am separation on TRU Resin as well as Pu separation on the TEVA Resin. A complete flow sheet is provided in Figures 1 and 2.

Based on worker duties and work location, the sample will be analyzed for any combination of actinides and strontium. When a sample needs to be analyzed for Pu and/or Np only the TEVA Resin is needed. For Pu and U samples, when a U-232 tracer is used, a second smaller (1 mL) TEVA column is used to remove any remaining traces of Th from the Pu fraction. As can be seen in Table 1, the spike recoveries for the actinides were all excellent.

Strontium 90 results were also excellent. On samples ranging from 5.4 to 188 pCi/L average bias was -4.8%. The US DOELAP criteria requires bias to fall between -25% and +50%. Average bias for a variety of actinide isotopes fell well within this range.

Sherrod and Dave's team have also developed a fecal method and applied it to <10 grams of ashed sample for Pu and Am. This method provides for dissolution of Pu oxides using a nitric acid /HF solution.

Figure 2

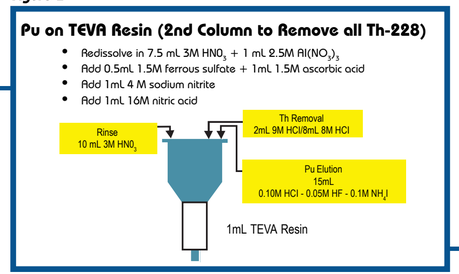
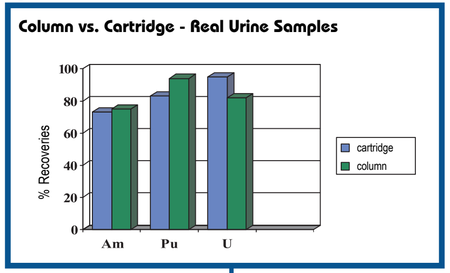


Figure 3



The totally dissolved actinides are then loaded onto Eichrom's Diphonix Resin®. Diphonix Resin is unique in its ability to load actinides out of solutions containing HF. The actinides are recovered by digesting the Diphonix Resin and the sample is then processed similarly to the urine samples. Because iron is likely to be present in the fecal samples, the load solution is collected after passing through the TEVA Resin, evaporated and then brought back up in an appropriate load solution for Am separation on the TRU Resin.The results of this method have been excellent. They are shown in Table 2.

Eichrom's development team has also been busy adding to your arsenal of fast bioassay methods. Under the leadership of Anil Thakkar, Manager of Technical Support, Eichrom now offers an Americium, Plutonium and Uranium in Urine procedure (ACU02) which has been evaluated in our Rad-lab using both UTEVA and TRU columns and cartridges. The complete method is available on our web site or from Eichrom.

The procedure uses a similar calcium phosphate precipitation to bring down the actinides as discussed earlier. The ashed sample is brought up in our traditional load solution of 3 M nitric acid and 1 M aluminum nitrate. Pu is reduced to (III) with ferrous sulfamate and ascorbic acid. The sample is loaded onto UTEVA Resin where the remaining tetravalent actinides and uranium hold. Pu, Am and the rest of the matrix are washed through the UTEVA Resin in the load and nitric acid rinse solution. With the cartridge/vacuum box system, the UTEVA and TRU Resin cartridges are linked together so that no separate step is needed to load the Pu and Am onto the TRU Resin. This is done automatically with the common load and first rinse solutions. After these, the cartridges are easily separated. Further purification of uranium on the UTEVA cartridge is accomplished through an HCl/oxalic acid rinse to remove Th and Np. Finally, uranium is stripped with 0.01 M HCl. Back on the TRU Resin, Pu is reoxidized to (IV) with sodium nitrite and the Am is stripped with 9 M and 4 M HCl. Finally, Pu is stripped with 0.01 M ammonium bioxalate. A cerium fluoride precipitation is carried out and the samples are counted via alpha spectrometry.

Typically, the gravity flow column separation on UTEVA and TRU Resins is fast by any standard, at 5.5 hours. (Before TRU Resin, an americium sample alone took 3 days.) But with the cartridge/vacuum box system now available from Eichrom, the time is cut to only 2.5 hours for all three analytes.

The results comparing the cartridges with our traditional columns is shown in figure 3. In all cases the results show tracer yields of over 70%. The results using the cartridge approach also showed excellent correlation with the French Bioassay Intercomparison Study (PROCORAD).

We realize that many of you have contributed to the body of knowledge that has brought us to this current state. Thank you for your contributions as we all strive to deliver very fast and accurate bioassay results to our fellow radiation workers.

Table 1

Average Actinide Tracer Recoveries using Figures 1 and 2 Flow

Pu-236	Np-237	Am-243	U-232
98.4(+/-7.9@1σ)%	94.8(+/-7.6@1σ)%	96.9(+/-10.6@1σ)%	84.7(+/-12.9@1σ)%

Table 2

Assay of DOE-RESL Fecal Standards

	Pu-239 (picocuries)	% Bias	Am-241 (picocuries)	%Bias
DOE-RESL values	2.18		2.76	
SR-194 #1	1.860		2.405	
SR-194 #2	1.841		2.608	
Avg.	1.850	-15.1%	2.507	-9.2%
SR-234 #1	2.062		2.488	
SR-234 #2	2.139		2.335	
Avg.	2.101	-1.4%	2.411	-10.7%