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New Method for Removal of Spectral Interferences for Beryllium Assay using  
Inductively-Coupled Plasma Atomic Emission Spectrometry

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### **Abstract**

Beryllium has been used widely in specific areas of nuclear technology. Frequent monitoring of air and possible contaminated surfaces in U.S Department of Energy (DOE) facilities is required to identify potential health risks and to protect DOE workers from beryllium-contaminated dust. A new method has been developed to rapidly remove spectral interferences prior to beryllium (Be) measurement by inductively-coupled plasma atomic emission spectrometry (ICP-AES). The ion exchange separation removes uranium (U), thorium (Th), niobium (Nb), vanadium (V), molybdenum (Mo), zirconium (Zr), tungsten (W), iron (Fe), chromium (Cr), cerium (Ce), erbium (Er) and titanium (Ti). A stacked column consisting of Diphonix Resin<sup>®</sup> and TEVA Resin<sup>®</sup> reduces the levels of the spectral interferences so that low level Be measurements can be performed accurately. If necessary, an additional anion exchange separation can be used for further removal of interferences, particularly chromium. The method has been tested using spiked filters, spiked wipe samples and certified reference material standards with high levels of

interferences added. The method provides very efficient removal of spectral interferences with very good accuracy and precision for beryllium on filters or wipes. A vacuum box system is employed to reduce analytical time and reduce labor costs.

## **Introduction**

Beryllium has been used widely in specific areas of nuclear technology because of its ability to reflect neutrons and its efficiency in the production of neutrons when exposed to alpha emitters.<sup>1</sup> The U. S. Department of Energy (DOE) promulgated the Chronic Beryllium Disease (CBD) prevention program in 1999 (10CFR Part 850) to protect DOE workers from beryllium-contaminated dust. This requires frequent monitoring of air and possible contaminated surfaces in U.S DOE facilities to identify potential health risks. Samples which include filters and smears are digested and analyzed using inductively coupled plasma atomic emission spectrometry (ICP-AES). A number of elements can interfere spectrally with the beryllium measurement. Interference correction software is used at the ICP-AES but at higher levels the spectral interferences cannot be handled adequately. A rapid separation method to remove spectral interferences is needed to allow accurate measurement of beryllium at low levels to meet requirements.

PG Research Foundation (Darien, IL, USA) has developed a beryllium separation method for Eichrom Technologies (Darien, IL, USA) using Be Resin<sup>®</sup> to remove spectral interferences, but this method can be affected by large amounts of interferences that can reduce Be retention on the resin. In these cases one or more guard columns may be needed prior to Be Resin to remove large amounts of interferences. Fluoride, if present, must also be complexed with boric acid to prevent Be losses using the Be Resin method. An adjustment of each sample to pH 2 using an indicator is also required.<sup>2</sup> The U.S. Department of Energy (DOE) Y12 National Security Complex uses a pass-through approach using LN-3 Resin<sup>®</sup> (bis (2,4,4-trimethylpentyl) phosphinic acid extractant, Eichrom Technologies) to remove spectral interferences. LN-3 Resin will retain uranium, niobium and molybdenum under the conditions used (dilute sulfuric-nitric acid mixture) while allowing Be to pass through. Vanadium, however, a severe spectral interference, is not retained using this method. If vanadium is present, the Be Resin method must be used after LN-3 resin.<sup>3</sup>

A new method has been developed in the Savannah River Site (SRS) 735-B Environmental Bioassay Laboratory to remove spectral interferences prior to beryllium (Be) measurement by inductively-coupled plasma atomic emission spectrometry (ICP-AES). The ion exchange separation removes uranium (U), thorium (Th), niobium (Nb), vanadium (V), molybdenum (Mo), zirconium (Zr), tungsten (W), iron (Fe), chromium (Cr), cerium (Ce), erbium (Er) and titanium (Ti). A stacked column made of 5 ml of Diphonix Resin and 2 ml of TEVA Resin reduces

the levels of the spectral interferences significantly. Using this approach, Be is not affected by large amounts of interferences that can reduce Be recovery in methods where it is first retained and then eluted from a resin. Instead of retaining beryllium ions to separate interferences, the beryllium is passed through the Diphonix Resin-TEVA Resin column and the interferences are removed on the resins. An optional anion resin column separation can also be used to remove additional chromium if needed.

Diphonix Resin is similar to Be Resin in retention performance, but much less expensive. In the new SRS method, Diphonix Resin is used to remove spectral interferences instead of retaining the beryllium. Diphonix is a resin with geminally-substituted diposphonic acid groups chemically-bonded to a styrene-divinylbenzene matrix, that was developed by Argonne National Laboratory and the University of Tennessee.<sup>4,5,6</sup> Diphonix Resin is used in the SRS Environmental Bioassay Lab (EBL) to recover and measure actinides in fecal samples<sup>7</sup>. Diphonix Resin has a very high retention for uranium, thorium, vanadium, iron, erbium and cerium even in the presence of dilute hydrofluoric acid.

Other spectral interferences are removed on TEVA Resin, which is a resin coated with a liquid ion exchanger (Aliquat-336<sup>TM</sup>).<sup>8</sup> The retention of niobium, titanium, molybdenum, zirconium, and tungsten as anionic fluoride complexes on quaternary amine anion exchange resin is well-known.<sup>9,10,11,12</sup> Retention of hafnium, niobium and zirconium fluoride complexes using Aliquat-336 extractant has also been documented.<sup>13</sup> TEVA Resin retains niobium, titanium, molybdenum, zirconium, and tungsten from dilute hydrofluoric acid-hydrochloric acid solutions.

The filter digestion method already employed at SRS uses nitric acid, hydrochloric acid and hydrofluoric acid. Since fluoride is present, utilizing fluoride complexing to form anionic complexes with key spectral interferences was a logical approach to consider.

A 0.20M HF-0.1M HCl solution was used in the new column separation, following evaporation steps to convert to this acid matrix. Uranium, thorium, cerium, erbium, vanadium, chromium (partial) are retained on Diphonix Resin, while niobium, titanium, molybdenum, zirconium, and tungsten are retained on TEVA Resin as fluoride complexes. In 0.20M HF-0.1M HCl, beryllium forms a fluoride complex anion that will pass through the Diphonix Resin column. Without fluoride ions present,  $\text{Be}^{+2}$  cations would be retained on Diphonix Resin under low acid conditions. Be also passes through TEVA resin when 0.20M HF-0.1M HCl is used as the eluant. The concentration of chloride ions present is high enough to reduce beryllium ion retention on TEVA resin, while niobium, titanium, molybdenum, zirconium, and tungsten are still strongly retained. Chromium has different valence states and forms different cationic, neutral and anionic complexes.<sup>14</sup> As a result, Diphonix Resin removes a fraction of the chromium present, but not all of the chromium depending on the extent of chromium complexing with fluoride.

If more effective chromium removal is required, the sample may be further purified using an anion exchange resin separation using 0.2M HF. Under these conditions, Be is retained while chromium passes through the anion resin. Be can be eluted using 1M HCL-0.25M HF. If the 234.861 nm emission line is used when

residual Cr is present (since this line is not significantly affected by Cr), the additional anion resin separation is not required.

The new method was tested using spiked filters, spiked wipe samples and certified reference material standards with and without high levels of interferences added.

## **Experimental**

### **Reagents**

The resins employed in this work are TEVA Resin® (Aliquat <sup>TM</sup>336), Diphonix resin® (100-200 mesh) and Anion Resin (1-X8, chloride form, 100-200 mesh) available from Eichrom Technologies, Inc., (Darien, IL, USA). Nitric acid and hydrochloric acid were high purity-grade acids (Optima <sup>TM</sup>-Fisher Scientific, Inc.). Hydrofluoric acid was reagent grade (Fisher Scientific, Inc.). All water was obtained from a Milli-Q2<sup>TM</sup> water purification system. Element standards (1,000 or 10,000 parts per million Be, Cr, Nb, Mo, Ti, Fe, U, Th, Ce, Er, V, Zr, W) were obtained from High Purity Standards (Charleston, SC, USA). Certified reference material filter standards were obtained from High Purity Standards (Charleston, SC, USA).

### **Procedures**



*Column preparation.* TEVA Resin cartridges containing 2 ml of each resin were obtained from Eichrom Technologies, Inc.. Small particle size (50-100 micron) resin was employed, along with a vacuum extraction system (Eichrom Technologies). Diphonix Resin columns (5 ml) and Anion Resin columns (2.8 ml) were prepared from a water slurry (~1:1 water : resin) using small Fast Rad Columns (Environmental Express Inc., Mt Pleasant, SC, USA) and a 24 column vacuum box.

*Sample Preparation.* Figure 1 shows a flow chart of the sample preparation method prior to ion exchange. Filters (37 mm cellulose ester filters, Environmental Express Inc., Mt Pleasant, SC, USA) and wipes ( 15 cm x 15 cm Ghostwipes<sup>®</sup>, Environmental Express Inc., Mt Pleasant, SC, USA) were placed in 150 ml digestion tubes (Environmental Express Inc., Mt Pleasant, SC, USA), Be and/or spectral interferences were added to each tube. Four milliliters of 15.7M nitric acid ( $\text{HNO}_3$ ) and one milliliter of 12M hydrochloric acid ( $\text{HCl}$ ) were added to each tube. After the reaction subsided, 1 ml of 28M hydrofluoric acid ( $\text{HF}$ ) was added to each digestion tube. The digestion tubes were placed on a Hot Block heating system (Environmental Express Inc., Mt Pleasant, SC, USA) and heated at ~95C until the samples reached complete dryness. Wipes were a medium dark color when digested to dryness on this initial step. For filters, 2 ml of 30 wt% hydrogen peroxide and 2 ml of 1M HF were then added to each digestion tube. For wipes, 0.5 ml of 15.7  $\text{HNO}_3$  was added and the tube was swirled to fully contact the wipe residue. Then 2 ml of 30 wt% hydrogen peroxide and 2 ml of 1M HF were added to each wipe digestion tube. The digestion tubes were heated to dryness. For wipes, care was given to ensure the wipe residue was not overheated at dryness and to prevent charring and overheating at this step. A lightcolor wipe residue was obtained at dryness by

minimizing heating after dryness was reached. Five ml of 0.2M HF-0.1M HF was added to each tube and heated to dryness. Three ml of 0.2M HF was added to each digestion tube and heated to dryness. Samples were redissolved in 10 ml of 0.2M HF-0.1M HCl, heating filters for ~6 minutes and wipes for ~8 minutes to redissolve.

*Column separation.* Figure 2 shows a flow chart of the ion exchange method using Diphonix Resin and TEVA Resin cartridges. Figure 3 shows a picture of the stacked Diphonix –TEVA resin columns on the vacuum box. Fifty milliliter centrifuge tubes were used to collect rinse or final purified fractions. Each stacked Diphonix –TEVA Resin column was conditioned using 8 ml of 0.2M HF-0.1M HCl. This solution was discarded. The redissolved filter or wipe samples were loaded onto the Diphonix –TEVA Resin columns at ~1 drop per second and this purified solution was collected in labeled 50 ml tubes. The digestion vessel was rinsed well with 3 ml of 0.2M HF-0.1M HCl, capping the vessels and swirling vigorously. After the initial load solution has passed through the columns, the rinse solutions were added and allowed to drain at ~1 drop per second. Eight ml of 0.2M HF-0.1M HCl was added to each column and allowed to drain at ~1-2 drops per second. The tubes were removed and the volume was adjusted to 20 ml with deionized water. It should be noted that when samples analyzed on an ICP-ES without an HF resistant nebulizer the volume maybe adjusted to 20 ml with 0.8M boric acid instead of water.

An additional anion exchange removal is not typically required. But if additional removal of chromium was needed, a 10 ml aliquot of the initial purified solution was evaporated to dryness in a digestion tube on the Hot Block system. Five ml of 0.2M HF were added and evaporated to dryness. The samples were redissolved in 8 ml of 0.2M HF,

warming for ~5 minutes to redissolve each sample. Anion resin columns (2.8 ml resin) were placed on the vacuum box and conditioned by adding 5 ml of 0.2M HF at ~1 ml per minute. Redissolved samples were loaded to the anion resin columns at ~1 ml per minute. Three ml of 0.2M HF was added to each digestion vessel to rinse the tube and this rinse was added to each column. Ten ml of 0.2M HF were added to each column and allowed to drain at 1-2 ml/minute. The rinse solutions were discarded and new labeled tubes were placed in the vacuum box. Be was eluted from the anion resin using 10 ml of 1M HCl-0.25M HF at 1-2 ml/minute.

### Apparatus

A Hot Block heating system (Environmental Express, Mount Pleasant, SC) was used to digest filters and wipes. Polycarbonate vacuum boxes with 24 positions and a rack to hold 50 ml plastic tubes were used. A Perkin Elmer 3000 XL simultaneous axial ICP-ES with cyclonic spray chamber, low-flow GemCone (cross flow) nebulizer was used to perform the elemental measurements. Polycarbonate vacuum boxes with 24 positions (Eichrom Technologies, Darien, IL, USA) and a rack to hold 50 ml plastic tubes were used.

### Results and Discussion

Table 1 shows the levels of interferences (at the instrument after dilution to 20 ml volume). The new method was tested with this level of interferences to determine the removal capability of this method. Filters and wipes were spiked with low level Be and certified reference material Be and BeO filters (High Purity Standards, Charleston, SC, USA) were also tested.

The results in Table 2 show the average spectral interference removal results for spiked Be wipes (1-6), and filters (7-10). The interference levels added are based on a final dilution volume of 20 ml. The data in Table 2 shows that the removal for spectral interferences is very good. Uranium removal was 99.996% at the 300 ppm U level. Chromium removal is shown with and without additional anion exchange. It should be noted that the 234.861 nm emission line for Be, which is not significantly impacted by Cr, can be used to measure Be with only minimal interference correction applied. Although its sensitivity is slightly less than the 313.042 nm and 313.107 nm emission lines, the report limit of 0.01ug Be per filter sample and 0.03 ug/wipe sample can be met using the 234.861 nm line when needed. Iron was also tested at the 2500 ug/ml level and was removed down to the same level of iron shown in this table. The iron removal is extremely efficient and makes the use of the 234.861 nm beryllium emission line feasible, since iron has a very adverse impact on the 234.861 nm line.

Table 3 shows the Be Recovery results for spiked Be wipes (1-6), and Be filters (7, 8) and BeO filters (9, 10) based on measurements using the 234.861 nm emission line. The interference levels added are based on a final dilution volume of 20 ml. Be was added at the 1 ppb level at the ICP-AES, equivalent to 0.02 ug Be/sample. The Be and BeO filters are certified reference material from High Purity Standards (Charleston, SC, USA). The results are not affected by Cr levels shown in Table 1, with minimal interference correction applied. The performance limits for Be testing and accreditation by the American Industrial Hygiene Association (AIHA) are  $\pm 25\%$ . The average Be recovery was 104.4%, with a relative standard deviation

of 9.67%.

Table 4 shows the Be Recovery results for spiked Be wipes (1-6), and Be filters (7, 8) and BeO filters (9, 10) based on measurements using the 313.042 nm emission line. The initial results are affected by Cr levels shown in Table 1 at the 1 ppb Be level (but not the 5 ppb level), but after the Cr levels are lowered using the additional anion exchange step the Be results look very good even at the 313.042 nm emission line. The average Be recovery after anion exchange was 96.6%, with a relative standard deviation of 7.62%.

Table 5 shows the Be Recovery results for spiked Be wipes (1-6), and Be filters (7, 8) and BeO filters (9, 10) based on measurements using the 313.107 nm emission line. The initial results are affected by Cr levels shown in Table 1 at the 1 ppb Be level (but not the 5 ppb level), but after the Cr levels are lowered using the additional anion exchange step the Be results look very good. The average Be recovery after anion exchange was 110.3%, with a relative standard deviation of 14.2%.

Table 6 summarizes additional Be recovery results using the 234.861 nm emission line on for spiked wipes and certified reference material filters. The spiked wipes are at the 0.02 ug level (1 ppb at instrument), while the certified reference material filters have reference values of 0.1 ug/sample (5 ppb at the instrument). The average Be recovery was 99.18% and the relative standard deviation for this set of data is 9.16%. This is very good performance considering that the 1 ppb level at the ICP-AES is only about 2 times the method report limit.

Table 7 summarizes additional Be recovery results using the 313.042 nm emission line on for spiked wipes and certified reference material filters. The spiked wipes are at the 0.02 ug level (1 ppb at instrument), while the certified reference material filters have reference values of 0.1 ug/sample (5 ppb at the instrument). The average Be recovery was biased high at the 1 ppb level due to Cr interference at this line, but not at the 5 ppb level. After additional anion exchange to lower Cr levels, the average Be recovery was 106.42 % and the relative standard deviation for this set of data is 14.79%

Table 8 summarizes additional Be recovery results using the 313.107 nm emission line on for spiked wipes and certified reference material filters. The spiked wipes are at the 0.02 ug level (1 ppb at instrument); while the certified reference material filters have reference values of 0.1 ug/sample (5 ppb at the instrument). The average Be recovery was biased high at the 1 ppb level due to Cr interference at this line, but not at the 5 ppb level. After additional anion exchange to lower Cr levels, the average Be recovery was 107.7% and the relative standard deviation for this set of data is 14.69%

Table 9 summarizes additional testing of BeO filters from High Purity Standards (Charleston, SC) at the 0.2 ug Be/filter level. The average Be recovery was -16.4% using column separation at this level. For comparison, the average recovery at the 0.2 ug Be/filter currently analyzed routinely in SRS F/H lab has been -21.87% and -14.8% in SRS 735-B laboratory. These results are all within +/- 25% performance limits and demonstrate no impact to the BeO results by using the

column separation. The column separation digestion process actually undergoes additional digestion compared with the routine digestion method. To convert to the 0.2M HF-0.1M HF matrix, the samples are evaporated in 5 ml 0.2M HF-0.1M HF. This results in additional heating with HCL and HF, which become concentrated just before dryness.

Table 10 summarizes LCS (Laboratory Control Sample) results on filters, using the 313.107 nm line, without interferences added, analyzed using the ion exchange method. The plot includes data from the 1 ug Be/filter and 10 ug Be/filter spike levels. The average bias for 8 data points at the 1 ug Be/filter level was -2.85%, with a 3.09% relative standard deviation (50 ppb Be at the instrument). The average bias for 4 data points at the 10 ug Be/filter level was 4.95%, with a 4.16% relative standard deviation.

Table 11 summarizes LCS results on wipes, using the 313.107 nm line, without interferences added, analyzed using the ion exchange method. The plot includes data from the 1 ug Be/wipe spike level (50 ppb at the instrument). The average bias for 10 data points at the 1 ug Be/filter level was -7.39%, with a 9.30% relative standard deviation.

Table 12 summarizes Be spike recovery results at or near the Report Limit of 0.01 ug Be/filter, using the 313.107 nm line, without interferences added, analyzed using the ion exchange method. The plot includes data at the 0.01 ug Be and 0.02 ug Be/filter levels. The average bias for 20 data points at the 0.01 ug Be/filter level was 2.56% with a 8.96% relative standard deviation. The average bias for 25 data points

at the 0.02 ug Be/filter level was 4.65% with a 6.0% relative standard deviation.

Table 13 summarizes Be spike recovery results at or below the Report Limit of 0.03 ug Be/wipe, using the 313.107 nm line, without interferences added, analyzed using the ion exchange method. The plot includes data from the 0.02 ug Be and 0.03 ug be/wipes levels. The average bias for 29 data points at the 0.02 ug/wipe level, less than the 0.03 ug/wipe report limit, was 0.45% with an 8.8% relative standard deviation. The average bias for 5 data points at the 0.03 ug/wipe level was 7.33% with a 4.2% relative standard deviation.

Table 14 summarizes Method Detection Limit (MDL) and Report Limit (RL) data for filters, while Table 15 summarizes the MDL and RL data for wipes. The data shows that the required Be Filter Report Limit of 0.01 ug Be/filter and the Be Wipes Report Limit of 0.03 ug Be/wipes can be met using the new ion exchange method. The Method Detection Limit (MDL) was calculated by measuring the standard deviation of the measurement at 1 ppb Be and multiplying that value times the t-table value for 9 degrees of freedom. The Report Limit for the method was calculated by multiplying the MDL by a factor of five. The Report Limit per sample was calculated by multiplying the RL (ppb) by the 0.02L dilution volume.

The ion exchange steps using Diphonix Resin and TEVA Resin take about 1 to 1.5 hours for a batch of 20 samples. If the additional anion resin separation is required to lower Cr levels, an additional 1.5 hours of ion exchange time is required after about ~2 hour evaporation step. It is anticipated that the anion resin separation will only be used rarely, but it is available if needed. The filtering of the samples



through ion exchange also filters out any residual organic material which can clog the ICP-AES nebulizer. By performing the ion exchange accuracy and precision at very low levels of Be are very good, often with less errors associated with spectral interference corrections, which are effective but sometimes have large uncertainties associated with these corrections.

Samples may also be analyzed by the ICP-AES first to see whether ion exchange is required. If a 10 ml aliquot (out of an initial 20 ml digest volume) is later reprocessed for ion exchange removal of interferences, an additional evaporation step to lower the final volume after ion exchange back to 10 ml may be required to maintain a report limit of 0.01ug Be/sample or less.

The Diphonix-TEVA cartridge method may also be applied to sulfuric acid/hydrogen peroxide/nitric acid digestions used by some U.S laboratories after evaporation of this matrix.<sup>15</sup> The removal results are shown in Table 16. The removal results are very good, including the removal of vanadium. The titanium removal is not as efficient when the sulfuric acid matrix digestion is used, presumably due to sulfate interference on titanium retention on TEVA Resin. Sulfate ions are still present after evaporation due to the relatively high boiling point of sulfuric acid. By modifying the anion resin method described above so that the load and rinse solutions are collected for Be (since  $\text{SO}_4^-$  ion prevents Be retention on the anion resin), additional titanium removal was achieved using this modified anion resin method (Figure 4). This modified approach does not, however, remove additional Cr. Titanium levels may be low enough without the additional anion removal so this step may not be necessary. Table 17 shows Be recovery results using the 234.861

nm emission line for spiked filters and wipes with the sulfuric acid digestion and the Diphonix –TEVA Resin separation. The spiked filters and wipes are at the 0.02 ug/sample level (1 ppb at instrument). The average bias for 4 data points was -6.42 % with a relative standard deviation of 4.26%. These results show that the Cr levels (65-78 ppm Cr) and the Ti levels (10-18 ppm Ti) remaining after the Diphonix-TEVA Resin separation did not significantly affect the Be measurement using the 234.861 nm emission line.

## **Conclusions**

A new method has been developed to remove spectral interferences prior to beryllium (Be) measurement by inductively-coupled plasma atomic emission spectrometry (ICP-AES). The method is a simple, single pass method that effectively removes spectral interferences. The ion exchange separation removes uranium (U), thorium (Th), niobium (Nb), vanadium (V), molybdenum (Mo), zirconium (Zr), tungsten (W), iron (Fe), chromium (Cr), cerium (Ce), erbium (Er) and titanium (Ti). A stacked column consisting of Diphonix Resin and TEVA Resin reduces the levels of the spectral interferences so that accurate low level Be measurements can be performed. If necessary, an additional anion exchange separation can be used for further removal of interferences, particularly chromium. The method has been tested using spiked filters, spiked wipe samples and certified reference material standards with high levels of interferences added. The method provides a high level of removal for spectral interferences with very good accuracy and precision for beryllium. This

ion exchange method can be used with other acid digestion methods as well. The ion exchange method can also be applied to be assay by inductively-coupled plasma mass spectrometry (ICP-MS) to prevent introduction of high levels of metallic impurities into the ICP-MS.

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### **Table Captions**

Table 1	Spectral Interference Levels at the ICP-AES
Table 2	Average Removal Results for Spectral Interferences
Table 3	Be Recovery Results (234.861 nm line) - Interferences Added
Table 4	Be Recovery Results (313.042 nm line) - Interferences Added
Table 5	Be Recovery Results (313.107 nm line) - Interferences Added
Table 6	Additional Be Recovery Results (234.861 nm line)-Interferences Added
Table 7	Additional Be Recovery Results (313.042 nm)- Interferences Added
Table 8	Additional Be Recovery Results (313.107 nm)- Interferences Added
Table 9	BeO Filter Recovery Results (234.861 nm line)-Interferences Added
Table 10	Be Recovery Results- LCS Filters (313.107 nm)
Table 11	Be Recovery Results- LCS Wipes (313.107 nm)
Table 12	Be Recovery Results- Report Limit Filters (313.107 nm)
Table 13	Be Recovery Results- Report Limit Wipes (313.107 nm)
Table 14	Summary of MDL and RL Results for Beryllium Filter Method
Table 15	Summary of MDL and RL Results for Beryllium Wipes Method
Table 16	Average Removal Results for Spectral Interferences with Sulfuric Acid Digestion
Table 17	Be Recovery Results (313.107 nm)- Interferences Added-Sulfate Matrix

### **Figure Captions**

Fig.1 Sample Digestion to Prepare for Ion Exchange

Fig. 2 Ion Exchange Removal of Spectral Interferences

Fig. 3. Vacuum Box System with stacked Diphonix Resin and TEVA Cartridges

Figure 4 Ion Exchange Removal of Spectral Interferences-Sulfate Digestion

Table 1. Spectral Interferences Levels at the ICP-AES

Iron	1000	ug/ml
Uranium	300	ug/ml
Thorium	100	ug/ml
Chromium	100	ug/ml
Niobium	100	ug/ml
Titanium	100	ug/ml
Molybdenum	100	ug/ml
Zirconium	100	ug/ml
Tungsten	100	ug/ml
Erbium	100	ug/ml
Cerium	50	ug/ml

Table 2 Average Removal Results for Spectral Interferences

Interference	Added (ppm)	Measured (ppm)	Removal (%)
Iron*	1000	0.039	99.996
Uranium	300	0.112	99.963
Niobium	100	ND	~100
Molybdenum	100	0.002	99.998
Vanadium	100	0.003	99.997
Zirconium	100	0.082	99.918
Tungsten	100	0.010	99.990
Thorium	50	ND	~100
Titanium	100	0.736	99.964
Cerium	50	ND	~100
Erbium	100	ND	~100
Chromium	100	50.01	49.99
Chromium**	100	8.98	91.02

N=10

ND= none detected

Added ppm added to have this level interference in 20 ml at ICP-AES

\*Fe has similar removal at 2500 ppm

\*\*additional anion exchange to remove more Cr

Table 3 Be Recovery Results (234.861 nm)- Interferences Added

	Sample	Be Added (ppb)	Be Measured after IEX (ppb) (234.861 nm)	Recovery (%)	Bias (%)
1	wipe	1	1.128	112.80	12.80
2	wipe	1	1.151	115.10	15.10
3	wipe	1	1.10	110.00	10.00
4	wipe	1	1.08	108.00	8.00
5	wipe	1	1.096	109.60	9.60
6	wipe	1	1.14	114.00	14.00
7	Be filter	5	4.916	98.32	-1.68
8	Be filter	5	4.926	98.52	-1.48
9	BeO filter	25	22.8	91.20	-8.80
10	BeO filter	25	21.6	86.4	-13.60
			Avg. RSD	104.39 9.67%	4.39

Table 4 Be Recovery Results (313.042 nm)- Interferences Added

		Be Added (ppb)	Be Measured after IEX (ppb) 313.042 nm	Recovery (%)	Bias (%)	After Anion exchange	Recovery (%)	Bias (%)
1	wipe	1	1.41	141.00	41.00	1.01	101	1
2	wipe	1	1.23	123.00	23.00	1.13	113	13
3	wipe	1	1.47	147.00	47.00	0.95	95	-5
4	wipe	1	1.76	176.00	76.00	0.94	94	-6
5	wipe	1	1.57	157.00	57.00	0.98	98	-2
6	wipe	1	1.72	172.00	72.00	0.98	98	-2
7	Be filter	5	4.88	97.60	-2.40	4.84	96.8	-3.2
8	Be filter	5	5.4	108.00	8.00	4.74	94.8	-5.2
9	BeO filter	25	22.42	89.68	-10.32	22.75	91	-9
10	BeO filter	25	21.25	85	-15.00	21.07	84.28	-15.72
			Avg. %RSD	129.63 26.0 %	29.63		96.60 7.62 %	-3.41



Table 5 Be Recovery Results (313.107 nm)- Interferences Added

		Be Added (ppb)	Be Measured after IEX (ppb) 313.107 nm	Recovery (%)	Bias (%)	After Anion exchange	Recovery (%)	Bias (%)
1	wipe	1	1.77	177.00	77.00	1.314	131.4	31.4
2	wipe	1	1.23	123.00	23.00	1.289	128.9	28.9
3	wipe	1	1.74	174.00	74.00	1.14	114	14
4	wipe	1	1.76	176.00	76.00	1.18	118	18
5	wipe	1	1.79	179.00	79.00	1.19	119	19
6	wipe	1	1.83	183.00	83.00	1.15	115	15
7	Be filter	5	4.89	97.80	-2.20	5.02	100.4	0.4
8	Be filter	5	5.44	108.80	8.80	5.04	100.8	0.8
9	BeO filter	25	22.62	90.48	-9.52	22.7	90.8	-9.2
10	BeO filter	25	21.36	85.44	-14.56	21.1	84.4	-15.6
			Avg.	139.45	39.45		110.27	10.27
			%RSD	29.9 %			14.2%	

Table 6 Additional Be Recovery Results (234.861 nm)- Interferences Added

	Sample	Be Added (ppb)	Be Measured after IEX (ppb) (234.861 nm)	Recovery (%)	Bias (%)
1	wipe	1	1.16	116.00	16.00
2	wipe	1	1.12	112.00	12.00
3	wipe	1	1.02	102.00	2.00
4	wipe	1	1.03	103.00	3.00
5	wipe	1	0.92	92.00	-8.00
6	wipe	1	0.93	93.00	-7.00
7	Be filter	5	4.80	96.00	-4.00
8	Be filter	5	4.90	98.00	-2.00
9	Be filter	5	4.50	90.00	-10.00
10	Be filter	5	4.49	89.80	-10.20
			Avg.	99.180	-0.820
			%RSD	9.16	

Table 7 Additional Be Recovery Results (313.042 nm)- Interferences Added

		Be Added (ppb)	Be Measured after IEX (ppb) 313.042 nm	Recovery (%)	Bias (%)	After Anion exchange	Recovery (%)	Bias (%)
1	wipe	1	1.98	198.00	98.00	1.11	111	11
2	wipe	1	2.07	207.00	107.00	1.45	145	45
3	wipe	1	1.45	145.00	45.00	1.16	116	16
4	wipe	1	1.38	138.00	38.00	1.07	107	7
5	wipe	1	1.58	158.00	58.00	0.89	89	-11
6	wipe	1	1.55	155.00	55.00	0.99	99	-1
7	Be filter	5	5.44	108.80	8.80	5.18	103.6	3.6
8	Be filter	5	5.86	117.20	17.20	5.16	103.2	3.2
9	Be filter	5	5.17	103.40	3.400	4.75	95	-5
10	Be filter	5	5.04	100.8	0.800	4.77	95.4	-4.6
			Avg.	143.12	43.12		106.42	6.42
			%RSD	26.25			14.79	

Table 8 Additional Be Recovery Results (313.107 nm)- Interferences Added

		Be Added (ppb)	Be Measured after IEX (ppb) 313.107 nm	Recovery (%)	Bias (%)	After Anion exchange	Recovery (%)	Bias (%)
1	wipe	1	2.06	206.00	106.00	1.21	121	21
2	wipe	1	2	200.00	100.00	1.45	145	45
3	wipe	1	1.18	118.00	18.00	1.16	116	16
4	wipe	1	1.16	116.00	16.00	1.04	104	4
5	wipe	1	1.43	143.00	43.00	1.02	102	2
6	wipe	1	1.4	140.00	40.00	0.97	97	-3
7	Be filter	5	5.31	106.20	6.20	5.14	102.8	2.8
8	Be filter	5	5.49	109.80	9.80	5.1	102	2
9	Be filter	5	4.94	98.80	-1.20	4.66	93.2	-6.8
10	Be filter	5	4.99	99.8	-0.20	4.7	94	-6
			Avg.	133.76	33.76		107.7	7.7
			%RSD	29.49			14.69	

Table 9 BeO Filter Recovery Results (234.861 nm line) - Interferences Added

BeO CRM Filters		0.2 ug/filter			
		Be Added (ppb)	Be Measured after IEX (ppb) 234.861 nm	Recovery (%)	Bias (%)
1	BeO filter	10	8.35	83.50	-16.50
2	BeO filter	10	8.37	83.70	-16.30
3	BeO filter	10	8.44	84.40	-15.60
4	BeO filter	10	8.27	82.7	-17.30
			Avg.	83.58	-16.42
			%RSD	0.84	
BeO bias-No IEX			SRS F/H lab		-21.87
BeO bias-No IEX			SRS EBL Lab		-14.80

Table 10 Be Recovery Results- LCS Filters (313.107 nm)

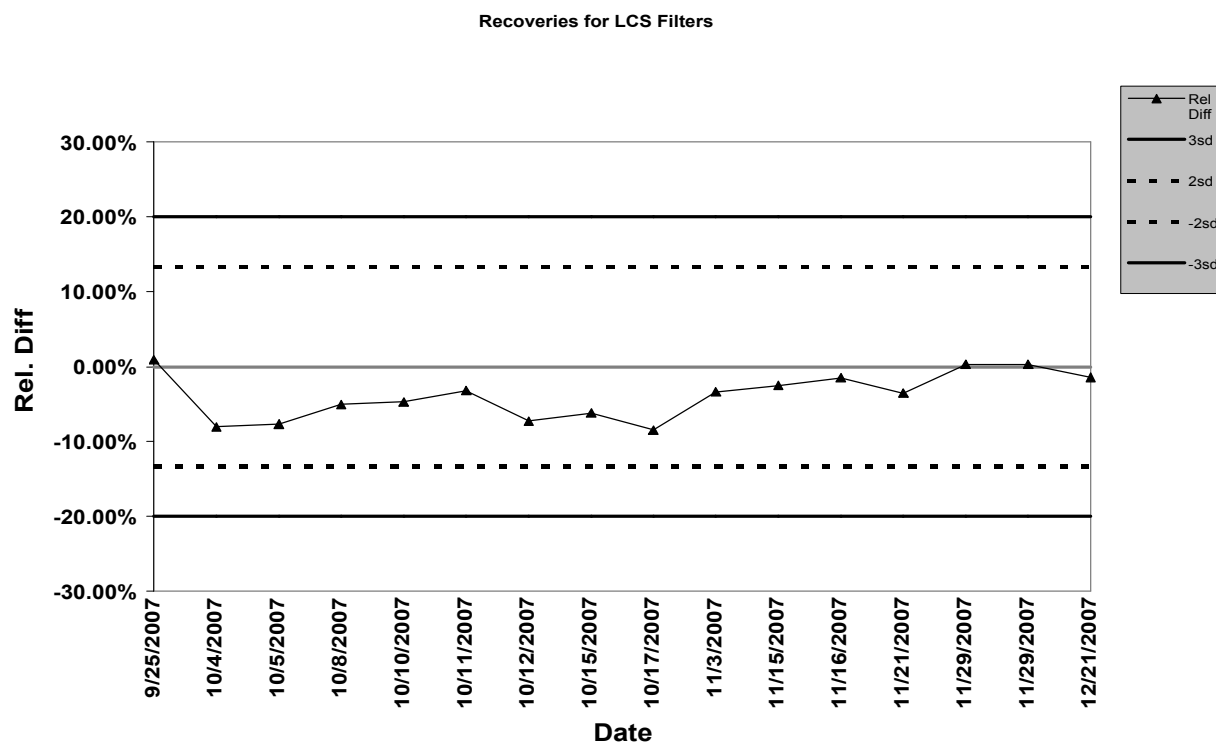


Table 11 Be Recovery Results- LCS Wipes (313.107 nm)

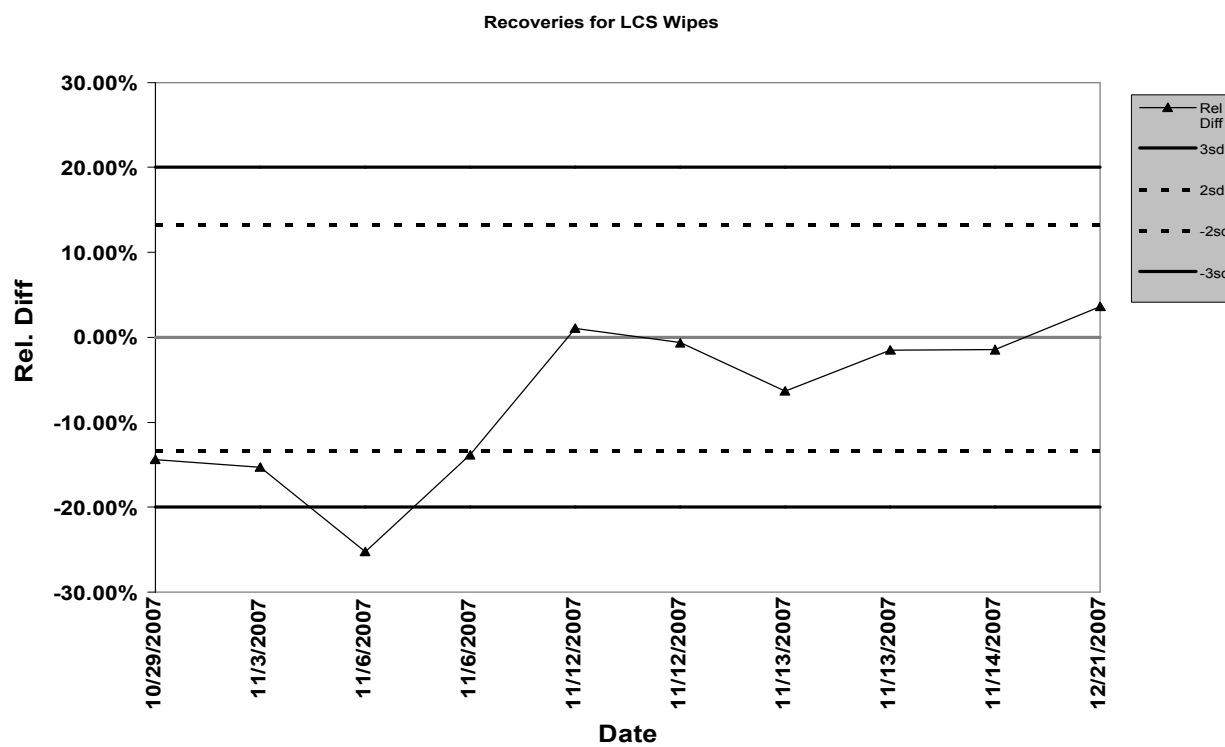


Table 12 Be Recovery Results- Report Limit Filters (313.107 nm)

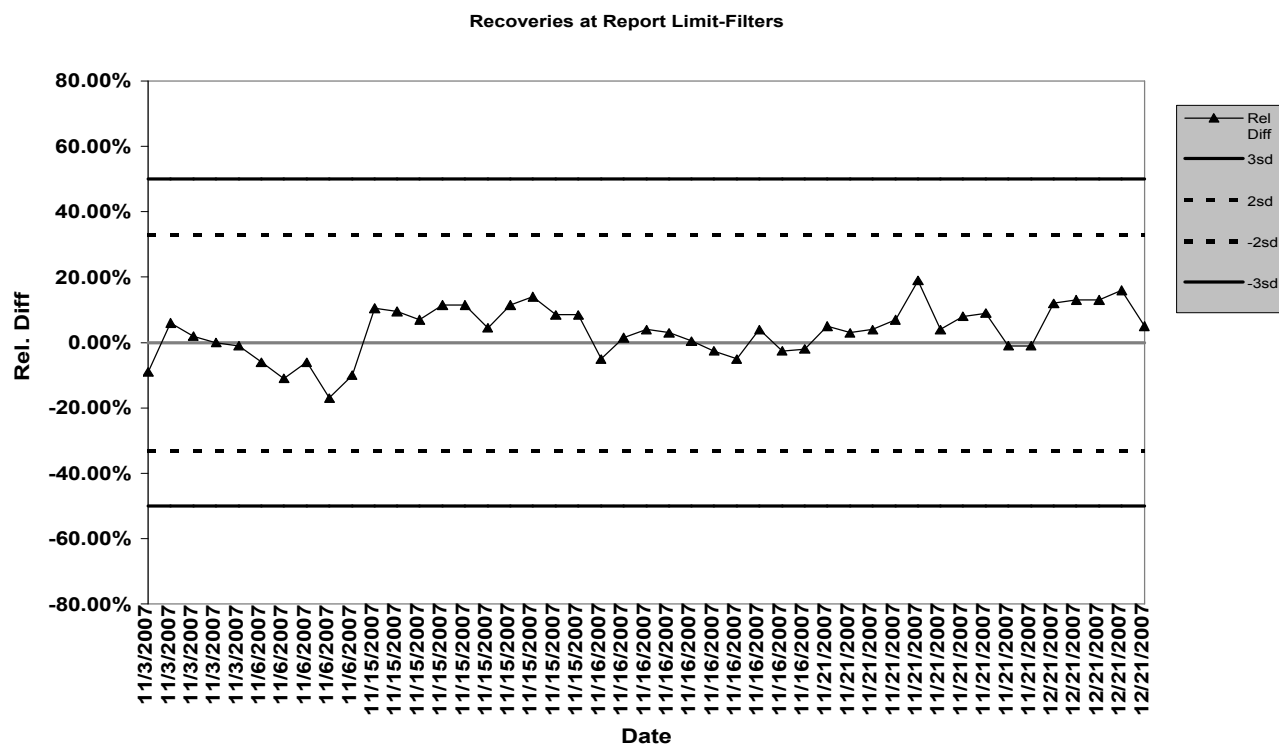


Table 13 Be Recovery Results- Report Limit Wipes (313.107 nm)

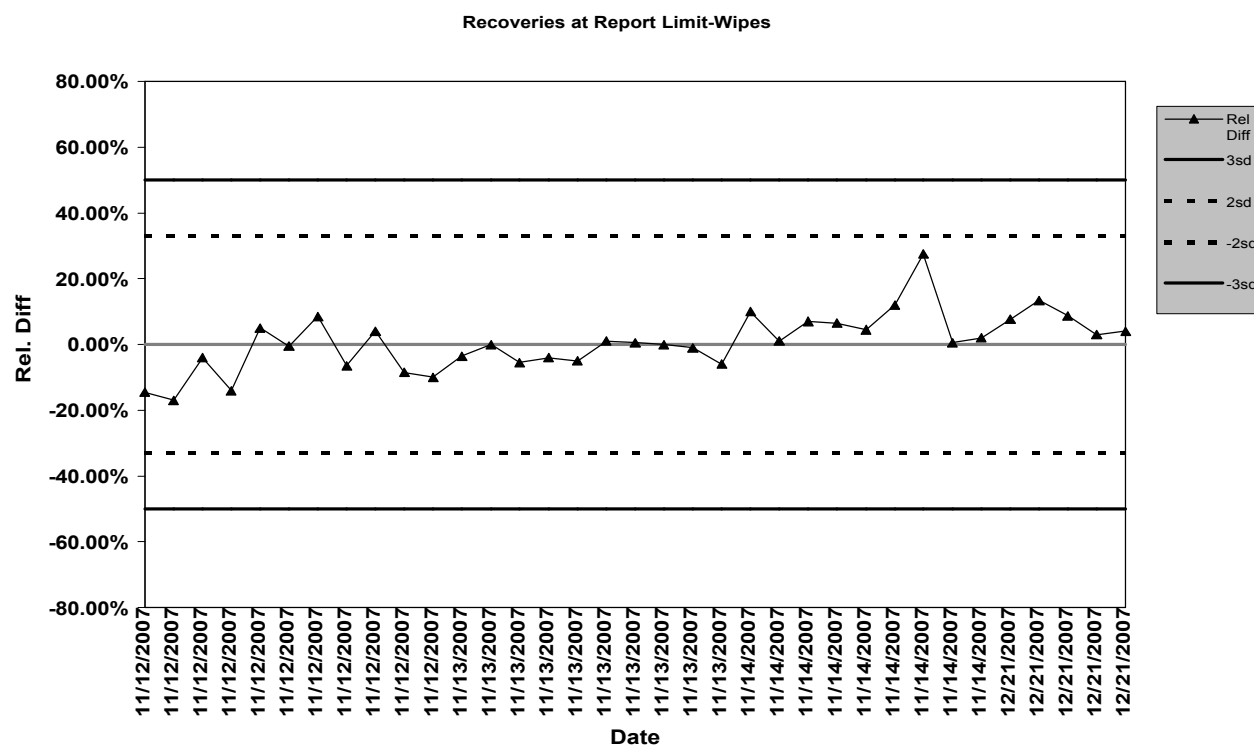


Table 14 Summary of MDL and RL Results for Beryllium Filter Method

Prepared on:	10/17/2007	10/17/2007	10/17/2007	11/15/2007	11/15/2007	11/15/2007	11/16/2007	11/16/2007	11/16/2007
Analyzed:	10/17/2007	10/17/2007	10/17/2007	11/15/2007	11/15/2007	11/15/2007	11/16/2007	11/16/2007	11/16/2007
ppb	1	1	1	1	1	1	1	1	1
	Be 234.861	Be 313.042	Be 313.107	Be 234.861	Be 313.042	Be 313.107	Be 234.861	Be 313.042	Be 313.107
1	1.086	1.032	0.983	1.416	0.857	1.106	1.128	0.947	0.952
2	1.082	1.043	1.021	1.430	0.816	1.094	1.112	1.106	1.016
3	1.113	0.995	0.951	1.438	0.771	1.068	1.168	1.020	1.040
4	1.178	1.104	1.068	1.433	0.816	1.114	1.096	1.003	1.030
5	1.133	1.093	1.059	1.459	0.827	1.114	1.147	1.001	1.005
6	1.099	1.092	1.007	1.460	0.809	1.047	1.094	0.964	0.975
7	1.128	1.106	1.056	1.535	0.827	1.115	1.096	0.977	0.951
8	1.136	1.090	1.044	1.560	0.848	1.139	1.168	1.031	1.040
9	1.102	1.115	1.038	1.430	0.717	1.084	1.088	0.960	0.973
10	1.164	1.065	1.034	1.503	0.783	1.086	1.153	0.979	0.979
AVERAGE	1.12206	1.07341	1.02602	1.46648	0.80707	1.09679	1.12479	0.99870	0.99605
STD DEV	0.031843	0.038831	0.036713	0.049504	0.040893	0.026412	0.031859	0.046317	0.034562
RSD% at 2*StdDev	5.68	7.24	7.16	6.75	10.13	4.82	5.66	9.28	6.94
%Recovery	112.206	107.341	102.602	146.648	80.707	109.679	112.479	99.87	99.605
MDL, ppb Be	0.090	0.110	0.104	0.140	0.115	0.075	0.090	0.131	0.097
Report Limit [MDL*5] , ppb Be	0.449	0.548	0.518	0.698	0.577	0.373	0.449	0.653	0.487
Report Limit [MDL*5*0.020L] , ug Be /filter	0.009	0.011	0.010	0.014	0.012	0.007	0.009	0.013	0.010



Table 15 Summary of MDL and RL Results for Beryllium Wipes Method

Prepared on:	11/13/2007	11/13/2007	11/13/2007	11/14/2007	11/14/2007	11/14/2007
Analyzed:	11/13/2007	11/13/2007	11/13/2007	11/14/2007	11/14/2007	11/14/2007
ppb	1	1	1	1	1	1
	Be 234.861	Be 313.042	Be 313.107	Be 234.861	Be 313.042	Be 313.107
1	1.003	1.033	0.964	1.088	1.089	1.099
2	1.000	1.076	1.001	1.033	0.977	1.012
3	0.957	0.997	0.944	1.058	1.054	1.068
4	0.977	1.016	0.960	1.081	1.047	1.068
5	0.928	0.980	0.953	No Sample	No Sample	No Sample
6	0.995	1.044	1.008	0.996	1.035	1.046
7	0.928	1.015	1.005	1.108	1.078	1.118
8	0.978	1.061	0.999	1.246	1.273	1.276
9	0.944	1.022	0.990	0.963	0.983	1.004
10	0.911	1.003	0.942	0.914	0.951	1.019
AVERAGE	0.96218	1.02472	0.97656	1.05410	1.05407	1.07881
STD DEV	0.033457	0.029333	0.026731	0.095662	0.094815	0.083318
RSD% at 2*StdDev	6.95	5.73	5.47	18.15	17.99	15.45
%Recovery	96.218	102.472	97.656	105.41	105.41	107.88
MDL, ppb Be	0.094	0.083	0.075	0.277	0.275	0.241
Report Limit [MDL*5], ppb Be	0.472	0.414	0.377	1.385	1.373	1.206
Report Limit [MDL*5*0.020L], ug Be /filter	0.009	0.008	0.008	0.028	0.027	0.024

Table 16 Average Removal Results for Spectral Interferences with Sulfuric Acid Digestion

Interference	Added (ppm)	Measured (ppm)	Removal (%)
Iron	1000	0.021	99.998
Uranium	100	0.262	99.74
Niobium	100	ND	~100
Molybdenum	100	0.027	99.973
Vanadium	100	ND	~100
Zirconium	100	3.795	96.205
Zirconium*	100	ND	~100
Tungsten	100	0.006	99.994
Thorium	50	ND	~100
Titanium	100	13.86	86.14
Titanium*	100	0.030	99.97
Cerium	50	ND	~100
Chromium	100	69.18	30.82

N=4

ND= none detected

Added ppm added to have this level interference in 20 ml at ICP-AES

\*additional anion exchange

Table 17 Be Recovery Results (234.861 nm)- Interferences Added-Sulfate Matrix

	Sample	Be Added (ppb)	Be Measured after IEX (ppb) (234.861 nm)	Recovery (%)	Bias (%)
1	filter	1	0.99	99.20	-0.80
2	filter	1	0.93	92.90	-7.10
3	wipe	1	0.92	92.40	-7.60
4	wipe	1	0.90	89.80	-10.20
			Avg.	93.575	-6.425
			% RSD	4.26	

Figure 1 Sample Digestion to Prepare for Ion Exchange

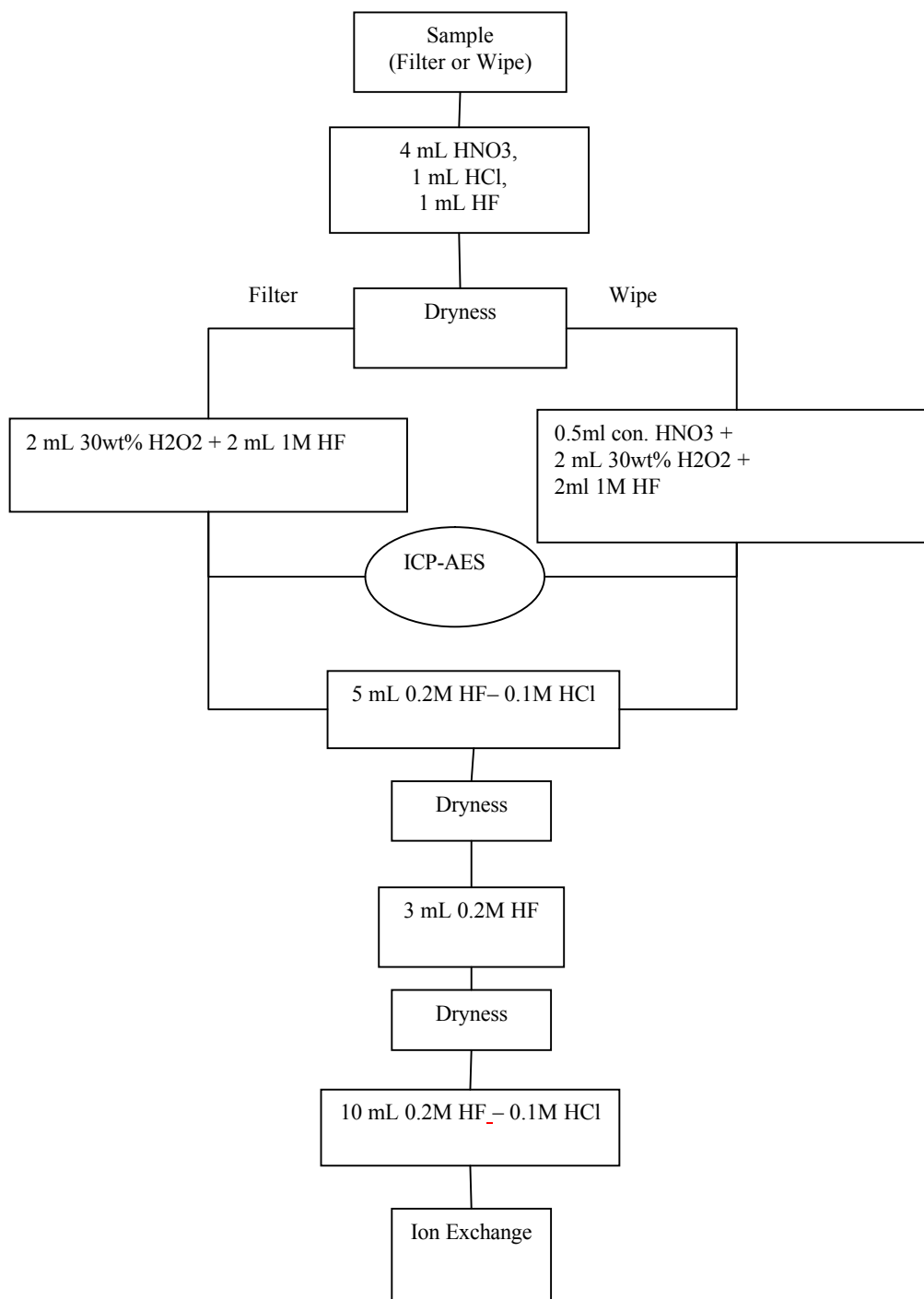


Figure 2 Ion Exchange Removal of Spectral Interferences

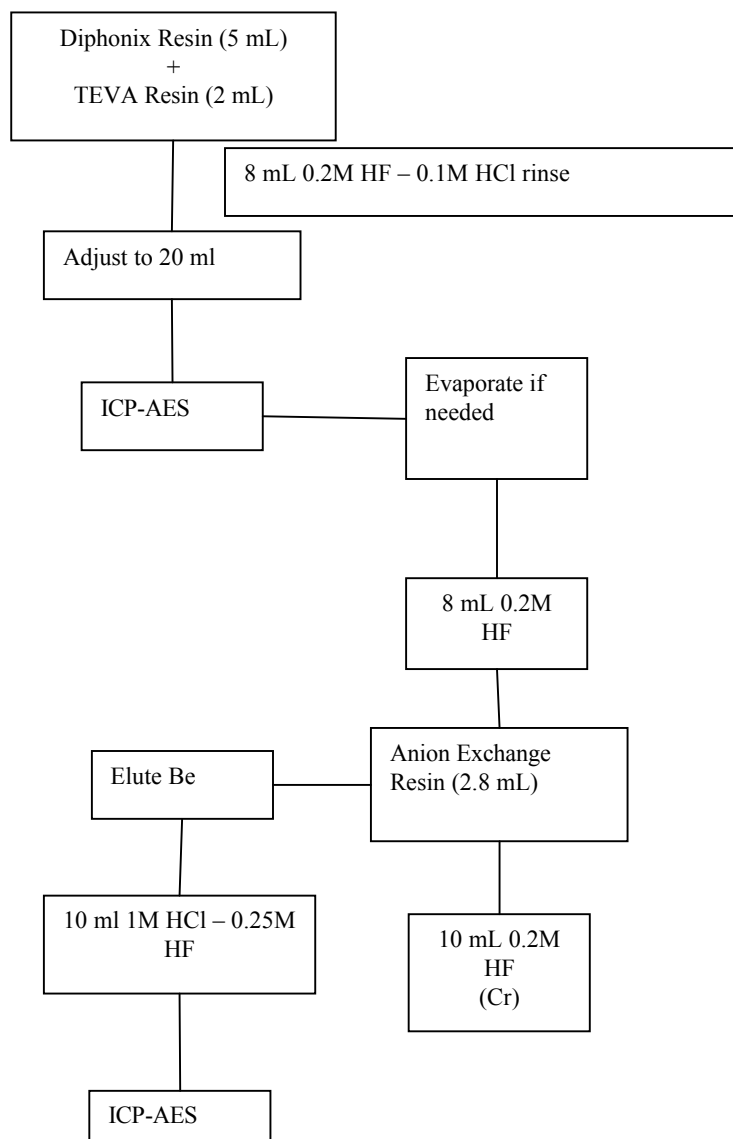


Figure 3 Vacuum Box System with Diphonix Resin and TEVA cartridges



Figure 4 Ion Exchange Removal of Spectral Interferences-Sulfate Digestion

