

Summary

The Savillex VC Ultra Acid Vapor Cleaning System is an automated system for cleaning microwave digestion vessels and other labware. This technical note assesses the effectiveness of the VC Ultra for the cleaning of microwave digestion vessels following the digestion of a sample (NIST 1575a Pine Needles) containing relatively high amounts of trace metals. Following cleaning, the vessels were run as blanks in the microwave, and the resulting blank solutions were analyzed by ICP-MS.

Using an automated 4-hour cleaning cycle, the VC Ultra demonstrated the ability to clean vessels that had been used to digest a sample with relatively high elemental content down to blank levels.



VC Ultra Acid Vapor Cleaning System

Methodology

Testing the cleaning efficiency of the VC Ultra, a sample containing relatively high levels of elements was digested in vessels before cleaning. A Standard Reference Material, NIST 1575a Trace Elements in Pine Needles, was chosen to represent a sample with several elemental concentrations up to several thousand ppm. Using the CEM MARS 6 microwave digestion system (CEM, Matthews, NC, USA) and CEM Xpress vessels (55 mL), CEM conducted a routine digestion of the sample. The sample was weighed into 20 vessels: 0.5g sample in each, along with 10 mL of reagent grade nitric acid. The vessels were capped, loaded into the MARS 6, and a digestion run performed using the Plant Material One Touch iWave method at 200°C. After the completion of the run, on cooling, the vessels were emptied, the digests discarded, and the vessels rinsed with DI water.

The vessels were then loaded into the VC Ultra on Xpress vessel racks for cleaning. The caps and insert plugs were placed on the slide-out trays inside the VC Ultra, above the vessel racks. Reagent grade nitric acid was added to the reservoir fill line (~950 mL). Note that high purity acid is not required for cleaning since the VC Ultra generates high purity acid vapor by sub-boiling distillation. The acid can also be reused multiple times. A standard 4-hour cleaning cycle was performed. The VC Ultra cycle was started at the end of the day: during the evening, the cycle was completed, and the VC Ultra shut down. The following morning, the vessels, caps, and inserts were removed and rinsed with DI water, and allowed to air dry. This time, 10 mL of distilled high purity grade nitric acid was added. The vessels were capped, loaded into the CEM MARS 6 and a blank digestion run performed using the Xpress One Touch iWave method at 150°C since no solid sample was present to be digested. After completing the run, on cooling, the blank digests were rinsed into standard trace labware containers and made up to 200 mL with DIW.

Results

The blank digests were run on a Thermo iCAP Q ICP-MS in KED mode. A 4 point calibration using a multielement CRM was used, and concentration values for the blank digests were generated. Table 1 shows blank digest concentration values for sixteen elements. Only potassium (1.35 ppb) and sodium (0.9 ppb) were at or above one ppb in the blank. The remaining elements were at ppt levels or below the reporting limit.

Element (Mode)	23Na (KED)	24Mg (KED)	39K (KED)	51V (KED)	52Cr (KED)	55Mn (KED)	57Fe (KED)	59Co (KED)
Concentration (ppb)	0.99	0.12	1.35	ND	0.03	ND	0.17	ND
Element (Mode)	60Ni (KED)	63Cu (KED)	68Zn (KED)	75As (KED)	88Sr (KED)	111Cd (KED)	136Ba (KED)	206Pb (KED)
Concentration (ppb)	0.02	ND	0.39	ND	0.01	ND	0.03	ND

Table 1: Elemental concentrations in the blank digests (after a VC Ultra cleaning cycle), by ICMS. Data courtesy of CEM, Matthews, NC, USA.

Elemental concentrations (certified and reference values) in NIST 1575a are shown in Table 2, along with equivalent concentrations in the digest both in ppm and ppb, along with blank concentrations in the vessels after cleaning for comparison.

NIST 1575a Pine Needles				
Element	Certified or Reference Value (ppm)	In Digest (ppm)	In Digest (ppb)	Vessel Blank After Cleaning (ppb)
Al	580	1.5	1450	
As	0.039	0.0	0.1	ND
Ba	6	0.0	15	0.03
Ca	25000	62.5	62500	
Cd	0.233	0.0	0.6	ND
Co	0.061	0.0	0.2	ND
Cu	2.8	0.0	7	ND
Fe	46	0.1	115	0.17
Hg	0.0399	0.0	0.1	
K	41700	104.3	104250	1.35
Na	63	0.2	158	0.99
Mn	488	1.2	1220	ND
P	10700	26.8	26750	
Pb	0.167	0.0	0.4	ND
Rb	16.5	0.0	41.25	
Zn	38	0.1	95	0.39

Table 2: Elemental concentrations shown in the NIST 1575a digest solution, with the vessel blanks after cleaning. (ND = not detected). Data courtesy of CEM, Matthews, NC, USA.

Conclusions

Given that the measurements were performed in a non-cleanroom lab, the concentrations obtained in the blanks (after the VC Ultra cleaning cycle) were all at or close to reporting limits, even though the NIST 1575a digest contained several elements at ppm levels. This demonstrates that the VC Ultra effectively cleaned vessels that had been used for relatively high-level digestions in a single 4-hour cleaning cycle. All data is courtesy of CEM, Matthews, NC, USA.



Savillex

10321 West 70th St. | Eden Prairie, MN 55344-3446 USA | Phone: 952.935.4100

Email: info@savillex.com | www.savillex.com