# Eichrom Technologies' Product Catalog for 2014

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### **Eichrom Technologies LLC**

Eichrom was founded in 1990 to commercialize chemical separation technology developed in the Chemistry Division at Argonne National Laboratory. Since that time, the company has been at the forefront of new product development and commercialization in the areas of radiochemistry, geochemistry, hazardous metals analysis and environmental screening for dioxins and related compounds.

Eichrom's line of extraction chromatographic resins represents a revolutionary new approach to the chemical separation of actinides and fission products from various sample matrices. These products have become the standard technology in the field of radiochemistry with methods used by accredited laboratories and regulatory agencies worldwide (e.g., the US Environmental Protection Agency and the International Atomic Energy Agency.)

Eichrom's proprietary chemical technology also includes a family of phosphonic acid-based ion exchange resins that have been used in the fields of hydrometallurgy, bottled water purification and nuclear waste treatment.

In 2004, we launched a water testing laboratory in Rennes, France that applies Eichrom's unique position in radiochemical analysis to the testing of drinking and bottled water in the European Union.

In 2008 Eichrom acquired Nuclear Power Outfitters our radiation shielding arm. With over 30 years of experience in radiation protection NPO is the industry leader in developing shielding solutions to meet ALARA objectives. NPO manufactures a variety of shielding products including lead blankets, supplied air respirators, tungsten flexible shielding and custom engineered shielding solutions for the nuclear Navy, Department of Energy and nuclear power generators. Check out the NPO website: www.alarasolutions.com for more information about NPO's radiation shielding solutions.

With offices in suburban Chicago and Rennes, France, Eichrom provides comprehensive customer service and technical support globally.

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### Sample Preparation Products for Radiochemical Analysis:

Eichrom was founded in 1990 to commercialize three novel extraction chromatographic resins developed at Argonne National Laboratory. These resins, currently named Sr Resin, TRU Resin and UTEVA® Resin, offered a significant breakthrough in the analysis of actinides and radioactive strontium. Through the years, the addition of new extraction chromatographic materials, analytical grade ion exchange resins, and high performance filters for alpha spectroscopy source preparation round out a comprehensive product line that serves the sample preparation needs of the radiochemist and geochemist. Our resins are available in labor saving, ready-to-use prepackaged formats that can be used with either gravity flow or vacuum assistance.

In this section of our catalog, you'll find technical information and part numbers for our radiochemical product line, a listing of methods developed at Eichrom and methods published by independent organizations and application notes. We have included a brief overview of the Principles of Extraction Chromatography. The complete versions of the methods, newsletters, and an extensive bibliography of references to Eichrom products and presentations are available through the Eichrom website at www.eichrom.com. In addition, on the web site, some of the bibliography references are also made available where copyright laws allow and links to the publishers are provided otherwise.

Eichrom commercialized its breakthrough chemical separation techniques in the 1990s. Today, accredited laboratories and regulatory agencies worldwide trust Eichrom's line of analytical-chemistry products, all developed through the expertise of our team of dedicated chemists.





### A brief overview of the Principles of Extraction Chromatograph:

Extraction chromatography (EXC) is a technique that is ideally suited to the separation of radionuclides from a wide range of sample types. This technique combines the selectivity of liquid-liquid extraction with the ease of operation of column chromatography. The table at the bottom of the page lists a number of EXC resins that are manufactured by Eichrom.

Surface of Porous Bead



The figure to the left is a simplified depiction of a portion of an extraction chromatographic resin bead showing the three major components of an EXC system: the inert support, the stationary phase, and the mobile phase. The inert support usually consists of porous silica or an organic polymer ranging in size from 50 to  $150\mu$ m in diameter. Much smaller or larger particles have been studied for special applications. Liquid extractants, either single compounds or mixtures, are used as the stationary phase. Diluents can also be used to help solubilize the extractant and to increase the hydrophobicity of the stationary phase. The mobile phase is usually an acid solution, e.g., nitric or hydrochloric acid, although complexants, such as oxalic or hydrofluoric acids, are frequently used to enhance selectivities or the stripping of strongly retained metal ions from columns.

### Achievement of Separation:

To achieve separation in EXC, band spreading must be sufficiently small to avoid early breakthrough and to avoid excessive cross-contamination of the constituents that one is trying to separate. Even if the extractants comprising the stationary phase exhibit very high selectivity for one of the ions, poor column efficiency, as manifested in excessive band spreading, can result in essentially no practical separation. Column efficiency is generally expressed in terms of the height equivalent to a theoretical plate. Plate

height is a complex function of a number of physical and chemical factors. In EXC systems column efficiency is determined primarily by flow phenomena, diffusion in the stationary phase and extraction kinetics. The relative importance of each of these three factors to column efficiency depends on the specific chemical system, the particle size and porosity of the support, the extractant loading and mobile phase velocity, and the operating temperature.

### **Achievement of Selectivity:**

The EXC resins listed below cover a wide range of selectivities and enable one to perform a number of novel highly efficient separations of selected fission products and individual actinides or groups of actinides. Figures detailed with the individual resins show the acid dependencies for the uptake, as measured by k', of selected actinides and non-actinides. Summarize below are the important properties of selected resins.

	Available Particle Sizes (µm)		20-50	, 50-100, 100-150	
	Bed Density	/ (g/mL)	0.33 to 0.39		
	Free Colum	n Volume (% of bed volume)		65 to 69	
Resin		Mmol/ml of bed based on extractant/metal ratio		50% max loading (mg * experimentally dete	
Actinide		0.18 (U)		8.6 (Nd), 21(U), 14 (A	Am),
DGA & DGA-	Branched	0.083 (Eu)		6.3 (Eu)	
Ln, Ln2, Ln3		0.12, 0.091, 0.094 (Eu)		9.1, 6.9, 7.1 (Eu)	
Sr & Pb		0.14 (Sr, Pb) & 0.11 (Pb, Sr)		14 (Sr, Pb) & 0.11 (Pb, Sr) 6 (Sr),14 (Pb) & 11 (Pb),4.7 (Sr)	
TEVA	0.16 (Th, Np, Pu) 15 (Pu)		0.16 (Th, Np, Pu)		
TRU		0.036 (Am)		4.5 (Am)	
UTEVA		0.25 (Th, U)		32 (U)	



### Actinide Resin and Berillium Resin:

Eichrom's Actinide Resin and Beryllium resins are based on the DIPEX® Extractant shown to the right. These resin exhibits an extraordinarily high affinity for the actinide elements (figure lower right.) For acid concentrations less than 1M, the retention of actinides is dramatically higher on Actinide Resin than on TRU Resin. This makes the resin quite useful for the preconcentration of actinides out of large volume aqueous samples. In fact the resin is ideal for monitoring actinides in aqueous discharges. The resin may be contacted in batch mode and then counted directly by liquid scintillation making for a very rapid analysis.

Dr. E. Philip Horwitz, and his group at Argonne National Laboratory, measured the uptake of Am by the Actinide Resin from acidified river water. In their study, the ratio of sample size to amount of Actinide Resin was varied. The results are shown below and suggest that one gram of the resin should be able to extract 99% of Am activity from up to 4L of water and 90% from up to 10L of water.





Additional studies have shown the resin to be an excellent choice for preconcentrating elements typically included in gross alpha measurements in a similar manner. Please refer to Eichrom method ACW11. This approach was employed by Pieter Kwakman of RIVM (Netherlands) in a European gross alpha radioactivity in water intercomparison exercise sponsored by OPRI (France.) His method employed a 4 hour batch contact of 0.3g Actinide Resin and 100 mL water sample. LSC counting for 4 hours resulted in a detection limit of 0.03Bq/L (0.8 pCi/L.)

The results obtained by Kwakman's laboratory were in excellent agreement with the OPRI target value and had significantly less uncertainty than the typical laboratories' results. His results were presented at Eichrom's European Users' Meeting in Paris, May, 2002. The maximum loading capacity of Actinide Resin for various elements is shown in the table below. The resin exhibits a high capacity for actinide elements, in particular hexavalent uranium (50 mg/mL of resin) and tetravalent thorium (30 mg/mL of resin.) The effect of iron on the retention of the actinides is often a concern in radiochemistry. The figures below demonstrate the effect of Fe(III) and Fe(III) on the retention of Am(III) in a variety of HCI solutions. While the effect of ferric ion [Fe(III)] is significant, the effect of ferrous ion is minimal. In samples with suspected high concentrations of iron, the addition of a reducing agent, such as ascorbic acid, will minimize the effect of iron on the retention of americium.

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The effect of fluoride (added as HF) is shown below. The uptake of actinide elements is not significantly affected by HF. This feature allows for the Actinide Resin to be used as a preconcentrator for actinides directly from soil samples digested in HCI/HF solutions. HF used to dissolve silica in soil samples will not significantly interfere with actinide retention on the Actinide Resin if its concentration is held below 1M. Because the retention of actinides on this resin is so high, it is not efficient to strip the actinides from the extractant. Instead it is necessary to dissolve the DIPEX® extractant with isopropanol. See Burnett, (BW197) and Qu, et.al. (QH198) for details.

### **Beryllium Resin**:

At the request of scientists at the Y-12 National Security Complex in Oak Ridge, TN, Eichrom has developed a new method to improve the efficiency of beryllium measurements performed on area surface swipes. The method is compatible with traditional ICP-AES measurements. After surface swipes are digested using standard acids, Eichrom's Beryllium Resin is used t o remove spectrally interferina elements before ICP-AES analysis is performed. This essentially eliminates the need to rerun troublesome samples via ICP-MS. Y-12 has completed their initial testing of this method and presented their findings at the Spring ACS National Meeting in San Diego, CA, March 2005. The data from Eichrom and PGRF and development leading up to this research successful application was also presented at the 2005 ACS meeting and were published in Talanta and Solvent Extraction and Ion Exchange.

Actinide Resin:	Bulk Bottles	Part Number
	25 grams	AC-B25-A
	50 grams	AC-B50-A
Particle Size:	100 grams	AC-B100-A
100-150 µm	200 grams	AC-B200-A
	Columns (2mL)	Part Number
	Package of 50	AC-C50-A
Beryllium Resin:	Cartridges (2mL)	Part Number
Particle Size: 50-100 μm	Package of 50	BE-R50-S



Analyte	Extraction condition	Dw, mL.g-1
Ag	1M H <sub>2</sub> SO <sub>4</sub>	650000
Ag	Sulfuric acid, pH 3	600000
Ag	Sulfuric acid, pH 5	350000
Cd	1M H <sub>2</sub> SO <sub>4</sub>	<1
Ce	1M H <sub>2</sub> SO <sub>4</sub>	4
Со	1M H <sub>2</sub> SO <sub>4</sub>	<1
Cu	1M H <sub>2</sub> SO <sub>4</sub>	<1
Fe	$1M H_2SO_4$	<1
Mn	$1M H_2SO_4$	<1
Ni	1M H <sub>2</sub> SO <sub>4</sub>	<1
Pd	$1M H_2SO_4$	87000
Zn	$1M H_2SO_4$	25

Table 1: DW values CL Resin of selected cations in sulfuric acid.

The loading of the resin with silver cations allows good selectivity for anions, especially halides, forming sparely or insoluble Ag complexes. DW 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 DW experiments was loaded with 20 mg Ag+ per g of CL Resin prior to 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: 16.3 +/- 1.6 mg iodide per 2 mL column (approx. 25 mg iodide per g resin) and 4.3 +/- 0.2 mg chloride per 2 mL column (approx. 6.5 mg chloride per g resin). Higher capacities for halides can be obtained by increasing the silver load of the CL Resin.

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Figure 1 : DW of CI- and I- on Ag+ loaded CL Resin at pH 7 and varying SCN- concentrations



Figure 2: DW of I- on Ag+ loaded CL Resin at pH 7 and varying Na2S concentrations

In order to evaluate best suited conditions for the separation of chloride and iodide DW values of chloride and iodide were determined on silver loaded CL Resin in varying SCN- and S2- concentrations: Fig. 1 and 2 show the obtained results.

Chloride can be easily eluted from the resin using SCN- solutions whereas iodide remains fixed. Iodide can then be eluted from the resin using a high concentration solution of S2-. 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 SnSO4 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.

### loading the resin with silver.

CL Resin:

Table1 shows DW values of selected cations on the CL Resin, for practical reasons sulfuric acid was chosen as extraction medium. It can be seen that the CL Resin has high selectivity for Pd and Ag, whereas the DW values of the other elements tested are low. Ag further shows high DW values over a wide pH range (from 1M  $H_2SO_4$  to dilute sulfuric acid (pH 5)). It will thus be extracted or remain fixed over a wide range of pH values.

The CL Resin 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. The selectivity for halides is introduced by

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The sample is preferably loaded onto the silver loaded CL Resin from 1M  $H_2SO_4$  (slightly acidic or even neutral conditions are also acceptable). During a first rinse (deionised water) matrix elements and potential interferents are removed from the column. Chloride is then eluted in a small volume of NH<sub>4</sub>SCN or 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 accordingly rinsed with 1% NaOH before iodide is finally eluted in a small volume of a Na2S solution (Remark: all work with the Na<sub>2</sub>S solutions should be performed under a fume hood, including the addition of the liquid scintillation cocktail.).

The small elution volumes used for elution allow for direct measurement of the obtained fractions by LSC (Remark: some LSC cocktails reduce traces of Ag<sup>+</sup> co-eluted from the column resulting in "blackened" LSC samples; it is thus advisable to test your cocktail before use).

CL Resin is manufactured in two particle sizes  $(50-100\mu m, and 100-150\mu m)$  and is sold in bottles or ready to use in prepackaged columns (for gravity flow).

Source for all published data: (1) A. Zulauf, S. Happel, M. B. Mokili, A. Bombard, H. Jungclas: Characterization of an extraction chromatographic resin for the separation and determination of 36Cl and 129I. J. Radanal Nucl Chem, 286(2), 539-546 (DOI: 10.1007/s10967-010-0772-5)

Particle Size	Bottles	Part Number
	25 grams	CL-B25-A
	50 grams	CL-B50-A
100 150	100 grams	CL-B100-A
100-150 μm	200 grams	CL-B200-A
	Columns (2mL)	Part Number
	Package of 50	CL-C50-A
Particle Size	Bottles	Part Number
	25 grams	CL-B25-S
50-100 µm	50 grams	CL-B50-S
50-100 μm	100 grams	CL-B100-S
	200 grams	CL-B200-S
Particle Size	Bottles	Part Number
150-300 μm	25 grams	CL-B25-L
	50 grams	CL-B50-L
	100 grams	CL-B100-L
	200 grams	CL-B200-L

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### Cs Resins: (AMP-PAN & KNiFC-PAN)

Both resins, AMP-PAN and KNiF-PAN have been developed by Dr. Šebesta at the Czech Technical University in Prague. Like the MnO2-PAN resin both resins are based on very fine and selective inorganic materials embedded in an organic matrix based on polyacrylnitrile (PAN) in order to improve their mechanical characteristics. The active components are the widely employed ammonium phosphomolybdate (also Ammonium MolybdoPhosphate, AMP) and potassium nickel hexacyanoferrate(II) (also potassium Nickel FerroCyanate, KNiFC).

Both resins are used for the concentration and separation of Cs from various liquid samples.

AMP-PAN resin is based on ammonium phosphomolybdate, an inorganic ion exchanger known for its high selectivity for Cs even at elevated acid concentrations, quick kinetics and radiation stability.

One of the main restraints to the use of AMP is its unfavorable microcrystalline structure accordingly considerable work has been performed to improve its granulometry. Embedding the AMP in an organic matrix allows for controlling particle size, topography, porosity, hydrophilicity and cross-linking of the resin matrix as well as the amount of AMP embedded in the resin.

Šebesta and Štefula showed that embedding the AMP in a PAN matrix only has limited impact on its Cs uptake kinetics, which remain very rapid, and on the Cs capacity of the embedded AMP. It could further be shown that the resin is chemically stable even under relatively harsh conditions such as 1M HNO<sub>3</sub> / 1M NaNO<sub>3</sub> or 1M NaOH / 1M NaNO<sub>3</sub>, even after storing the resin under these conditions for 1 month no visible mechanical damage could be observed, KD values, sorption kinetics and

capacity also remained unchanged. Radiolysis stability of the resin was evaluated in acidic solution by exposing it to doses up to 106 Gy, again no changes in KD or sorption capacity were found.

Desorption of the cesium is only possible using concentrated ammonium salts, 10 bed volumes of 5M NH<sub>4</sub>Cl for example elute 92% of Cs from a column (alternatively  $NH_4NO_3$  might be used) or by destruction of the AMP using strong alkaline solutions (like 5M NaOH).

Its high selectivity for Cs even under harsh chemical conditions and high levels of radioactivity make the AMP-PAN resin a candidate resin for the treatment of radioactive waste solutions. Brewer et al. tested the resin for the removal of Cs-137 from real and simulated acidic high-active liquid radioactive waste containing high amounts of potassium and sodium. Small scale tests were performed using 1.5 mL columns and two feed solutions, one simulated tank waste (spiked with 100 Bq.mL-1 Cs-137) and one actual tank waste. Both solutions were filtered, and pumped through the column using a pump system at a flow rate of 26 – 27 bed volumes per hour, aliquots were taken at regular intervals and analyzed for Cs-137 activity. After the experiment the AMP-PAN columns were eluted using 30 bed volumes of 5M NH<sub>4</sub>NO<sub>3</sub>, reconditioned and the effluents were passed over the column a second time. For the real waste samples a Cs breakthrough of 0.15% was observed after a sample loading volume of 1000 bed volumes during the first loading cycle (corresponding to a Cs decontamination factor greater than 3000) and 0.53% after 830 bed volumes during the 2nd loading cycle. Cs recoveries in the respective eluates were 87%.







AMP-PANs robustness against high salt concentrations also makes it interesting for use in environmental analysis, especially the analysis of Cs-134/7 in sea water.

Pike et al. used AMP-PAN for concentrating and purifying Cs from 20L seawater samples (acidified to pH 1 – 2, stable Cs was added for yield determination by ICP-MS). The authors employed 5 mL columns and worked at a flow rate of 35 mL.min<sub>-1</sub>. After extraction the resin was rinsed from the column using 0.1M HNO<sub>3</sub> and analyzed by gamma spectrometry. Yields were found to be 93.5% +/- 5.0% (n=55). The authors further analyzed an internal lab standard (WHOI) in triplicate and IAEA sea water reference material.

Sample reference	Reference value / Bq.m-3	Obtained value / Bq.M-3
WHOI	3.4 ± 0.4	3.7 ± 0.2
IAEA-443	340—370	369 ± 8

Even larger seawater samples were analyzed by Kamenik et al.. The authors evaluated, in addition to the AMP-PAN resin, also the use of KNiFC-PAN resin, which is based on potassium-nickel hexacyanoferrate(II) embedded in a PAN matrix.

The authors passed 100L of acidified seawater samples (in case of KNiFC-PAN unacidified seawater samples were tested as well) through 25 3mL beds of AMP-PAN or KNiFC-PAN resin at flow rates up to 300 mL.min.1 allowing for processing 100L samples in less than 6h. As described before stable Cs was added to the seawater samples to allow for the determination of the chemical yield e.g. via ICP-MS. After loading resins were rinsed from the columns, dried and measured by gamma spectrometry using a coaxial HPGe detector with 43% rel. efficiency in Petri dish geometry. Chemical yields obtained are summarized .

Resin	Matrix	Chemical yield / %
AMP-PAN	sea water (pH 1)	88.1 ± 3.3
KNiFC-PAN	sea water (pH 1)	92.9 ± 1.1
KNiFC-PAN	sea water	90.2 ± 2.7

Yields are generally high, KNiFC-PAN showing slightly higher yields for the acidified seawater samples than AMP-PAN resin and comparable chemical yields for acidified and non-acidified seawater samples.

Higher flow rates were tested for the processing of non-acidified sea water samples on KNiFC-PAN resin; even at a flow rate of 470 mL.min<sub>-1</sub> Cs yield is still greater than 85%. The authors calculated the minimum detectable activity (MDA) for 100L samples at 50 - 70 h counting time and average chemical yields. For Cs-137 they calculated an MDA of 0.15 Bq.m.<sub>3</sub> and 0.18 Bq.m.<sub>3</sub> for Cs-134.

KNiFC-PAN resin was further used for the determination of Cs isotopes in milk and urine. The chemical yield for the Milk and Urine studies were  $\sim$ 95\$ for Cs on the KNiFC-PAN resin. For the Milk analysis the minimum detectable activity was calculated to be 2 mBq.L-1 for 137Cs in 5L mil sample counted for 1,000 hours.

Cs Resins	AMP-PAN Resin		KNiFC-PAN Resin	
Particle size	Bulk Bottles	Part Number	Bulk Bottles	Part Number
	50 grams	HC-B50-M	50 grams	NC-B50-M
	100 grams	HC-B100-M	100 grams	NC-B100-M
	200 grams	HC-B200-M	200 grams	NC-B200-M
Particle Size:	Columns	Part Number	—	-
100-600 µm	2mL-Package of 200	HC-C200-M	_	_
	5mL-Package of 20	HC5-C20-M	_	_
	8mL-Package of 20	HC8-C20-M	_	-
	10mL-Package of 20	HC10-C20-M	_	_



### Cu Resin:

Cu Resin is used for the selective separation of copper, and more particularly for the separation of Cu-64 and Cu-67 radio-isotopes. The CU Resin has been characterized for different elements, especially for those representative of Zinc or enriched Nickel targets, and for different acidic media.

Cu Resin retains selectively copper from pH 2 to 5 for HCl, HNO<sub>3</sub> and H<sub>2</sub>SO<sub>4</sub> acids over all the tested cations including Ni and Zn. In HCl and H<sub>2</sub>SO<sub>4</sub> media, iron is co-extracted but in a lesser extent: the selectivity Cu/Fe,  $\alpha_{Cu/Fe}$ , decreases as pH increases ( $\alpha_{Cu/Fe}$  is about 1000 at pH 2 and about 70 at pH 5) (Fig 1-3).

Cu uptake is generally high at pH values greater than 2 while it can be easily eluted with mineral acids of concentrations greater than 0.1M.

Main application of the CU resin is the separation of Cu isotopes (e.g. Cu-64, Cu-67) from irradiated targets (typically Zn or Ni targets). Accordingly, besides having high selectivity for Cu over Ni and Zn, the resin also has to be robust against interference by elevated amounts of Zn and Ni. Figures 2a and 2b summarize the influence of Zn or Ni on the Cu extraction. As can be seen even high amounts of both elements interfere only slightly with the Cu uptake in HCl at pH 2, even at 1g of Ni or Zn per g of CU resin employed the DW(Cu) remains greater than 1000.











A method for the separation of Cu from Ni and Zn targets was optimized using simulated target solutions (1). Two types of solutions were tested:

- simulated Ni target solutions containing 10  $\mu g$  each of Cu, Co, Zn and 200 mg of Ni in 5 mL HCl at pH 2,

- simulated Zn target solutions containing 10  $\mu g$  each of Cu, Co, Ni and 200 mg Zn in 5 mL HCl at pH 2)

For both simulated target solutions, Ni, Zn and Co are quantitatively removed from the column during sample solution loading and rinsing whereas Cu is recovered in high yield (>85%) in 1 – 1.5 mL 8M HCl (2,3).

Further optimization of the elution conditions led to the method shown in Figure 3 (2).

The method can be performed at elevated flow rates (e.g. using a vacuum box) without impacting its performance. Loading of the column and Cu elution should be done at approx. 1 mL/min, rinsing of the column can be done at up to 6 mL/min; the final Cu fraction can thus be obtained in 3 - 5 minutes.

Overall decontamination factors obtained are high (Table 1). Cu yields were found to be in the order of 90% in 1 mL of 8M HCl and > 95% in 1.5 mL 8M HCl; Cu is thus recovered near quantitatively in a very small volume.

For certain applications the Cu eluate might be too acidic, in these cases (alternative to evaporation of the Cu fraction and redissolution in a more suitable solvent) it is possible to convert the Cu eluate using a small anion exchange column. Fig. 4 schematically shows such a conversion method using anion exchange resin (AIX resin). In addition to the converting the Cu elute from high acid conditions to low acid or neutral conditions the conversion step also further concentrates the Cu and increases Ni, Zn, Au and organic impurity decontamination.

CU Resin is manufactured in one particle size 100-150  $\mu m$  and is only available in bottles at this time.



Element	Decontamination Factor	
Ni	> 20,000	
An	> 40,000	
Ga	> 10,000	
Со	> 30,000	
Au	> 30,000	



Particle Size	Bottles	Part Number
100-150 µm	25 grams	CU-B25-A
	50 grams	CU-B50-A
	100 grams	CU-B100-A
	200 grams	CU-B200-A

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### DGA Resin & DGA Branched Resin:

Eichrom's DGA Resins are extraction chromatok' graphic materials in which the extractant system N,N,N',N'-tetra-n-octyldiglycolamide is either (DGA Resin) or N,N,N',N'-tetrakis-2ethylhexyldiglycolamide (DGA Resin, Branched). The structure of the DGA molecule is shown above, where the R-groups are straight chain or branched C8 groups. The bed density of both DGA Resins is approximately 0.38 g/mL, with a working capacity of 7.23 mg <sup>90</sup>Sr per mL of resin and 11 mg Yb per mL of resin.

Both forms of DGA Resin have very high affinity for americium under certain conditions and americium is readily eluted under other conditions (See Figures to the right). Diphonix resin shows high affinity towards Am(III), however, its elution is not as easy as with DGA Resin. The TRU Resin also shows a good affinity for Am(III) with a retention factor of 100 for 0.5 to 5 M HNO<sub>3</sub>. At those acid concentrations, however, the retention factor k' Am(III) on either form of DGA Resin is 30 to 500 higher than on TRU!

The properties of the DGA Resins allow for the selective separation of Am(III) without fear of breakthrough due to weak retention. Am(III) is strongly fixed to either DGA Resin in 5 M HNO<sub>3</sub> or HCl and can be eluted with 0.01 M HNO<sub>3</sub> or 0.5 M HCl.

Retention profiles for U, Pu and Th are also presented. The diagrams show that higher Pu/Am separation factors are obtained for the normal version of DGA Resin. Pu(IV) shows strong affinity for the resin with a retention factor k' > 3000 over the entire acid range studied. U and Th affinities towards the DGA Resins depend upon the acid, its concentration and the type of DGA Resin.

Given these data, the combination of TEVA and DGA Resin, would allow one to first separate tetravalent elements on TEVA, leaving U and Am which would be retained on DGA resin. U would be first eluted with 0.5 M HNO<sub>3</sub> followed by Am(III) with 0.5 M HCI.



k' for Th(IV), U(VI), Am(III) and Pu(IV) on TRU, DGA Resin, Normal and DGA Resin, Branched (50-100  $\mu m$ ) versus HNO<sub>3</sub>, 1 hour equilibration time, 22(1)°C.



k' for Th(IV), U(VI), Am(III) and Pu(IV) on TRU Resin, DGA Resin, Normal and DGA Resin, Branched (50-100  $\mu m$ ) versus HCl, 1 hour equilibration time, 22(1)°C.

### DGA Resin & DGA Branched Resin: (cont.)

### **Ra/Ac Separation:**

Figure to the left shows the acid dependency of k' for radium and cerium in nitric acid and hydrochloric acid. In HNO<sub>3</sub> media, Ra shows no real affinity for the resin (1 < k'Ra < 7). However, Ce has k' > 1000 for concentrations higher than 1M. It can be assumed that actinium and cerium will show similar uptake behavior. So Ac should be well retained on the resin for HNO<sub>3</sub> concentrations of 1 - 3 M while Ra is eluted. Ac could then be stripped at low acid concentrations. In HCI media, the separation is efficient at 8M (selectivity  $\alpha$  $(Ce/Ra) > 10^4$ ). Cerium (or actinium) is then stripped at low acid concentrations. (<0.5 M HCI).



### Y/Sr Separation :

Figure below shows the uptake of four alkaline earth cations on DGA Resin, Normal. None show any significant uptake from HCI and only Sr and Ca show moderate uptake from nitric acid concentrations of  $\sim$ 0.5M to  $\sim$ 5 M. The figure on the next page compares the uptake of Sr and Y in both nitric acid and hydrochloric acid. Y(III) is much more strongly retained on the resin than Sr(II) from both acids across all concentrations. Coupling Eichrom's Sr Resin with DGA would allow for excellent separation of Y from Sr for radiopharmaceutical purification or a single step Sr-89/90 measurement procedure.



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The figures on the right show the uptake of various transition and post transition elements. Bismuth is retained across all concentrations of both HNO3 and HCI. Care should be taken if decontamination from Bi is critical to the analysis.



DGA, Normal Resin	Cartridges 2mL	Part Number
	Package of 50	DN-R50-S
Particle Size,	Bottles	Part Number
	Per gram (10g min)	DN-B01-S
50-100 μm	25 grams	DN-B25-S
	50 grams	DN-B50-S
DGA, Branched Resin	Cartridges 2mL	Part Number
Destista Cina	Package of 50	DB-R50-S
Particle Size, 50-100 µm	Bottles	Part Number
	Per gram (10g min)	DB-B01-S

Please note that Eichrom recommends the use of prepackaged cartridges as DGA resin and DGA Branched resins are difficult to wet.

### Diphonix<sup>®</sup> Ion Exchange Resins:

Eichrom industries Diphonix<sup>®</sup> resin is a high performance, gel-type cation resin for the selective separation of metals. Awarded a U.S. patent and the prestigious R&D 100 Award, Diphonix<sup>®</sup> resin is currently, and successfully, utilized in a growing lineup of applications - including the removal of metals from wastewater, the selective separation of iron from acidic solutions and the purification of acids.



Diphonix® resin is constructed of a polystyrene/divinylbenzene matrix in a spherical bead form. The resin is polyfunctional, containing diphosphonic and sulfonic acid groups bonded to the polymer matrix. The diphosphonic acid ligands contribute to the resin's unique selective capability by preferentially removing selected metals from the solution. The hydrophilic sulfonic acid ligands enhance metal ion accessibility into the polymer matrix and significantly improve the exchange kinetics.

### **Superior Performance:**

The advantage of Diphonix resin over conventional ion exchange resins are best characterized by the resin's high degree of selectivity, exception exchange kinetics, ability to achieve a high throughput ratio, it's extremely stable nature and use in highly acidic solutions.





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**Selectivity:** As can be seen on the previous page, in near neutral pH streams, Diphonix® resin is selective for the multivalent cations of various metals.-including zinc, manganese, chromium, uranium, lead, cobalt, nickel and copper- over alkali cations such as sodium, calcium, and magnesium. The resin also removes ferric iron, chromium, uranium, plutonium, and certain metals other metals from strongly acidic solutions. Coordination of these metal cations to Diphonix® is unaffected by increasing hydrogen ion concentrations.

**Throughput:** By selectively removing only targeted metals; a given volume of Diphonix<sup>®</sup> resin can provide a much higher throughput than other commercially available resins. (Throughput is defined as the volume of liquid processed - prior to exhaustion of the resin - to a specified metal's effluent concentration.) This advantage increases as the ratio of non-targeted metals to targeted metals increases. Also, for certain metals or highly acidic solutions, Diphonix<sup>®</sup> resin may be the only viable resin. Diphonix<sup>®</sup> resin may also drive the targeted metal concentrations lower than other resins, allowing discharge limits and process quality criteria to be met.

**Kinetics:** Eichrom's Diphonix® resin provides exceptional exchange kinetics since the hydrophilic sulfonic acid groups allow quick access to the selective diphosphonic acid groups. This gives the resin the ability to react much faster than commercially available chelating resins. Recommended process flux rates depend on the solution chemistry. For typical wastewater treatment, operating flux rates range from 5 to 15 gallons per minute per square foot of cross-sectional resin bed area. Some applications may require lower flux rates to achieve desired effluent quality.

**Physical Stability:** The plastic matrix used in Diphonix<sup>®</sup> over a wide range of thermal and chemical conditions. The diphosphonic acid functional group is incorporated into the matrix at the time of polymerization and is thus very stable.

**Use Criteria:** This highly selective complexing ion exchange resin is applicable to nearly all processes that require the separation of certain classes of metals from a solution. The resin is fully compatible with commercially available ion exchange resin equipment and is recommended under the following circumstances:

- The frequency of regeneration/disposal is high due to high concentration of calcium, magnesium, sodium, etc., exhausting the capacity of the current resin or the disposal costs of spent regenerant and/or spent resin is high.
- The process must remove multivalent metals and not calcium, magnesium, sodium, etc.
- Effluent metals concentrations are not meeting specifications or discharge limits.

**Forms Available:** Diphonix® resin is available in a variety of bead sizes and cross-linkage percentages to suit a number of laboratory and process applications.

Diphonix Resin	Quantity	Part Number
20-50 mesh size	500 grams	DP-B500-20-50
50-100 mesh size	500 grams	DP-B500-C-H
100-200 mesh size	500 grams	DP-B500-M-H
60-100 (on silica)	500 grams	DS-B500-C-H

### Ion Exchange Resins for Analytical Applications:

Eichrom supplies an expanding range of cation and anion exchange resins, purified for the needs of analytical applications. These resins are comparable in quality and performance to the AG® Resins supplied by Bio-Rad® Laboratories. Eichrom Technologies has 16 years of expertise in analytical sample preparation and possesses technical support facilities for radiological and wet chemical applications. Our focus is the development and support of sample preparation methodologies for chemical and biochemical analysis.



### **Cation Exchange Resins:**

Eichrom's line of cation exchange resins for analytical applications are type 50W strong acid cation exchange resins (sulfonic acid exchange groups on styrene divinyl benzene polymeric beads.) Our resins are available in 4% and 8% DVB cross linkages. (<u>Contact us for availability of additional cross linked versions.</u>) All our cation exchange resins are provided in hydrogen form. Our cation resins are supplied in the following dry mesh ranges: 50 - 100, 100 - 200, and 200 - 400.

Cation Exchange Resin Specifications			
Minimum Wet Exch. Cap. (meq/ ml)Water Ret. Cap. (H Form)Maximum Ex- tractable Resi- due*			
4% 8%	1.1 1.7	64 - 72% 50 - 58%	1 mg/g 1 mg/g

\*Per ASTM D 5627, Standard Test Method for Water Extractable Residue from Particulate Ion Exchange Resins

Typical Applications for Cation Exchange Resins: 4% and 8% cross-linked:

- Removal of ethidium bromide from DNA samples
- Removal of propidium iodide from DNA samples
- Separation and preconcentration of peptides and amino acids.
- Removal of cations (desalting)
- Separation of metal ions

Cation Exchange Resin			
	Mesh size	Quantity	Part Number
(50Wx4, Hydrogen form)	100-200	Bottle (500 g)	C4-B500-M-H
		Drum (10 kg)	C4-DR10-M-H
(50Wx8, Hydrogen form )	50-100	Bottle (500 g)	С8-В500-С-Н
		Drum (10 kg)	C8-DR10-C-H
	100-200	Cartridge (2mL / Package of 50)	
		Bottle (500 g)	C8-B500-M-H
		Drum (10 kg)	C8-DR10-M-H
	200-400	Bottle (500 g)	C8-B500-F-H

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### Anion Exchange Resins:

Eichrom's line of analytical anion exchange resins are type 1 strong base anion exchangers (quarternary amine functional groups on styrene divinyl benzene polymeric beads.) DVB cross linkages of 4% and 8% are currently available in dry mesh sizes of 50 - 100, 100 - 200, and 200 - 400. All resins are available in the chloride form. <u>Contact us for availability of other cross-linked or mesh size versions.</u>



Anion Exchange Resin Specifications			
% crosslinked	Minimum Wet Exch.	Water Ret. Cap. (Cl	Maximum Extractable
	Cap. (meq/ml)	Form)	Residue*
4%	1.0	55 - 63%	1 mg/g
8%	1.2	39 - 45%	1 mg/g

\*Per ASTM D 5627, Standard Test Method for Water Extractable Residue from Particulate Ion Exchange Resins

Our ion exchange resins are available in a variety of package sizes including: 500 gram bottles, cases of 9 bottles, and 10 kg drums.

Typical Applications for Anion Exchange Resins: 4% and 8% cross-linked.

Exchange of anions of salts Separation of uranium complexes (uranyl ions) Low molecular weight inorganic anions Cyclic nucleotide assays Fractionation of organic acids.

Anion resin is also available in pre-packed columns and cartridges, please contact Eichrom for currently available packaging options and prices.

Anion Exchange Resin			
	Mesh size	Quantity	Part Number
	50.400	Bottle (500 g)	A4-B500-C-CL
(1-x4, Chloride Form)	50-100	Drum (10 kg)	A4-DR10-C-CL
	100-200	Bottle (500 g)	A4-B500-M-CL
	100-200	Drum (10 kg)	A4-DR10-M-CL
	20-50	Drum (5 kg)	A8-DR5-I-CL
	50-100	Bottle (500 g)	A8-B500-C-CL
		Drum (10 kg)	A8-DR10-C-CL
	100-200	Columns (2mL / Package of 50)	A8-C50-M-CL
(1-X8, Chloride form)		Cartridges (2mL / Package of 50)	A8-R50-M-CL
		Bottle (500 g)	A8-B500-M-CL
		Drum (10 kg)	A8-DR10-M-CL
	200-400	Bottle (500 g)	A8-B500-F-CL
	200-400	Drum (10 kg)	A8-DR10-F-CL

### Ln, Ln2 and Ln3 Series of Resins:

Ln Resin, like most of Eichrom's products, grew out of the transformation of a selective organic extractant into an extraction chromatographic resin. The extractant used in Ln Resin is di(2-ethylhexyl) orthophosphoric acid (HDEHP). References to HDEHP in a chromatographic system date back more than thirty years. A number of these references are shown in the bibliography available at www.eichrom.com.

Early work by Horwitz et al.  $(1975)^1$ , provided distribution coefficients (K<sub>d</sub>) versus nitric acid for various metal ions using HDEHP on a hydrophobic support. This data is reproduced in figure 1 and has been used as the starting point for a number of radiochemical separations in use today. Note that for Eichrom's Ln Resin, K<sub>d</sub> can be converted into k' (an approximation of free column volumes to peak maximum) by dividing by 4.33. Ln Resin has been applied to the analysis of radium, neodymium, promethium and as the data in Figure 1 might indicate, many more analytical applications are possible and may already be operational.

Bill Burnett et al.(1995)<sup>2</sup> provided an excellent reference for the determination of <sup>228</sup>Ra using Ln Resin. Although this reference also demonstrates that Eichrom's TRU Resin can be used to separate <sup>228</sup>Ra from its direct beta particle-emitting daughter, <sup>228</sup>Ac, Ln Resin may find favor with American regulators as it relies on the same HDEHP extractant used in EPA Method Ra-05-1 (EPA Procedures Manual 520/ 5-84-006) without the generation of mixed organic waste. An outline of Bill Burnett's method is provided as Fig. 2. After a barium sulfate precipitation and conversion to barium carbonate, the sample is loaded in 10 mL 0.095M HNO<sub>3</sub> onto a prepackaged Ln Resin column (0.7 g). The column is rinsed with 15 mL of the same nitric acid to remove alkali earths, lead and some other interference. (The load and rinse fractions can be collected and analyzed for <sup>226</sup>Ra by radon emanation.) Finally 10mL of 0.35M HNO<sub>3</sub> is used to quantitatively elute actinium. A source can then be prepared for gas flow proportional counting. For routine work a minimum detectable activity (MDA) of <1.0 pCi/L (0.037 Bg/L) may be obtained for a 2-L sample with 80% barium recovery with less than a 30 minute count. This method was used to analyze samples from









a US-EPA inter-laboratory comparison for a number of <sup>228</sup>Ra samples. All results agreed with the expected values at the 95% confidence interval.

Additional development of this method at Eichrom has resulted in an improved procedure. The BaSO<sub>4</sub> precipitation/ carbonate metathesis steps have been replaced with a cation exchange concentration step. Alpha spectrometry is also indicated for Ra-226 as well has the shorter-lived Ra-224. This method has been published as RAW03, Radium in Water, and is available in the methods section of this website. Additional performance data was presented at the Eichrom North American Users' Meeting in May, 2002.

Ln Resin is also useful in the analysis of Pm-147. In analysis of environmental and waste samples from nuclear facilities it is necessary to separate promethium from other fission products and the actinides to obtain an accurate measurement. A method for water is outlined by Cable, et al. (1997.) A 0.5-2 L water sample can be concentrated by a CaHPO<sub>4</sub> scavenge or by evaporation. The resulting sample is dissolved in 0.2M HNO<sub>3</sub> with ascorbic acid. The ascorbic acid reduces any Fe(III) to Fe(II). A test with 55 mg Fe(II) in solution resulted in no breakthrough of Pm after 35 mL of 0.2M HNO<sub>3</sub>. A typical procedure would use a total of 18-20 mL to load and rinse a standard prepackaged Ln Resin column. Potentially interfering americium is rinsed through the column along with strontium. Promethium is retained along with bismuth, yttrium and the potential tracers samarium or gadolinium. The promethium along with any samarium or gadolinium tracer is eluted with 5 mL of 1M HNO<sub>3</sub>. Corrected recoveries of Pm-147 were >88% using Gd-148 as a yield monitor in one reported test. Excellent decontamination was achieved from Co-60, Cs-134,137 and Sr-89,90. One other potential interference is Ce-144, a beta and gamma emitter. A check with gamma spectroscopy could be used to eliminate this as a potential false positive.

Christian Pin et al. (1996)<sup>6</sup> reported a method for the sequential separation of Sm, Nd, Th and U in silicate rocks. The method uses Eichrom's TRU Resin in series with Ln Resin. For samples high in iron, such as basaltic samples, a 50WX4 strong acid cation exchange column (available from Eichrom) is used up front. After dissolution and possible treatment with cation exchange resin, the sample is loaded onto a TRU Resin column in 1M HNO<sub>3</sub>. Unwanted cations are eluted with rinses of the same acid. The light rare earth elements (LREE) can then be stripped with 0.05M HNO<sub>3</sub>. This fraction from the TRU Resin column of our S-grade 50-100 micron bulk resin. Figure 3 (page 21) shows the elution of La, Ce, Pr, Nd, Sm and Eu. A total of 5.5 mL of 0.25M HCl was used to strip the La, Ce, Pr and Nd with no detectable Sm (ID-TIMS). Then after passing 0.75 mL of 0.75M HCl, Sm appeared in the next 0.5 mL. Finally Eu was collected in the next 0.5 mL of 0.75M HCl. Comparison with 15 international standard reference materials of silicate rocks showed good agreement. Element concentrations were at the mg/g to  $\mu$ g/g level.

Ln Resin has a calculated maximum capacity for Nd of approximately 22 mg/mL of resin. In practice it is normally not recommended to exceed 10-20% of this amount in analytical procedures. Therefore a useful working capacity for Ln Resin would be 2-4 mg of Nd/mL or 4-8 mg/ 2mL pre-packaged column or cartridge.

In 2007 Dan McAlister and Dr. Horwitz published characterizing data for two additional members of the Ln series of resins Ln2 - 2-ethyl-1-hexyl(2-ethyl-1-hexyl)phosphonic acid (HEH[EHP]) and Ln3 - bis(2,4,4-trimethyl-1-pentyl) phosphinic acid (H[DTMPP]). Line drawing comparison of the extractants is presented in the upper right hand side of page 18. The acidity of the ionizable proton of the extractants decrease as the ester oxygens are removed. Therefore, the LN extractant with two ester oxygens is the most acidic followed by LN2 with one ester oxygen and LN3 with no ester oxygens. As a result, each successive member of the Ln series have a weaker extracting group which can allow for the tailoring of target element retention and strip conditions with various chemical systems.

The LN Series of Resins

OH

H[TMPeP] (LN3)





 $M^{3*} + 3(\overline{HY})_2 \leftrightarrow \overline{M(HY_2)_2} + 3H^*$ 

Figure 2. k' Am(III) vs HNO3 and Ga(III) vs HCl on LN, LN2 and LN3

The cartridges of Ln resins can be stacked to allow different members of the lanthanide series to be retained on separate cartridges from the same load solution as is show in the lanthanide series separations to the right.





\*Load = 0.02 M HNO; or HCl \*\*Load = 0.1 M HNO; or HCl

LN Resin	Container	Quantity	Part Number
		25 grams	LN-B25-A
Particle Size,	Bottles	50 grams	LN-B50-A
100-150 μm		200 grams	LN-B200-A
	Columns (2mL)	Package of 50	LN-C50-A
Particle Size, 50-100 µm	Bottles	25 grams	LN-B25-S
		50 grams	LN-B50-S
	Cartridges (2mL)	Package of 50	LN-R50-S
20-50 µm	Bottle	Per gram (10g minimum)	LN-B01-F
LN3 Resin Particle Size	Container	Cartridges (2mL)	Part Number
50-100 μm	Cartridge (2mL)	Package of 50	L3-R50-A



### MnO2 Resin:

MnO2 Resin is one of the products Eichrom launched in 2005. It's first application, the separation of Ra in water samples, was developed and published by Moon, et al.1 The next few paragraphs highlight their work.



Figure 1: Absorption recovery in Ba-133 on MnO2 resin. 10mL deionized water, 25 mg of resin, magnetic stirring for 60 minutes at  $20^{\circ}$ C, pH adjusted with HCl or NaOH. Measurement done with a Nal well-type detector.

Figure 2: Absorption kinetics of Ba-133 with respect to the salinity of the water sample. 10 mL of water sample traced with Ba-133 with 0%, 0.02%, 0.35% and 3.5% added salt. 25 mg MnO2 resin are added to pH 7 solutions under magnetic stirring for different times.

Different parameters such as pH, reaction time, resin quantity, salt effect and flow rate have been evaluated when separating Ra from water using MnO2 resin. For most of the experiments, Ba-133 was used as tracer for Ra. According to Figure 1 and Figure 3, MnO2 resin shows the greatest affinity for Ba-133 when the pH of the solution is between 4 and 8.



The higher the salt content of the water, the slower the kinetics of Ba (or Ra) uptake (Figure 2). The equilibrium is reached after 15 minutes of contact for samples with a salt content between 0 and 0.02%, whereas 25 minutes are required when the water contains 0.35% salt. For waters which composition is similar to seawater (3.5% salts), the equilibrium is reached in 90 minutes.



Figure 4: Absorption recovery of Ba-133 and Ra-228.

The batch uptake of radium and barium by the MnO2 resin from deionised water and seawater have been studied and found comparable. The data in Figure 4 indicates equivalent uptake by the resin under two conditions: 1 gram resin per liter of water and 2.5 gram resin per liter of water.



The recovery of Ba-133 and Ra-228 with respect to flow rate has been studied for 2 types of waters: deionised and artificial seawater. For salt water, the flow rate must not exceed 20mL/min, otherwise a loss in recovery of 30% may be observed. Additionally the chemical recoveries of Ba and Ra start to differ from each other, Ba-133 can thus no longer be considered as a chemical homologue, making its use as an internal standard undesirable.

The MnO2 resin is currently used with Ln Resin and DGA Resin in a method developed by Sherrod Maxwell of Savannah River Site. It is used to pre-concentrate Ra from 1 to 1.5L water samples. 1.25 g/L of MnO2 resin is used per sample. The sample is initially stabilized at pH 6-7 and 25 mg Ca are added per liter. The sample is then loaded onto MnO2 resin with a flow rate of about 15 mL/min. Ra is eluted with 15mL 4M HCl/1.5% H<sub>2</sub>O<sub>2</sub>. The 15 mL solution is left a minimum of 36 hours for Ac-228 ingrowth before being loaded onto 2 cartridges stacked on top of each other: Ln Resin (retention of U and Th) and DGA Resin (retention of Ac-228). Ac-228 is eluted from DGA resin with 10 mL 0.5M HCl, then micro-precipitated with CeF<sub>3</sub> on Resolve<sup>TM</sup> Filter. This method is currently under validation in our laboratory.

Eichrom's MnO2 Resin is available in one particle size (75-150 micron), in both bulk bottles and prepackaged cartridges. See part number information below.

MnO2 Resin	Bottles	Part Number
	100 grams	MN-B100-A
Particle Size, 75-150 µm	Cartridges (2mL)	Part Number
	Package of 50	MN-R50-A



### Nickel Resin:

Eichrom's Nickel Resin is based on the traditional dimethylglyoxime (DMG) precipitation chemistry for nickel analysis, but greatly simplifies the procedure. The Nickel Resin contains the DMG inside the pores of a polymethacrylate resin. The nickel-DMG precipitate occurs on the resin, where it is held and readily separated from the supernatant. Figure 1 shows the chemical structure of DMG and the nickel- DMG complex.

Nickel Resin is an 11% (w/w) loading of DMG, with a resin density of 0.25g/mL. The working capacity of the resin for Nickel is 1.5 mg Ni per mL resin; and is 3.0 mg per 2mL pre-packed column or cartridge.

Figure 2 outlines the nickel procedure. The sample, with a 2 mg Ni carrier, is loading from pH 8-9 ammonium citrate solution. After a 20mL rinse with the same solution, the DMG complex is dissolved and eluted from the column using a small amount of 1.5 - 3M nitric acid. Ni-63 can then be measure by LSC and Ni-59 by X-ray.



Measure Ni-63 by LSC and



Strip: 3 mL 3 M HNO

If Fe-55 needs to be measured from the sample then it should be separated prior to loading on the Ni Resin. Refer to Eichrom method <u>FEW01, Fe-55 in Water</u>, where the separation of iron from nickel is made on the TRU Resin.

Carolina Power & Light evaluated the Nickel Resin. Table 1 presents the results of a side-by-side comparison of the nickel resin with the standard DMG precipitation method on a variety of sample types with a range of Ni-63 activity from  $10^{-6}$  to  $10^{0}$  mCi/unit. Across the whole activity concentration range, the correlation between the precipitation method and the Nickel Resin column was excellent (+/- 10%.)

A decontamination study was also conducted at Carolina P&L. A rad-waste cleanup resin (RWCU Resin) sample was used for this study. Decontamination factors (the ratio of concentration before and after processing the sample through the Nickel Resin procedure) for various activation and fission products are shown in Table 2.

Table 3 outlines general observations made at Carolina P&L about the column method. An overall comparison of these factors, shows that the column method is significantly faster and easier than the precipitation method. Chemical recoveries of the column method are higher and turnaround time is 1/3 of the precipitation method. In the case of this study, the column method allowed for the use of an "environmentally friendly" scintillation cocktail that eliminated the generation of mixed waste in the laboratory.

Table 1: Co	Table 1: Comparison of Precipitation and			
	Column Met Results in µC			
Sample Type	Sample Method	Nickel Column	Ratio	
TL/HS Tank	2.30E-6	2.38E-6	0.97	
Lab Waste Tank	2.66E-6	2.49E-6	1.08	
WECT Tank	4.31E-6	4.17E-6	1.03	
Ni-63 spike	5.07E-4	5.35E-4	0.95	
Ni-59 spike	1.00E-2	1.07E-2	0.93	
DAW     4.18E-2     4.63E-2     0.90				
Radwaste filter	7.40E-1	7.79E-1	0.95	
RWCU Resin	1.83E+0	2.05E+0	0.89	
D. Cahill, Carolina Power & Light, New Hill NC				

Table 2 Decontamination Factors (RWCU Resin Sample)			
Cr-51	<b>Cr-51</b> >37,000		
Mn-54	<b>Mn-54</b> 270,000		
Co-58	110,000		
<b>Co-60</b> 113,000			
Nb-95 13,700			
Cs-134	<b>Cs-134</b> >9,000		
<b>Cs-137</b> 58,000			
D. Cahill, Carolina P&L			

Table 3   General Observations			
Parameter Standard Method Column Meth- od			
Chemical Yield	63 +/- 9%	90 +/- 14%	
LLD (50mL/30 min) 1.27E-7 6.5E-8			
Analysis Time			
Overall 12 hours 4 hours			
Hands-on 8 hours 2 hours			
LSC cocktail Mixed Waste —			
D. Cahill, Carolina P&L, New Hill NC			

The Nickel resin is manufactured on a 100-150 $\mu$ m resin and is available in bulk and in pre-packaged 2mL columns and cartridges. Columns are guaranteed for 6 months from date of purchase.

Nickel Resin	Bottles	Part Number
	25 grams	NI-B25-A
	50 grams	NI-B50-A
	Cartridges (2mL)	Part Number
	Package of 50	NI-R50-A
Particle Size, 100-150 μm	Columns (2mL)	Part Number
	Package of 50	NI-C50-A
	Columns (5mL)	Part Number
	Package of 20	NI5-C20-A
	Cartridges (2mL)	Part Number
	Package of 50	NI-R50-A



### Pb Resin:

Eichrom's Pb Resin is an extraction chromatographic material based on the same crown ether extractant used in the Sr Resin (Figure 1) but at a lower concentration and with a longer chain alcohol for a diluent to facilitate the stripping of Pb from the resin.

Figure 2 shows the uptake of Pb together with several +1 cations and Figure 3 shows Pb together with representative alkali earth metals. Note the similarities to the uptake of these elements on <u>Sr Resin</u>. The retention of Pb is high (i.e., k' > 100) across the range of nitric acid concentrations from 0.1M to 10M. Pb can be eluted from the column using 10mL of 0.1M Citric Acid or 20 mL of water if using a Pb Carrier.

The effect of matrix constituents on lead uptake by the Pb Resin is similar to that for strontium on the Sr Resin. In Figure 4 it can be seen that calcium and sodium have little or no effect on Pb uptake even at concentrations up to  $1\underline{M}$ . Potassium does reduce Pb uptake as its concentration increases. Fortunately the retention of Pb on the Pb Resin is sufficiently high that methods can accommodate up to  $1\underline{M}$  potassium before the k' of Pb drops below 100.









While the uptake of Pb on Pb Resin is maximized in nitric acid, it is possible to load Pb on the resin from HCl. Figure 5 shows a comparison of the uptake curves for Pb from nitric and hydrochloric acid. Above  $0.1\underline{M}$  there is a divergence in the curves, with the nitric acid curve heading higher. There is still adequate retention from HCl at concentrations up to 1 $\underline{M}$ . Above this HCl concentration, the retention of Pb drops off steeply. Vajda, et al. (VN195) published an HCl uptake curve for Pb on Sr Resin that corresponds to this behavior. Vajda reported a method for lead and polonium that allowed Pb to be stripped with 6 $\underline{M}$  HCl. By analogy, this should be possible with Pb Resin as well.

Pb Resin is manufactured in three particle sizes (20-50 $\mu$ m, 50-100 $\mu$ m, and 100-150 $\mu$ m) and is sold in bottles or ready to use in prepackaged columns (for gravity flow) and cartridges.



Source for all published data: Horwitz, E.P.; Gale, N.H.; et al, A Lead-Selective Extraction Chromatographic Resin and Its Application to the Isolation of Lead from Geological Samples, *Analytica Chimica Acta, Vol. 292, pp. 263-273 (1994).* 

Particle Size	Bottles	Part Number
	25 grams	PB-B25-A
	50 grams	PB-B50-A
100-150 μm	100 grams	PB-B100-A
100-150 μm	200 grams	PB-B200-A
	Columns (2mL)	Part Number
	Package of 50	PB-C50-A
Particle Size	Bottles	Part Number
	25 grams	PB-B25-S
50,100 um	50 grams	PB-B50-S
50-100 μm	Cartridges	Part Number
	Package of 50	PB-R50-S
Particle Size	Bottles	Part Number
20-50 μm	Per gram (10g minimum)	PB-B01-F



### **RE Resin:**

Eichrom's RE Resin is an extraction chromatographic material which consists of 1 M octyl(phenyl)-N,Ndiisobutylcarbamoyl-methylphosphine oxide (abbreviated as CMPO) in tributyl phosphate (TBP) coated on an inert methacrylic polymeric support. The CMPO molecule is shown above.



RE Resin is a favorable tool for the group separation of rare earth elements and has been used in conjunction with geological dating and radionuclide transport studies. Due to the high retention of yttrium on RE Resin it has also been applied to the purification of yttrium (Y<sup>90</sup>) used in cancer therapy.



The RE Resin is analogous to Eichrom's TRU Resin. Both are composed of the same extractant system (CMPO/TBP), but the concentration of CMPO is higher in the RE Resin. This increases its affinity for rare earth elements and yttrium out of acidic aqueous solutions. Figures above compare the uptake of selected lanthanide elements on the two resins on a log scale. It can be seen that the uptake of these elements is typically twice as high on RE Resin as on TRU Resin.

Figures on the next page show plots of the distribution factor, k', vs. nitric acid for all of the lanthanide elements, as reported by Huff and Huff. Note that for all the lanthanide elements, k' increases with increasing nitric acid up to 4M. At higher acid concentrations, k' increases for the heavier lanthanides, but falls off for the lighter lanthanides.



Figures in this section were calculated from weight distribution data generated by Huff & Huff (HD193). Eichrom's commercial product is manufactured to established quality specifications. Esser, et. al. developed a method for measuring rare earth elements in environmental water samples. They observed breakthrough of the heavier lanthanides during load (15 FCV) and rinse (25 FCV) steps when the load/strip solutions were 2 and 4N nitric acid. When 6N nitric acid was used, no breakthrough of any lanthanides was observed. This demonstrates the acid dependency of the heavy lanthanides shown in Figure 5. Experiments conducted by Huff & Huff show that only Zr, Th and U are retained on RE Resin over the whole nitric acid range. These researchers did not evaluate the retention of other actinide elements, but, by extrapolation from known data on TRU Resin, it is expected that all the actinides would show strong retention at 1M and higher nitric acid.

RE Resin is manufactured in three particle sizes ar	l is sold in bottles or ready to	use in prepackaged columns (for
gravity flow) and cartridges (for vacuum assisted flo	v.)	

Particle Size	Bottles Part Number			
	25 grams	RE-B25-A		
100 150	50 grams	RE-B50-A		
100-150 μm	Columns (2mL)	Part Number		
	Package of 50	RE-C50-A		
Particle Size	Bottles	Part Number		
50-100 μm	25 grams	RE-B25-S		
	50 grams	RE-B50-S		
	Cartridges (2mL)	Part Number		
	Package of 50	RE-R50-S		
Particle Size	Bottles	Part Number		
20-50 μm	Per gram (10g Minimum)	RE-B01-F		



### Sr Resin:

The extractant system in Eichrom's Sr Resin is 1.0M 4,4'(5')-di-t-butylcyclohexano 18-crown-6 (crown ether) in 1-octanol. A 40% (w/w) loading of this organic solution is loaded onto an inert chromatographic support. The bed density of Sr Resin is approximately 0.35 g/mL.



Diluent: 1-octanol

Figures below show the <u>acid dependency of k'</u> for strontium on Sr Resin, plotted with curves for various other elements. Horwitz, et al. reported these data from studies performed with experimental batches of Sr Resin. Eichrom's commercial product conforms to established specifications that ensure proper performance of Eichrom issued methods.



The uptake of strontium by Sr Resin increases with increasing nitric acid concentration. At  $8\underline{M}$  HNO<sub>3</sub>, k' is approximately 90, and it falls to less than 1 at concentrations of nitric acid less than 0.05M. The alkali and alkaline earth metals (Figures on left) show much lower affinity for the resin than strontium across the concentration range of HNO<sub>3</sub> shown. Among the alkaline earth metals, calcium has lowest uptake and it is easy to separate Sr from Ca. Historical methods to accomplish this separation are tedious and require the use of fuming nitric acid, which is hazardous. Barium retention is somewhat high, but it's uptake peaks at about  $3\underline{M}$  HNO<sub>3</sub> and falls off at higher concentrations. To ensure adequate decontamination of Ba in Sr analysis, load Sr on the resin from  $8\underline{M}$  HNO<sub>3</sub>. With adequate rinsing, Ba will be washed from the column leaving a pure Sr fraction.

The tetravalent actinides show significant retention on the Sr Resin (Figures on right). The addition of oxalic acid as a competitive complexing agent will prevent the retention of actinide elements on the column.

The retention of Pb by Sr Resin is high across a broad range of  $HNO_3$  concentrations. (Third figure from left) It is difficult to strip Pb from this resin without complexing agents. A modified version of this resin has been developed for Pb analysis. Eichrom's Pb Resin has a lower concentration of crown ether (to allow for easier stripping of Pb) and a less water soluble diluent (isodecanol.)

Sr Resin shows resilience to the interference of a number of cations commonly found in environmental and biological







samples. The figure on the top left shows the effect of various cations on strontium uptake by Sr Resin. Ca and Na cause minimal interference up to 0.5M in concentration. The macro levels of calcium found in certain sample types, like milk, can dramatically affect Sr recoveries. In a study conducted at BNFL (formerly Magnox Electric, Berkley, UK), a Sr-85 spike was loaded out of 8M HNO<sub>3</sub> onto a preconditioned, 2 mL Sr Resin column. The column was rinsed with 8M HNO<sub>3</sub> and Sr was eluted with water. Recovery was quantitative for calcium levels up to 320mg; above that level, however, chemical yields declined.

The experimentally determined maximum capacity of the resin for Sr is approximately 21mg per 2mL column. Historically, in the absence of definitive studies otherwise, we have recommended working capacities for all our resins of 10-20% of maximum capacity. In this case, because of the work at Magnox Electric, we can recommend using a carrier of up to 8mg Sr.

As shown in the first figure on this page, potassium can cause significant reduction in Sr uptake even at relatively low levels. Additionally, the presence of <sup>40</sup>K in the Sr fraction could cause a bias in the <sup>90</sup>Sr measurement. For these reasons, in samples with sufficient potassium to be problematic, a selective precipitation of the divalent oxalates is recommended to eliminate the potassium interference.

The second figure demonstrates the different elution profiles of Sr Resin in two different particle sizes. The smaller the particle size is, the narrower the elution band. This means better chromatographic performance, but at the cost of slower flow rates, and, consequently, longer analysis times. Use of vacuum assisted flow permits the use of smaller particle size resins (20-50 $\mu$ m) and (50-100 $\mu$ m) with faster flow rates than gravity flow would allow.

Particle Size	Container	Quantity	Part Number		
100-150 μm		25 grams	SR-B25-A		
	Bottles	50 grams	SR-B50-A		
		100 grams	SR-B100-A		
		200 grams	SR-B200-A		
	Columns (2mL)	Package of 50	SR-C50-A		
	Columns (5mL)	L)Package of 20SR5-C20-AL)Package of 20SR8-C20-AmL)Package of 20SR10-C20-A	SR5-C20-A		
	Columns (8mL)	Package of 20	SR8-C20-A		
	Columns (10mL)	Package of 20	SR10-C20-A		
		25 grams	SR-B25-S		
50-100 μm	Bottles	50 grams	SR-B50-S		
		100 grams	SR-B100-S		
	Cartridges (1mL)	Package of 50	SR1ML-R50-S		
	Cartridges (2mL)	Package of 50	SR-R50-S		
20-50 µm	Bottles	Per gram (10g min)	SR-B01-F		



### **TEVA® Resin:**

One of the more versatile of Eichrom's analytical products is TEVA Resin. It has been applied on a routine basis to the analysis of technetium, the measurement of the tetravalent actinides, and the separation of americium from lanthanides. It is used alone or can be readily combined with other resins to perform more elaborate separations of multiple analytes.

The active component of the TEVA Resin is an aliphatic quaternary amine (see figure to right). As such it has properties similar to those of typical strong base anion exchange resins. However, because the functional groups are in a liquid form, rather than fixed to a polymer backbone (as with IX resins) these groups have greater flexibility to coordinate around target anions. This means that the uptake of these ions is generally higher and often at lower acid concentrations.

This behavior is shown in figures below, which are the acid dependency curves of TEVA Resin for the actinides and technetium from  $HNO_3$  and HCI. Tetravalent plutonium, neptunium and thorium show maximum uptake in the region of 2 <u>M</u> to 4 <u>M</u>  $HNO_3$ . In this acid concentration range, hexavalent uranium and trivalent americium are not well retained, and as such, the resin can readily separate the tetravalents from the other actinides. This ability has been widely exploited.

The decrease in k' for the tetravalent actinides as nitric acid concentrations greater than 2-4M is due to competition from nitrate anions for complexation sites on the resin.

The differences between the uptake curves for nitric and hydrochloric acid can be exploited to separate certain actinides from each other. For example, all the tetravalent actinides can be loaded from  $3\underline{M}$  HNO<sub>3</sub>. Valence adjustment may be required to assure that the actinides are tetravalent. Then, by switching to  $6\underline{M}$  HCI, Th(IV) can be selectively eluted while Pu(IV) and Np(IV) remain on the column.

The retention of Tc(VII), pertechnetate, is also shown above. The resin takes up this anion strongly in solutions of lower nitric or hydrochloric acid concentration. Although not shown above, neutral, and even basic solutions, additionally show a strong uptake of technetium. The use of TEVA Resin in the analysis of Tc has become an industry standard. TEVA Resin is available in a disc format which can tolerate flow rates of up to 200mL/min when used for Tc analysis. Work performed by Darrin Mann's laboratory at K-25 in Oak Ridge demonstrated that the TEVA Resin could be used to isolate Tc-99 from a variety of matrices including alkaline solutions and neutralized acids (See reference MA193 available at www.eichrom.com.)











The figure above shows the effect of uranium and thorium content in a sample on update of neptunium by TEVA Resin from 5M HNO<sub>3</sub>. While the effect of Th is significant and linear, uranium doesn't have much negative impact on Np retention until the concentration reaches 0.05M. In a 15 mL load solution, for example, this would correspond to about 180 mg.

The matrix effects of various polyatomic anions on Np uptake by TEVA Resin from 2M HNO<sub>3</sub> is shown in the top right figure. Oxalate shows the strong effect on neptunium uptake. Oxalato complexes form readily with the tetravalent actinides and these complexes are not extracted by the TEVA Resin. Oxalates are often used in stripping solutions of tetravalent actinides from TEVA Resin.

Phosphates are commonly found in a variety of sample matrices. Its effect on all tetravalent actinides (Pu, Th, and Np) would be similar, but since Th is the least strongly retained by TEVA Resin, it is most readily affected by high levels of phosphate in a sample. Addition of aluminum to the load solution will reduce this effect. Phosphates will preferentially complex aluminum, leaving the tetravalent actinides free to form extractable nitrato complexes.

Another solution to the problem of anionic matrix interferences is shown in the figure at left. In the case of oxalate, increasing the nitric acid concentration of



the load solution from 2M to 3.5M increased the uptake of Np by more than an order of magnitude.

The figure to the right shows the separation of Pu from Np using TEVA Resin. This is accomplished by the selective reduction of plutonium from tetravalent to Pu(III). In this valence state, it behaves like Am, which is not retained at all under any concentration of nitric or hydrochloric acid. In the example in figure above right, plutonium is reduced with hydroquinone, although other reagents such as ammonium iodide and ferrous sulfamate may also be used.

The table below list the elution behavior of a number of commonly occurring elements on the TEVA resin in  $2M HNO_3$  (FCV 1-30), 4M HCL (FCV 31-40) and in water (FCV 41-50.)

		Elution of Selected Elements from a TEVA column							
		2M HNO <sub>3</sub>						4M HCI	H <sub>2</sub> O
	Element	1-5	6-10	11-15	16-20	21- 25	26- 30	31-40	41-50
	Li	100							
	Na	100							
Separation of Np and Pu on TEVA Resin	Mg	100							
8 M HCl + 9 M HCl Hydroquinone 0.5 M HCl	AI	100							
9 M HCI Hydroquinone 0.5 M HCI	К	100							
Pu Np	Ca	100							
	Cr	100							
	Mn	100							
	Fe	100							
0 <sup>1</sup> 0 <sup>2</sup> 0 <sup>1</sup> 0 <sup>1</sup> 0 <sup>1</sup> 0 <sup>1</sup> 0 <sup>1</sup> 0 <sup>1</sup> 0 <sup>1</sup> 0 <sup>1</sup>	Со	100							
	Ni	100							
	Cu	100							
	Zn	100							
	Rb	100							
	Sr	100							
	Y	100							
	Zr	100							
	Мо	100							
	Ag	100							
	Cd	100							
	Cs	100							
	Ba	100							
	La	100							
	Ce	100							
T=23°C	Pr	100							
0 10 20 30 40 50 60	Nd	100							
Free Column Volume	Sm	100							
	Eu	100							
	Hg	4.1	52.5	45.6	23.5				
	Th							100	
	U	100							

100


Another interesting use of the TEVA Resin is to separate americium from the rare earth elements. The figure to the left shows that the rare earths are eluted as a group in a load solution which comprises 1.0 M ammonium thiocyanate and 0.1 M formic acid. Americium is retained under these conditions and can be eluted later with hydrochloric acid. Although the figure left shows the elution of Am with 0.25M HCl, it has been later shown that 2M HCl is a more effective and reproducible eluent for stripping Am from TEVA Resin. Further examples of this application are shown in the bibliography available at <u>www.eichrom.com</u>.

TEVA Resin is manufactured in three particle sizes  $(20-50\mu m, 50-100\mu m, and 100-150\mu m)$  and is sold in bottles or ready to use in prepackaged columns (for gravity flow), cartridges (for vacuum assisted flow), or in a disc format for Tc-99 analysis of large volume water samples. See below for part numbers and descriptions.

Source: Horwitz, E.P., Dietz, M.L., Chiarizia, R., Diamond, H., Maxwell III, S.L., and Nelson, M., "Separation and preconcentration of actinides by extraction chromatography using a supported liquid anion exchanger: Application to the characterization of high-level nuclear waste solutions," Analytica Chimica Acta, 310 (1995) 63-78. (HP195)

Particle Size	Container	Quantity	Part Number
		25 grams	TE-B25-A
		50 grams	TE-B50-A
	Bottles	100 grams	TE-B100-A
100-150 µm		200 grams	TE-B200-A
	Columns (2mL)	Package of 50	TE-C50-A
	Columns (5mL)	Package of 20	TE5-C20-A
	Columns(10mL)	Package of 20	TE10-C20-A
		25 grams	TE-B25-S
		50 grams	TE-B50-S
50,400	Bottles	100 grams	TE-B100-S
50-100 μm		200 grams	TE-B200-S
	Cartridges (1mL)	Package of 50	TE1ML-R50-S
	Cartridges (2mL)	Package of 50	TE-R50-S
	Bottle	Per gram (10g Min)	TE-B01-F
20-50 µm	47mm Discs	50	TE-D50-F



#### **Tritium Column:**

Eichrom's Tritium Column is designed to replace distillation for most routine tritium analyses of aqueous samples. The column works by removing potential interferences in the LSC spectrum, just as distillation does. It is not intended to be an enrichment procedure, and as such, it should be used only in situations where the required detection limit can be achieved by the direct counting of a 5-10 mL aliquot of the sample (plus cocktail) processed through the tritium column.

Figure 1 shows the composition of the Tritium Column and Table 1 explains the purpose and capacity of each component. The Diphonix® Resin removes cations in exchange for hydrogen ions and its theoretical capacity is 0.8 mEq per column. The anion resin is standard chloride form analytical grade 1X8 resin. It exchanges anions in the sample for chloride ions. (It is recommended that the sample pH be >1.) The polymethacrylate component removes organically bound tritium and carbon-14.



Table 1			Table 2			
Component	<u>Target</u>	Capacity	Spike Recov	Spike Recovery Results using Tritium Column		
Diphonix® Resin	Cations	0.8 mEg	Sample Type	Volume	3H Spike	% Recovery
Dipriorities Resili	Cations	0.0 mEq	Distilled water	15 mL	97.2 Bq	96.4% (n=4)
Anion Exchange	A	0.0	Distilled water	25 mL	20.2 Bq	87.6% (n=4)
Resin	Anions	0.8 mEq	Distilled water	25 mL	19.5 Bq	94.9% (n=7)
Delymetheorylate	Organia		Ground water	25 mL	17.1 Bq	91.2% (n=5)
Polymethacrylate		50 mg	Sea water	25 mL	4.0 Bq	90.0% (n=6)
Resin	Molecules	-	Urine	25 mL	85.1 Bq	91.1% (n=4)

Table 2 shows the average spike recoveries for a variety of sample matrix types passed through the Tritium Column.

The column successfully removes potential radioactive interferences. In a study performed at Eichrom, a mixture of radioisotopes (<sup>60</sup>Co, <sup>137</sup>Cs, <sup>233</sup>U, <sup>90</sup>Sr/<sup>90</sup>Y, <sup>210</sup>Pb, <sup>230</sup>Th, total activity 16.9 Bq) was spiked into eight tritium-spiked solutions (distilled and sea water samples). In every case, after passing the sample through the Tritium Column, the number of counts in the region above the tritium window was not different than the number of counts in the same region of an un-spiked sample. (See Table 3)

Table 3					
	Results of	contamina <sup>3</sup> H	Contaminant	Contaminant	
Sample Type	Volume	Spike (Bq)	spiked (Bq)	found (Bq)	
Distilled Water	25 mL	19.5	16.9	<mda (n="2)&lt;/td"></mda>	
Distilled Water	25 mL	20.2	16.9	<mda (n="2)&lt;/td"></mda>	
Sea water	25 mL	4.0	16.9	<mda (n="4)&lt;/td"></mda>	

In another experiment, Daniel Cahill of Carolina Power & Light measured the activity of fission and activation products in PWR and BWR reactor coolant samples before and after passing through Eichrom's Tritium Column. Table 4 summarizes the before column activity of each sample. After the column, no measurable activity was detected in the BWR sample and only a trace level of activity of <sup>60</sup>Co with > 50% counting error was detected in the PWR sample. Because the amount of <sup>60</sup>Co measured was so low and the error so high, and because the BWR sample contained more than 10 times the <sup>60</sup>Co before the column and none after the column, it is assumed that the trace amount measured in the PWR sample is an artifact due to the background of the gamma counter.

See our newsletter archive for additional data on the performance of Eichrom's Tritium Column. The studies reported here, and elsewhere on our website, conducted in Eichrom and customer laboratories, demonstrate that the Tritium Column performs equivalently to the traditional distillation technique. We encourage you to try using this column as a substitute for distillation of your aqueous tritium samples.

Eichrom's Tritium column is manufactured as a 2mL prepackaged column (total bed volume of all three components.) It is available in packages of 20, 50 and 200.

Table 4 Carolina Power & Light Samples				
Isotope	PWR Sample	BWR Sample		
Cr-51	2,900	1,900		
Mn-54	518	5,590		
Co-58	4,740	4,960		
Fe-59	109			
Co-60	392	5,990		
Sn-113	230			
Nb-95	4,220	116		
Zr-95	2,210			
I-131	14,200			
Cs-134	1,1200			
Cs-137	1,320			
La-140		1,550		
Ce-144		203		
D. Cahill, Carolin	a Power & Light, N	New Hill NC		



Particle Size	Columns	Part Number
75-150 μm	Package of 50	H3-C50-A



# TRU Resin:

Eichrom's TRU Resin is an extraction chromatographic material in which the extractant system is octylphenyl-N,Ndi-isobutylcarbamoylmethylphosphine oxide (abbreviated CMPO) dissolved in tri-n-butyl phosphate (TBP). The CMPO molecule is shown in figure at the upper right. The bed density of TRU Resin is approximately 0.37 g/mL, with a working capacity of 2 mg Am per mL of resin or 4 mg Am per 2mL pre-packed column. This value represents 20% of the theoretical maximum loading capacity of the resin.



The CMPO/TBP solvent system complexes actinide elements and extracts them out of certain aqueous solutions. Figure, above left, shows the uptake of various actinide elements from nitric acid solutions by the TRU Resin. The y-axis, <u>k'</u>, is a measure of uptake, corresponding to the number of free column volumes (FCV) to peak maximum in a chromatographic column. FCV is a measure of the interstitial space (or void volume) of a column. Values higher than 100 indicate strong uptake, values between 1 and 50 indicate weak uptake, and values below 1 indicate no retention on a column. The data presented here was generated by Horwitz, et al. at Argonne National Laboratory using experimental batches of TRU Resin. Eichrom's commercial product conforms to established specifications that ensure proper performance of Eichrom issued methods.

The tetravalent actinides show extremely high retention on the column, with k' in the range of  $10^4$ - $10^6$  at nitric acid concentrations in excess of 2<u>M</u>. The k' for Hexavalent uranium is approximately one order of magnitude lower. The <u>k'</u> curve for trivalent americium plateaus at about 100 FCV in the range of 1-5<u>M</u> nitric acid. It is important to note that

trivalent plutonium behaves similarly to americium and that pentavalent neptunium exhibits very low retention at any nitrate concentration.

Figure on the left shows the retention behavior of the actinides on TRU resin in HCI. The very low affinity of the column for Am in chloride media forms the basis for the selective stripping of Am (and trivalent plutonium) from TRU Resin.



The uptake curves on the previous page were measured using spiked solutions, whereas figures above show the effect of a number of matrix components on the uptake of the actinides.

The effect of various cations on the retention volume (k') of americium in 2M nitric acid on the TRU Resin is plotted in figure 4. Among the cations shown, calcium and divalent iron show no effect on americium retention. This is particularly valuable since both calcium and iron are present in large quantities in many environmental and bioassay samples. They are often added as carriers in precipitation steps. Trivalent iron shows a significant, negative effect on Am retention. If it is suspected that iron is present in a sample, a reducing agent such as ascorbic acid should be added to ensure that all Fe(III) is reduced to Fe(II). Ammonium thiocyanate can be used as an indicator for this; it is a deep red color in the presence of Fe(III) and colorless in the presence of Fe(II).

Aluminum actually increases the uptake of Am on the TRU Resin column. This happens because the aluminum (III) cation is readily hydrated in solution. This has the effect of increasing the activity of nitrate ions in solutions, driving the formation of the americium-nitrato complex that is readily extracted by the CMP0/TBP extractant system.

As shown in the figures above, the commonly occurring polyatomic anions show no real effect on americium retention on the TRU Resin, but have a significant adverse effect on the retention of the tetravalent actinides. While the middle figure shows only the effects on the retention of neptunium, the behavior of the other tetravalent actinides under the same conditions is analogous. The curve for plutonium is nearly identical to the neptunium curve; that for thorium would be parallel, but approximately one order of magnitude lower.

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Fortunately, the retention of neptunium and plutonium is high enough that even relatively large concentrations of sulfate and phosphate (up to 0.5M) should not cause breakthrough under the conditions indicated in our published methods. If thorium is being separated on TRU Resin, care should be taken for samples with elevated levels of phosphate or sulfate. The addition of aluminum nitrate will reduce the matrix effect as phosphate will complex the aluminum preferentially leaving thorium less affected.

As shown in the right most figure on the last page, the effect of oxalate is quite significant for the tetravalent actinides which readily form oxalato complexes that are not extracted by the CMPO/TBP solvent system. Uranium retention is not affected significantly by oxalate up to 0.1M. For this reason, solutions of oxalate salts can be used to strip tetravalent actinides selectively from the TRU Resin without eluting uranium.

#### Elution of Selected Elements on a TRU Resin Column (fine particles)

	1-5	6-10	11-15	16-20	21-25	26-30	31-40
Li	98.4	<19	-	-	-	-	-
Na	92.8	<1.2	-	-	-	-	-
Mg	100	-	-	-	-	-	-
AI	99.8	<2.9	-	-	-	-	-
ĸ	81.8	40.9	-	-	-	-	-
Ca	100			-			-
Cr	100	-	-	-	-	-	-
Mn	100	-	-	-	-	-	-
Fe	102	12.3	-	-	-	-	-
Co	100	-	-	-	-	-	-
Ni	100	-		-		-	-
Cu	100	_	-	-	-	-	-
Zn	100	-	-	-	24	1	-
Sc	100	-	-	-	-	::+::	-
Y	23.4	76.8	3.5		-	-	-
Zr	-	_	-	-		-	75.0
Ru	82.6	<19.2	-	-	-	-	-
Rh	100	_	-	-	-	-	-
Ag	100	-	-	-	-	-	-
Cd	100	-	121		-	-	-
Ba	100	-	-	-	-		
La	-	-	-	-	-	30.0	72.0
Ce	-	-	-	-	-	<25.0	75.0
Pr	-	-	-	-	-		100
Nd	-	-	_	-	-	-	96.0
Sm	-		-	-		- :	100
Eu	-	-	-	-	-	-	>99
Hg .	(100)	(60)	(19)	-	-	-	-
Pb	100	-	-	-	-	-	-
Am*		_	<u> </u>	-	-	2	>99
radiometr 1–30 f.c.v.	ic : 2 <u>M</u> HND: v. : 0.05 <u>M</u>						
	use of unce	rtainties in					

The table above lists the elution behavior of a number of commonly occurring elements on the TRU resin in  $2M \text{ HNO}_3$  (FCV 1-30) and in 0.05M HNO<sub>3</sub> (FCV 31-40.)

TRU Resin is manufactured in three particle sizes (20-50 $\mu$ , 50-100 $\mu$ , and 100-150 $\mu$ ) and is sold in bottles or ready to use in prepackaged columns (for gravity flow) and cartridges (for vacuum assisted flow.)

Particle Size	Container	Quantity	Part Number
		25 grams	TR-B25-A
	Bottles	50 grams	TR-B50-A
		100 grams	TR-B100-A
100-150 µm		200 grams	TR-B200-A
	Columns (2mL)	Package of 50	TR-C50-A
	Columns (5mL)	Package of 20	TR5-C20-A
	Columns (10mL)	Package of 20	TR10-C20-A
		25 grams	TR-B25-S
	Bottles	50 grams	TR-B50-S
50-100 µm		100 grams	TR-B100-S
		200 grams	TR-B200-S
	Cartridges (1mL)	Package of 50	TR1ML-R50-S
	Cartridges (2mL)	Package of 50	TR-R50-S
20-50 µm	Bottles	Per gram (10g min)	TR-B01-F

# **UTEVA® Resin:**

UTEVA Resin has been applied to a variety of analytical challenges. These include uranium measurements in environmental samples, sample preparation of high uranium content samples prior to analysis for other elements, the sequential determination of uranium, plutonium, and americium, the measurement of actinides in urine, and the measurement of actinides in high level waste.



The extractant in the UTEVA Resin, diamyl, amylphosphonate (DAAP, see figure above), forms nitrato complexes with the actinide elements. The formation of these complexes is driven by the concentration of nitrate in the sample solution. Therefore, the uptake of the actinides increases with increasing nitric acid concentration. Figure at left is a plot of the <u>k'</u> (a measure of uptake corresponding to the number of free column volumes to peak maximum) vs. nitric acid concentration.

It can be seen that the uptake from  $HNO_3$  is very similar for each of the tetravalent actinides and uranium. All have strong retention (k'>100) above 5<u>M</u> HNO<sub>3</sub>. Note that Am is not retained at any HNO<sub>3</sub> concentration. This fact is important in developing analytical separation schemes. Plutonium can be reduced to Pu(III) with ferrous sulfamate. At this valence state, it behaves the same as Am(III).

Figure above left is a similar graph that shows the effect of HCl on the retention of tetravalent neptunium, thorium, and hexavalent uranium on UTEVA Resin. The large difference in k' for uranium and thorium in the range of 4-6M HCl allows for the selective elution of Th from the resin after both Th and uranium have been loaded.

Figure above left implies that uranium can be stripped efficiently from the UTEVA Resin with a relatively small volume of very dilute  $HNO_3$  (e.g., 0.01-0.05<u>M</u>). In practice, however, it appears that HCl is more efficient in stripping uranium and it is recommended that, where possible, HCl be used in place of nitric acid. Concentrations up to 1<u>M</u> HCl have been shown to quantitatively elute uranium. 15mL is a sufficient volume for a 2mL pre-packed column or cartridge.

Horwitz, et al. reported the data in the figures on this page from studies performed with experimental batches of UTEVA Resin. Eichrom's commercial product conforms to established specifications that ensure proper performance of Eichrom issued methods. Please refer to our <u>product specifications</u> for details.





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As in most analytical situations, the presence of significant concentrations of matrix elements can affect the proper operation of methods based on UTEVA Resin. The figures on this page show the effect of certain polyatomic anions on the retention of neptunium and uranium, respectively, from  $2\underline{M}$  HNO<sub>3</sub>. It should be noted that the effect on tetravalent neptunium is more significant than the affect on uranium. It has been seen in practice that thorium is affected similarly to neptunium by these anions.

Because phosphate occurs quite commonly in a variety of biological and environmental samples, its effect is most relevant. Fortunately the addition of aluminum to the sample matrix can significantly reduce this issue. Phosphate anions readily complex tetravalent actinides. This phosphato complex is not extracted by the DAAP. Added Al can effectively tie up the phosphate preventing its interference with Np (or Th) uptake by the resin. In some methods, as much as 1M Al(NO<sub>3</sub>)<sub>3</sub> might be added to counteract the effects of phosphate.

The theoretical maximum loading capacity of UTEVA Resin for uranium is approximately 37 mg/mL of resin bed. In practice, it is not recommended to exceed 20% of this amount, or 7.5mg per mL of resin. This corresponds to a working capacity for uranium of 15 mg per 2mL prepacked column. The bed density of UTEVA Resin is 0.39 g/mL.

The first table on the next page shows the elution behavior of nearly three dozen elements. As can be seen, nearly all of the test elements can be removed with on 10 FCV of 2M HNO<sub>3</sub>. (Zr and Ru each require  $\sim$ 15 FCV.) As expected from the first figures for UTEVA resin, no trace of uranium is observed even after 30 FCV of the 2M HNO<sub>3</sub>. Once the eluent is change to dilute (here, 0.02 M) HNO<sub>3</sub> however, the removal of uranium is essentially complete in only 10 FCV.



Table 3. Elution beha	vior of selected elements on a	U/TEVA-Spec (fine particle) colu	m
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	×			uting (%) <sup>a</sup> HNO3			-0.02 M HINO3 -
		,	Number of free		, b	71	0.02 81 11103
Element	1-5	6-10	11-15	16-20	21-25	26-30	31-40
Li	99.4	_	_	_		_	_
Na	95.0	<1.2	-	· - ·	_	_	_
Mg	101	_	_	-	_	_	_
AL	92.0	<5.8	_	_	_	_	_
ĸ	<123	-	-	-	_	_	_
Ca	92.4	<13.3	-	-	_	_	_
Cr	94.7	<3.3	_	_	_	_	-
Mn	96.5	<0.7	_	-	_	_	_
Fe	94.0	⊲0.3	_	-	_	_	_
Co	99.1	<7.1	_	-	_	_	_
Ni	97.8	<2.0	_	_	_	_	_
Cu	98.1	<2.6	_	_	_	_	_
Zn	96.8	<1.2	_	_	_	_	_
Rbc	102	_		_	_	_	_
Sr	101	_	_	_	_	_	_
Y	97.9	<2.9	_	_	_	_	_
Zr	56.4	35.7	(1.4)	_	— .	_	
Ru	59.6	<19.2	<19.2	_	_	_	_
Rh	91.7	_	_		_	_	
Ag	103	-	_	_	_	_	_
C4	96.4	<0.7		_	_	_	_
Csd	100	-			_	_	_
Ba	96.6	<14.4	_	-	_	_	_
La	108	_	_	_	_	_	_
Ce	96.3	<25	-	_	_	_	_
Pr	(136)	_	_	_	_	_	_
Nd	99.0	-	_	· – ·	-	_	_
Sm	120	-	-	-	-	_	_
Eu	100	-	· _	_	-	_	_
РЬ	98.4	<5.8	-	_	_	-	_
U.	_	-	_		_	_	>99

UTEVA Resin is manufactured in three particle sizes (20-50m, 50-100m, and 100-150m) and is sold in bottles or, ready to use, in prepackaged columns (for gravity flow) and cartridges (for vacuum assisted flow.) See below for a table of part numbers and descriptions.

Source: Horwitz, E.P., et al, Separation and preconcentration of uranium from acidic media by extraction chromatography, Analytica Chimica Acta, Vol.266, pp. 25-37(1992) (HP392)

<sup>a</sup> Because of uncertainties inherent in the ICP-AES method used for quantitation, the fractions shown for a given element may not total 100%. Values in parentheses are subject to considerable uncertainty and are intended only as a guide. Feed solution contained -0.02 M oxalic acid to solubilize zirconium.

b 1 FCV = 0.60 mL here.

Particle Size	Container	Quantity	Part Number
		25 grams	UT-B25-A
	Dettles	50 grams	UT-B50-A
	Bottles	100 grams	UT-B100-A
100-150 µm		200 grams	UT-B200-A
	Columns (2mL)	Package of 50	UT-C50-A
	Columns (5mL)	Package of 20	UT5-C20-A
	Columns (10mL)	Package of 20	UT10-C20-A
	Bottles	25 grams	UT-B25-S
		50 grams	UT-B50-S
50 100 um		100 grams	UT-B100-S
50-100 μm		200 grams	UT-B200-S
	Cartridges (1mL)	Package of 50	UT1ML-R50-S
	Cartridges (2mL)	Package of 50	UT-R50-S
50-100 μm (15ml 100-150 μm (3mL)		Package of 20	UT15-C20-S-3PA
20-50 µm	Bottles	Per gram (10g minimum)	UT-B01-F

Accessories for Resin Cartridges:

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#### Cartridge Vacuum Box:

24 Hole Vacuum Box:Part number: AC-24-BOX12 Hole Vacuum Box:Part number: AC-12-BOX

The vacuum boxes have a high density polyethylene (HDPE) top for acid compatibility and a clear bottom, made from PETG plastic, for visibility of collection tubes. Vacuum boxes come complete with a screw in PVC vacuum gauge, a rack insert which holds 50cc centrifuge tubes, a starter package of yellow outer tip and white inner support tube along with manifold plugs for sealing off unused holes. The HDPE top can be removed and set on the bench top, supported by 2.5 cm legs, without disturbing cartridges and/or tips in use. The set up and use of these vacuum boxes is described in <u>Eichrom method VBS01</u>.

#### Inner Liner: Part number: AC-24-LINER & AC-12-LINER

Clear plastic, one-piece liner is used to collect waste solutions. The total capacity is approximately 2 or 4 liters depending on 12 or 24 hole size.

## 10 ml and 20 ml Cartridge Reservoirs: Available with Frits

Similar to syringe barrels, these reservoirs attach to the top of Eichrom's prepacked cartridges with a Luer-slip mechanism. Graduations allow for easier monitoring of flow rates when using the vacuum box. Available in packages of 25 and 200.

Reservoir Size	Package Size	Non-Fritted Part Number	With Frit Part Number
10 mL	Package of 25	AC-25-RV10	AC-25-RV10-F
10 mL	Package of 200	AC-200-RV10	AC-200-RV10-F
20 mL	Package of 25	AC-25-RV20	AC-25-RV20-F
20 mL	Package of 200	AC-200-RV20	AC-200-RV20-F

#### Yellow Outer Tips and White Inner Support Tubes:

Disposable tips and tubes are used to connect the cartridges to the vacuum box. The The tips and support tubes are available in packages of 1000.

Yellow Outer tips PN:	AC-1000-0T
Inner support tube PN:	AC-1000-TUBE-PE



New for

2014







Accessories for Resin Columns:

25 mL Extension Funnels:

Part number AC-120

The extension funnels are two-piece polypropylene reservoirs with a capacity of 25 mL that snap securely into to the top of our 2mL columns. Available in packages of 20

#### Column Connectors:

Part number: AC-121

The Column connectors, available separately, are the piece that connects the 25mL Extension Funnels to the column. Available in packages of 100

#### 2ml Empty Columns:

Part number: AC-141-AL

The same columns used for our standard 2mL pre-packed columns, these polypropylene columns have an inner diameter of 0.8cm and a snap off tip. Use when a slightly different sized column is required for the analysis. The columns are supplied in packages of 100, with 100 caps, bottom frit inserted and 100 extra top frits.

## 15 Hole Column Rack:

Part number AC-103

This clear acrylic column rack measures 24 cm (long) x 11.5 cm (wide) x 16 cm (high). The 15 holes are arranged so that the columns can drip directly into 50cc centrifuge tubes placed in their original polystyrene packaging.

## **Column Tip Closures:**

Part number AC-130.

These flexible, plastic sleeves fit on the bottom of an opened column to prevent complete drying out of the bed when the column needs to be used a second time in a procedure. (e.g., <u>SRW01, Sr-89/90 in Water</u>) They are available in packages of 100.

5 ml Empty Columns: pk of 50	Part	number:	AC-50E-5M
20 ml Empty Columns: pk of 20	Part	number:	AC-20E-20M

These columns are used when a larger bed volume of one of Eichrom's extraction chromatography or ion exchange resins is required. Columns are supplied with bottom frit pre-inserted, in packages of 50 or 20, including bottom tip closures, caps and top frits.

#### 200 ml Extension Funnels

Part number: AC-20X-20M

These extension funnels are single piece plastic reservoirs that fit securely into the top of Eichrom's 5ml and 20 ml columns. They have a useful capacity of 200-250 ml. Available in packages of 20.









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# **Resolve™ Filters:**

#### 0.1µm PPE, 25mm diameter, package of 100 Part number: RF-100-25PP01

# 0.1µm PPE, 47mm diameter, package of 50 Part number: RF-100-25PP01

Filters typically are manufactured to meet specifications for removal of particles to result in defined solution purity characteristics. For example, a  $0.1\mu$ m rated pore size filter is typically defined as capable of removing 99.98% of particles  $0.1\mu$ m from a liquid. The pores present on the surface, however, may be much larger or smaller than 0.1 $\mu$ m. Maximum peak resolution in alpha spectroscopy requires as uniform a filter surface as possible to ensure a uniform deposition of the rare earth fluoride precipitate. Recently reported resolution guality concerns indicate that manufacturing specifications and guality control procedures of existing suppliers may not address the specific needs of the alpha spectroscopist. Eichrom's line of Resolve<sup>™</sup> Filters (0.1 $\mu$ m polypropylene, 25mm dia.) is manufactured to specifications appropriate for alpha source preparation.



Performance in Alpha Spectroscopy

Maintaining a more homogeneous pore size distribution in the membranes used in Resolve Filters, has allowed Eichrom to produce filters that give reproducibly good performance in alpha spectroscopy. We have evaluated the performance of multiple lots of filters in source preps of <sup>232</sup>U and <sup>241/243</sup>Am. Figures 4 and 5 show alpha spectra for a <sup>232</sup>U and <sup>241/243</sup>Am on a typical lot of Resolve<sup>™</sup> Filters.

## Quality Control

Incoming filter material is monitored and screened using by measuring resolution of <sup>241</sup>Am and <sup>243</sup>Am is performed on each filter lot manufactured. Specifications on this test are set at FWHM 50 keV for each peak.



# **Product Validation**

Some scientists consider that the two sides of filters may perform differently in the preparation of alpha sources. We have investigated whether different sides of Resolve Filters give different peak resolution. Two studies have been run, one internally at Eichrom and a second by Dr. Barry Stewart, at the Carlsbad Environmental Monitoring Research Center. In the study conducted in Carlsbad, various parameters were considered, including, "glossy" side vs. "non-glossy" side and the use or absence of substrate. Dr. Stewart made source preps of <sup>241</sup>Am and <sup>243</sup>Am on Resolve Filters using ASTM procedure C1163-98 (50  $\mu$ g La carrier.) His study matrix evaluated the two sides of the filter and the effect of substrate use. His results are shown in the table below.

Sample	Substrate?	Orientation	FWHM (keV)	<sup>243</sup> Am Recovery	<sup>241</sup> Am Recovery
1	No	Glossy down	28	98.4%	98.4%
2	No	Glossy down	25	95.7%	95.5%
3	No	Glossy up	22	101.9%	97.6%
4	No	Glossy up	26	100.2%	99.3%
5	Yes	Glossy down	25	100.5%	96.1%
6	Yes	Glossy down	23	98.6%	97.0%
7	Yes	Glossy up	25	95.1%	94.8%
8	Yes	Glossy up	26	97.6%	95.0%

The Carlsbad results indicate that each side of the filter works equally well and that excellent peak resolution of the  $^{243}$ Am peak (<30 keV) could be achieved with or without the use of substrate on the filter.

A similar study conducted at Eichrom evaluated the peak resolution of the two Am isotopes on each side of the filter. In this case, the geometry of the filter was described by the orientation of that filter surface in the roll of material prior to cutting into filter discs, inside and outside. In this study source preps were made following Eichrom's procedure <u>SPA01</u> which uses 50  $\mu$ g Ce. As shown in the following table, the FWHM of both the <sup>241</sup>Am and <sup>243</sup>Am did not vary as a function of the filter geometry.

<sup>243</sup> Am Outside	<sup>243</sup> Am Inside	<sup>241</sup> Am Inside	<sup>241</sup> Am Inside
36	29	41	26
28	22	33	37
31	38	32	45
32	35	37	44
43	42	37	42
43		41	
mean = 34	mean = 33	mean = 36	mean = 39
Std. Dev = 7	Std. Dev. = 8	Std. Dev. = 4	Std. Dev. = 8

While the actual peak resolution that is observed is a function of many factors, Eichrom's QC program ensures that the filter itself will not contribute to poor resolution in alpha spectrometry.



**Resolve Filter:** 

3μm, PTFE Laminate, 25mm diameter, package of 60 Part number: RP-50-47PTFE3

3μm, PTFE Laminate, 47mm diameter, package of 50 Part number: RP-50-47PTFE3



Eichrom's Resolve PTFE filter is designed to optimize performance of the radon/thoron correction algorithms in any alpha emission filter sample counter. Filter quality is assured through a series of radiochemical and physical performance testing.

Resolve	Resolve PTFE Filter Properties		Acceptance Criteria
Diameter	47 mm	Background	< MDA dpm alpha/beta
Reference Pore Size	3.0 μm nominal	Alpha Resolu- tion	< 60 keV FWHM
Thickness	0.15 – 0.4 mm thick	Alpha Recovery	>80% Post air sampling
DOP Efficiency	>99.99 % 0.3 um @5.33 cm/sec	Filter curling	≤2.5 mm
Chemical Com- patibility	Resistant to most chemicals and solvents	Use: Grid Side Down, Opposite Air Flow	
Air Permeability	6 ft <sup>3</sup> /ft <sup>2</sup> /min @ 0.5" H <sub>2</sub> O minimum		

The polytetrafluorethylene (PTFE) filtration surface was selected to minimize particulate penetration into the body of the air filter. The minimization has two impacts on the quality of the data obtained from the filter. First, alpha emission efficiency is increased due to the minimizing of absorption of the alpha emission by the filter. Secondly, the resolution of the alpha spectroscopy is increased with the reduction in the amount of matter that the alpha emission might hit on the path to the detector. The PTFE material is laminated to a stiff polymeric backing to minimize any changes to the filtration surface due to handling or atmospheric conditions.



Measurement performed on Canberra iSolo at Eichrom Technologies.

Post air sampling spectrum from glass fiber filter spike with <sup>233</sup>U & <sup>241</sup>Am.



Post air sampling spectrum from Resolve filter spiked with <sup>233</sup>U & <sup>241</sup>Am.



# Health Physics Sampling Smears:

Our first joint listing product of Eichrom/NPO is sampling smears for Health Physics area contamination monitoring. We offer flexibility of two formats for our smears, bulk package 1-3/4" diameter cloth smears with pressure sensitive adhesive backing and support and the complete smear/support with record paper. The pressure sensitive backing of the cloth smear allows for easy reattachment to the support or other surface. The support provides a "firmer" smear that users prefer. For part number I85SS2005, the bulk smears with support are glued on to a 3-1/2"x4-3/4" score fold record paper. The smear with support can be removed together from the record paper and placed loose-ly in a planchet pan for analysis. The smears with record paper are shipped in boxes of 500 each and the bulk smears are shipped in boxes of 2500.



Item Description	# of Boxes	Part Number	
Bapar backed Smaara far recording (500) par bay	1-49	185SS2005	
Paper backed Smears for recording (500) per box	≥50*		
Smaara Bulk paakagad (2500) par bay	1-49	I85SS2500	
Smears-Bulk packaged (2500) per box	≥50*		

# **Method Listings:**

<u>Eichrom Methods</u> are available at <u>www.eichrom.com</u>. Please note that methods are available in either Microsoft Word or Adobe Acrobat (pdf) format viewable online or delivered via email. You can also select to receive updates to Eichrom Methods through the website.

Reference No	Title	Revision No
ACS07	Uranium in soil (2 gram sample)	1.5
ACU02	Americium, Plutonium and Uranium in Urine	1.6
ACU02	VBS Americium, Plutonium and Uranium in Urine (with Vacuum Box System)	1.6
ACU03	Neptunium and Thorium in Urine	0.4
ACW01	Uranium and Thorium in Water	1.7
ACW03	Americium, Plutonium, and Uranium in Water	2.1
ACW03 VBS	Americium, Plutonium and Uranium in Water (with Vacuum Box System)	1.6
ACW04	Americium in Water	0.2
ACW08	Thorium and Neptunium in Water	1.7
ACW10	Thorium in Water	1.0
ACW11	Gross Alpha Radioactivity in Water	0.3
ACW13 VBS	Thorium, Plutonium and Uranium in Water (with VBS)	1.3
ACW16 VBS	Am_Np_Pu_Th_Cm_U in Water (with VBS)	1.0
ACW17	Am_Np_Pu_Th_Cm_U_Sr in Water (with VBS)	1.0
FEW01	Iron-55 in Water	1.0
NIW01	Nickel 63/59 in Water	1.2
OTS01	Lead in Soil	1.1
OTW01	Lead-210 and Polonium-210 in Water	2.0
OTW02	Tritium in Water	1.0
RAW01	Radium-228 in Water	1.1
RAW03	Radium in Water	1.3
RAW04	Radium-226/228 in Water (MnO2 Resin & DGA Resin Method) Cerium Fluoride Microprecipitation for Alpha Spectrometry Source Preparation of	1.0
SPA01	Actinides	1.1
SRW01	Strontium-89,90 in Water	1.4
TCS01	Technetium-99 in Soil	1.8
TCU01	Technetium-99 in Urine (TEVA Disc Method)	1.2
TCW01	Technetium-99 in Water	1.6
TCW02	Technetium-99 in Water (TEVA Disc Method)	1.3
VBS01	Setup and Operation Instructions for Eichrom's Vacuum Box System (VBS)	1.3



# **Compendial Methods:**

ASTM, HASL, the DOE Methods Compendium and others continue to update their methods with the latest technology. Methods using Eichrom's extraction chromatographic material are now included in these sources. Methods available on line can be directly accessed via the provided links at <u>www.eichrom.com</u> where you can download or purchase a complete copy. Additionally, methods referenced in this document can be obtained by contacting the publishing body:

ASTM: D19.04 (RADI	OACTIVITY IN WATER)
D5811-08	Standard test method for Strontium-90 in water
D7168-05	Standard test method for <sup>99</sup> Tc in water by solid phase extraction disk
D7535-09	Standard test method for Lead-210 in water
ASTM C26.05 (NUCL	EAR FUEL CYCLE, METHODS OF TEST)
C1000-05	Standard test method for radiochemical determination of uranium isotopes in soil by alpha spectrometry
C1001-05	Standard test method for radiochemical determination of plutonium in soil by alpha spectrosco- py
C1310-01 (2007)	Standard test method for determining radionuclides in soils by inductively coupled plasma-mass spectrometry using flow injection preconcentration
C1345-08	Standard test method for analysis of total and isotopic uranium and total thorium in soils by in- ductively coupled plasma-mass spectrometry
C1387-08	Standard guide for determination of Technetium-99 in soil
C1432-03(2008)	Standard test method for determination of impurities in plutonium: acid dissolution, ion ex- change matrix separation, and inductively coupled plasma-atomic emission spectroscopic (ICP/ AES) analysis
C1473-05	Standard test method for radiochemical determination of uranium isotopes in urine by alpha spectrometry
C1475-05	Standard guide for determination of Neptunium-237 in soil
C1507-07	Standard test method for radiochemical determination of Strontium-90 in soil
C1561-03	Standard guide for determination of Plutonium and Neptunium in Uranium Hexafluoride by al- pha spectrometry
C1614-05	Standard practice for the determination of <sup>237</sup> Np, <sup>232</sup> Th, <sup>235</sup> U and <sup>238</sup> U in urine by inductively coupled plasma-mass spectrometry (ICP-MS) and gamma ray spectrometry
C1636-06a	Standard guide for the determination of Uranium-232 in uranium hexafluoride
C1647-06	Standard practice for removal of Uranium or Plutonium, or both, for impurity assay in uranium or plutonium materials
Contact:	ASTM International, www.astm.org 100 Barr Harbor Drive, PO Box C700, West Conshohocken, Pennsylvania, USA 19428-2959

# Compendial Methods (Continued):

HASL-300, 28TH EDI	TION
SE-03	(sequential methods, page 19) Americium, Plutonium and Uranium in water
TC-02-RC	Technetium-99 in water - TEVA resin
AM-04-RC	(Americium, page 16) Americium in QAP water and air filters
Contact:	HASL-300 Methods published by US Department of Homeland Security Environmental Meas- urements Laboratory now hosted by NBL http://www.nbl.doe.gov/htm/EML_Legacy_Website/procman.htm
DOE METHODS CON	IPENDIUM
RP280 <sup>(A)</sup>	Determination of Lead-210 in water using extraction chromatography
RP300 <sup>(A)</sup>	Nickel-59 and Nickel-63 determination in aqueous samples
RP501 <sup>(A)</sup> , REV. 1	Determination of total radioactive Strontium in high-level samples using extraction chromatog- raphy
RP550 <sup>(A)</sup>	Technetium-99 analysis using extraction chromatography
RP725 <sup>(A)</sup>	Group actinide screening using extraction chromatography
RP800 <sup>(A)</sup>	Sequential separation of Americium and Plutonium by extraction chromatography



# Selected Bibliography:

Reference	Technical Papers Covering Eichrom Products:
CR296* Diphonix	Chiarizia, R., et al. "Diphonix® Resin: A Review of Its Properties and Applications". Separation Science and Technology. Vol. 32, (1997) pp. 1-35. (CR296).
HD193* TRU / RE	Huff, Edmund A and Huff Doris R. "TRU-Spec and RE-Spec Chromatography: Basic Studies and Applications". <i>34th ORNL/DOE Conference on Analytical Chemistry in Energy Technology</i> . Gatlinburg TN. October 1993. (HD193).
HP104* DGA	Horwitz, E. P., et. al., "Novel Extraction Chromatographic Resins Based on Tetraalkyldiglycolamides: Characterization and Potential Applications," <i>Solvent Extraction and Ion Exchange</i> , Vol. 23, p. 319-344 (2005)
HP105* Beryllium	Horwitz, E. P. and McAlister, D. R., "The Separation of Beryllium from Selected Elements Using the DipexÒ Extraction Chromatographic Resin," <i>Solvent Extraction and Ion Exchange</i> , Vol. 23, 611-629 (2005)
HP193* TRU	Horwitz, E.P. et al. "Separation and Preconcentration of Actinides from Acidic Media by Extraction Chromatography". <i>Analytica Chimica Acta</i> . 281 (1993) 361-372. (HP193).
HP194* Pb	Horwitz, E.P. et al "A lead-selective extraction chromatographic resin and its application to the isolation of lead from geological samples". <i>Analytica Chimica Acta</i> . 292 (1994) 263-273. (HP194).
HP195* TEVA	Horwitz, E P and Maxwell, Sherrod L. et al. "Separation and Preconcentration of Actinides by Extrac- tion Chromatography Using a Supported Liquid Anion Exchanger: Application to the Characterization of High-Level Nuclear Waste Solutions." <i>Analytica Chimica Acta</i> . 310 (1995) pp.63-78. (HP195).
HP197* Actinide Resin	Horwitz, E.P. et al. "DIPEX: A New Extraction Chromatographic Material for the Separation & Precon- centration of Actinides from Aqueous Solution". <i>Elsevier Reactive &amp; Functional Polymers</i> . 00 (1997) REACT1088. (HP197)
HP199*	Horwitz, E. P., Extraction Chromatography of Actinides and Selected Fission Products: Principles and Achievement of Selectivity (HP199)
HP204	Horwitz, E. P., McAlister, D. R., Bond, A. H., Barrans, R. E., Williamson, J. M., "A Process for the Sep- aration of <sup>177</sup> Lu from Neutron Irradiated <sup>176</sup> Yb Targets," <i>Applied Radiation and Isotopes</i> , Vol. 63, 23-36 (2005)
HP292* Sr	Horwitz, E P et al. "A Novel Strontium-Selective Extraction Chromatographic Resin". <i>Solvent Extraction and Ion Exchange</i> . 10 (1992) 313. (HP292).
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VN195*	Vajda, N. Et al. "A Novel Technique for the Simultaneous Determination of <sup>210</sup> Pb and <sup>210</sup> Po using a Crown Ether." Journal of Environmental Radioactivity. In Press.



Recent Presentations available at www.eichrom.com/radiochem/meetings.

Date / Event / Location					
Title	Author(s)				
October 2013, Radiochemistry and Radiobioass	October 2013, Radiochemistry and Radiobioassay Measurements Conference, Rohnert Park, CA				
CS Resin	Lawrence Jassin, <i>Eichrom Technologies</i>				
Separations using TEVA Resin	Andrew Knight, <i>University of Iowa</i>				
Rapid Preparation Method for the Measurement of Trace U-	Giaquinto, J.M.; Partridge, J.D., ORNL				
232 in HEU using Resolve Filters					
Technical Challenges with Divalent Cation Mitigation	Eric Eitrheim, University of Iowa				
Shielding Properties	Dan McAlister, Earl Jacobson, Rick Kaisner.				
	Eichrom Technologies				
Development of A Rapid Method for Po, Th, and U Analysis in Hydraulic Fracturing Flowback Water	Andrew Nelson, <i>University of Iowa</i>				
Utilizing combinations of Co-precipitation, solvent extraction	Dan McAlister and Phil Horwitz, <i>Eichrom Technologies</i>				
and chromatography to design efficient analytical and prepar-					
ative scale separations					
	say Measurements Conference, Fort Collins, CO				
Pushing the Limits for Detection of Trace Gallium Impurities in MOX Spent Fuels	Joe Giaquinto, J.S. Delashmitt and T.J. Keever , ORNL				
Rapid Analytical Protocol for Bk-249 Processing Campaigns	Jeff Delashmitt, R.D. Canaan, D.L. Denton, J.M. Giaquinto,				
	R.R. Smith, J.E. Sutherland, B.K. Woody, ORNL				
Separation Methods Utilizing Oxalate-HCI on Anion Exchange Resins	Dan McAlister and Phil Horwitz, PG Research Foundation				
Extraction Chromatography: a microscopic view	Phil Horwitz, PG Research Foundation				
Radioprotection for the radiochemist: How shielding and con-	Lawrence Jassin, Eichrom Technologies				
tamination control can help to protect you in the lab					
Rapid Fusion Method for Plutonium in Large Rice Samples	Sherrod Maxwell and B.K. Culligan,				
	Savannah River Nuclear Solutions				
Rapid Method for Determination of Radiostrontium in Large	Sherrod Maxwell and B.k. Culligan				
Soil Samples	Savannah River Nuclear Solutions				
Matrix and High Loading Effects on Eichrom Resins	Dan McAlister and Phil Horwitz, PG Research Foundation				
Eichrom User's Group Round Table: Questions and "soulful	Panelists: Phil Horwitz, Dan McAlister, Sherrod Maxwell,				
cogitations" to your most vexing separations	Larry Jassin				
March 2012, Marc IX Co	nference, Kailua-Kona, HI				
Eichrom Workshop on Emergency Response Methods- Acti- nides, Sr and Ra-226	Lawrence E. Jassin, <i>Eichrom Technologies</i>				
The Rapid Determination of Strontium-89 and Strontium-90 in	Terence O´Brien and Lawrence E. Jassin, <i>Eichrom Technolo-</i>				
Environmental Samples	gies, and E. Philip Horwitz and Daniel McAlister, PG Research				
	Foundation				
March 2012, ACS Nation	al Meeting, San Diego, Ca				
Recent Advances in the Recovery and Purification of Actinium	E. Philip Horwitz and Daniel McAlister, <i>PG Research Founda-</i>				
Isotopes	tion, and J.T. Harvey, Northstar Medical Radioisotopes				
Method for the Separation of Tc-99m from Low Specific Ac-	E. Philip Horwitz and Daniel McAlister, <i>PG Research Founda</i> -				
tivity Mo-99	<i>tion</i> , and J.T. Harvey, <i>Northstar Medical Radioisotopes</i>				
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November 2011, Radiochemistry and Radiobioassay Measurements Conference, Destin FL			
Influence of Large Univalent Anions on Ligand Selectivity	E. Philip Horwitz, Daniel McAlister		
initialite of Large Onivalent Anons on Ligana Selectivity	PG Research Foundation, Inc.		
Radium Separation Method:	E. Philip Horwitz, Daniel McAlister		
Crown Ether Extraction from Dilute Perchloric Acid	PG Research Foundation, Inc.		
Characterisation of a Cu Selective Resin for Use in the Pro-	Dr. Steffen Happel		
duction of Cu Isotopes	Triskem International SAS		
Lanthanide Separations	Daniel McAlister, E. Philip Horwitz PG Research Foundation, Inc.		
CL Resin based methods for the Separation and Determination of CI-36 and I-129 in environmental and Decommissioning Samples	Dr. Steffen Happel Triskem International SAS		
Rapid Radiochemical Analyses In support of Fukushima	Sherrod L. Maxwell, Brian K. Culligan Savannah River Nuclear Solutions		
The Science of Radiochemical Separations	E. Philip Horwitz PG Research Foundation, Inc/Eichrom Technologies LLC		
August 2011, CANDU Users' Mo	eeting, Toronto, Ontario, Canada		
Rapid, Fully Disposable No Distillation Tritium Sample Prepa- ration	Lawrence Jassin Eichrom Technologies/ NPO		
October 2010, Radiochemistry and Radiobioa	ssay Measurements Conference, Richland WA		
What Does Eichrom's Quality Control System Do for You?	<ul> <li>Sarah Tejchma, Joel Williamson, Jill Bryant, Terence O'Brien,</li> <li>Lawrence Jassin <i>Eichrom Technologies LLC</i></li> <li>E. Philip Horwitz, Daniel McAlister</li> <li><i>PG Research Foundation, Inc.</i></li> </ul>		
Rapid Radiochemical Methods	Sherrod L. Maxwell, Savannah River Nuclear Solutions		
Rapid Methods for the Isolation of Actinides Sr, Tc and Po from Raw Urine (Are you using the right aqueous phase chemistry?)	Daniel McAlister, E. Philip Horwitz, PG Research Foundation, Inc.		
September 2009, International Workshop on Eme	ergency Radiobioassay, Ottawa, Ontario, Canada		
Rapid Methods for the Isolation of Actinides Sr, Tc and Po	Daniel McAlister, E. Philip Horwitz		
from Raw Urine	<i>PG Research Foundation, Inc.</i> James T. Harvey, <i>Northstar Engineered Technologies, LLC</i>		
October 2009, Radiochemistry and Radiobioass	ay Measurements Conference, San Antonio, TX		
Removing a Needle from a Haystack with a Small Fork: Or can a series of small columns handle a kilogram of matrix?	E. Phillip Horwitz and Dan McAlister, PG Research Foundation, Inc.		
Rapid Methods for Actinides and Sr-89/90 in Soil	Sherrod L. Maxwell, Brian K. Culligan and Gary Noyes Savannah River Nuclear Solutions		
Radionuclide Generator Systems	Daniel R. McAlister and E. Philip Horwitz PG Research Foundation, Inc.		
Eichrom Technical Support Busters	Terence C. O'Brien and Lawrence E. Jassin Eichrom Technologies LLC		
Tandem Separations of Radionuclides Using an automat- ed system	Daniel R. McAlister and E. Philip Horwitz PG Research Foundation, Inc.		
Simplifying Separations by Column Coupling	E. Philip Horwitz and Daniel R. McAlister PG Research Foundation, Inc.		



# Eichrom Application Notes: (Available at <u>www.eichrom.com</u>)

Title	Reference Number
Eichrom Product Catalog 2014	EPC2014
Eichrom Bibliography	5/14
Determination of Thorium in High Purity Aluminum by ICP-MS	AN502
SrRSA: Strontium Isotope Residual Salt Analysis	AN503
Determination of Pb in Steel by flow injection – AAS	AN601
Beryllium Analysis Matrix Removal (ICP-AES)	AN602
Metal Impurities in Uranium, Plutonium and Mixed Oxides	AN603
Tritium Columns	AN1001
Resolve® Filters for Alpha Spectrometry Source Preparation	AN1101
Resolve® PTFE Laminate Filters	AN1102
Diphonix and Monophos Resin	AN1201
Ion Exchange Resin for Analytical Applications	ZZ407
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