

Beryllium Analysis-Matrix Removal for more reliable ICP-AES analysis

Introduction: Beryllium metal, alloys and salts have been used widely in industry for decades. Beryllium is particularly valuable in specific areas of nuclear technology. Its ability to reflect neutrons and its efficiency in the production of neutrons when exposed to alpha emitters has led to its use in nuclear reactors and nuclear weapons. However, the use of Be carries with it some serious health hazards, the most serious of which is chronic beryllium disease (CBD). Chronic beryllium disease is caused by the inhalation of beryllium compounds, particularly the oxide, and scarring of the lung tissue. Because of the seriousness of CBD, the United States Department of Energy promulgated in 1999 the CBD prevention program, 10CFR Part 850, to protect DOE workers from exposure to beryllium contaminated dust.

The Problem: Due to the incidence of Chronic Beryllium Disease, analysis of surface wipes and air filters by ICP-AES is widely practiced at U.S. DOE sites to monitor potential exposure to beryllium. However, several elements, including uranium, vanadium, cerium, zirconium and niobium can interfere with the determination of beryllium by ICP-AES, especially at the low detection limits required under 10 CFR 850.

The Solution: A beryllium sample preparation method has been developed to remove all spectrally interfering elements and many other matrix elements from samples prior to analysis by ICP-AES. The method was developed for samples containing sufficient amounts of spectrally interfering elements that correction by ICP-AES software is unreliable. The method involves the use of a chelating diphosphonic acid extractant sorbed onto acrylic ester beads and is marketed under the trade name, Beryllium Resin. Figure 1 shows the separation of beryllium from a number of spectrally interfering elements using Beryllium Resin. It highlights the simplicity of the method in three steps;

- 1: Adjust the digested filter wipe to pH 1 to 2 with sodium acetate and load on a 2 mL Beryllium Resin cartridge,
- 2: Rinse with 0.2 M nitric acid, and
- 3: Strip beryllium with 4M nitric acid.

The three step method is compatible with any digestion technique. Should hydrofluoric acid be used, 0.1 M boric acid must be added to the load solution. Samples are processed in about an hour using a vacuum box which serves as a sample collector using 50 ml disposable centrifuge tubes.

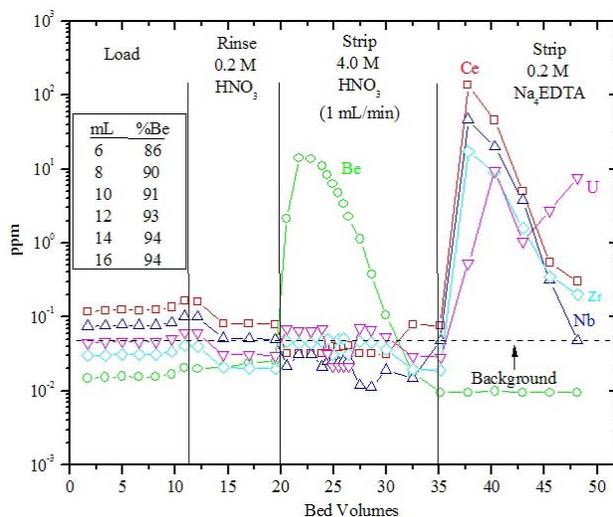


Figure 1. Elution of Be, Ce, U, Zr, and Nb on a 2 mL dry packed cartridge of Beryllium Resin, 50-100 μ m particle size, flow rate 2mL/min, 22(1) $^{\circ}$ C, Load: 5.5 cm ashless filter spiked with 140 μ g Be, Ce, U, Zr and Nb, digested with H_2SO_4 and H_2O_2 , diluted to 10mL with HNO_3 and neutralized to pH 1 with 3.4 M sodium acetate.

Data: The method has been tested at BWXT Y-12 on filter wipe samples which had yielded unacceptable results by ICP-AES measurement alone. Y-12 digests their samples with sulfuric acid and hydrogen peroxide. This is followed up by nitric acid. Analysis of samples containing between 75 and 500 ppm U of various enrichment all produced negative Be results. These dirty samples required re-testing by ICP-MS resulting in costly maintenance and turn around time issues.

These same samples run through the Beryllium Resin compared well with the ICP-MS results. **Yields ranged from 94 to 111%**, using the ICP-MS values as the standard. Beryllium values ranged from 0.0428 to 0.1452 micrograms per 100 square cm filter. By comparison, DOE has set 0.2 micrograms Be per 100 sq. cm as the limit for releasing equipment to non-beryllium areas.

Unlike U, vanadium tends to create a high bias. Two standards were created one containing 80 ppm vanadium and 0.001 ppm Be and the second containing 0.001 ppm Be, 80 ppm V, 400 ppm Cr, 800 ppm Fe, 2 ppm Sc and 100 ppm each of Mo, Nb, Th, Ti, U, Y, and Zr. Ten ml of 0.3 M oxalic acid was added to the samples. Virtually all interferences were removed. The results are shown in Table 1.

Table 1.

	Recovery w/ cartridge	Recovery w/o cartridge
Standard 1	99.9%	2010%
Standard 2	67.8%	4160%

Y-12 has implemented this method on samples showing negative Be or high U during an ICP-AES scan. Because of the consistency of the Beryllium Resin processed samples, no internal standard is necessary. The method is fast, reliable, robust and simple and can be used to concentrate beryllium. This provides ***a tool to address new lower measurement requirements being considered by ACGIH.*** Additionally, the method continues to be developed to look at the effect of even higher concentrations of interfering metals. Different resins can be paired with the Beryllium Resin to handle virtually any matrix combination.

References:

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