Application Note - Soil Digestion, EPA 3050B[1]

Introduction:

Throughout the world different strategies exist for contaminated soil digestion prior to ICP-AES analysis. One of these well documented methods is the United States of America's Environmental Protection Agency's (US-EPA) Method 3050B1. (see appendix 1)

The objective of this application note is to evaluate the EPA 3050B1 soil digestion technique. The samples were digested in a 48 position Graphite Block & Touch Screen Controller (Fig. 2, DigiPREP Digestion System) and an external PFA coated probe (DigiPROBE) to measure and accurately control the sample temperature.

A certified contaminated soil standard was selected: EnviroMAT SS-13 Certified Reference Material. SS-1 is a Type–1 naturally contaminated soil with a particle size of –200 mesh. It is designed for use as a quality control or method development control for analysis of soils by ICP-AES, ICP-MS, GFAA or AA Spectroscopy. The sample weight was maintained at 0.500g. In addition, the positions in the DigiPREP were randomly selected.

The samples were then analyzed using a Spectro Flame Sequential ICP-AES Spectrometer. A two-point calibration using a commercially available calibration standard (SCP SCIENCE, PlasmaCAL ICP-AES Calibration Standard) was created. Further correction for sample drift was made through the use of a standard-sample bracketing technique. This technique involves each sample being analyzed between a certified standard.

The Dixon Q-test4 was applied to each set of data to remove potential outliers and performed after confirming there was no connection between the outliers and the method of analysis. Due to the number of samples, the Student-t 4 was used to calculate the intervals of confidence and tolerance.

The EPA 3050B1 Sample Preparation Method is a soil leaching technique carried out at a sample temperature of 95oC. In this experiment, samples were placed in disposable 65 ml polypropylene digestion tubes which incorporate a Class "A" accurately calibrated, 50 ml volume marking that can be used to "fill to volume" in lieu of transferring the sample to a volumetric flask (fig.1). This method requires multiple additions of HNO3, HCl and H2O2.The samples were both weighed and digested in this tube. A ribbed polypropylene watch glass (010-500-019) was used to prevent sample cross-contamination and to promote refluxing during the digestion. DigiPROBE, as described above, was used to maintain the sample temperature at 95oC +/- 1oC.

For this study, sample 2 was randomly selected as the control sample.

Figure 1. Polypropylene Class "A" Graduated Digestion Vessel (SCP SCIENCE, DigiTUBE)

Figure 2. DigiPREP Digestion System (SCP SCIENCE, DigiPREP MS & Touch Screen Controller) with External Temperature Sensor (SCP SCIENCE, DigiPROBE)

DigiPREP

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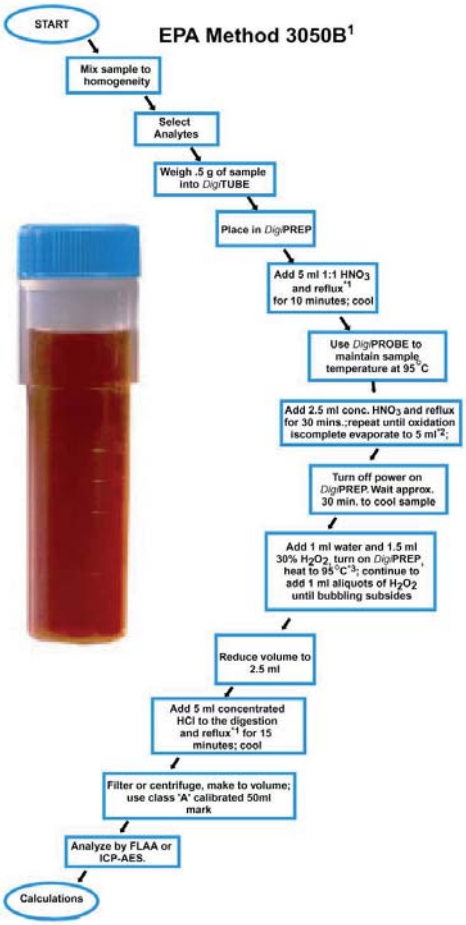
Sample Type:

Soil
Sample weight: 0.5 g

Supplies and Reagents:

1) 50 ml *Digi***TUBE** with watch glass
2) A 50 ml graduated Class A Polypropylene *Digi***TUBE**
3) *Digi***PROBE** (temperature control)
4) *Digi***PREP MS** & Touch Screen Controller
5) Ribbed polypropylene watch glass
6) A polypropylene funnel
7) Plastic Spatula
8) 4 Polypropylene Pipettes
9) Whatman 41 paper filter
10) Analytical Balance Melter AC-100
11) HCl (37%) - Baker Instrument - analyzed Reagent
12) H_2O_2 (30%) - Baker Ultrex II
13) HNO_3 (70%) - Baker Instrument - analyzed Reagent
14) Custom multi-element standard
15) SS-1

Sample Preparation Procedure:



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Results:

The digestions were completed, analyzed, and the results tabulated as follows:

Table 1 Precision of Recovery

Element Symbol	EPA Method 3050B Average (ppm)	EPA Method 3050B Standard Deviation (ppm)	EPA Method 3050B Relative Standard Deviation (ppm)
Al	9540	422	4.4%
Ca	135581	4191	3.1%
Cr	73	2	3.1%
Co	29	1	2.5%
Cu	707	17	2.4%
Cd	34	1	3.0%
Ba	106	4	4.1%
Li	12	0.4	3.0%
Na	216	8	3.7%
Mg	6492	183	2.8%
K	2190	77	3.5%
Mn	444	14	3.1%
Fe	23156	848	3.7%
Mo	9	1	16.3%
P	1042	29	2.8%
Ti	350	16	4.5%
V	19	1	4.5%
Ni	242	7	3.0%
Zn	7145	219	3.1%
Sr	202	5	2.3%
Pb	230	7	2.9%

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Table 2. Accuracy of Recovery

Element Symbol	EPA Method 3050B Average (ppm)	Reference Value (ppm)	Confidence Interval (ppm)
Al	9540	9518	8417-10619
Ca	135581	137375	131222-143528
Cr	73	64	55-73
Co	29	28	26-30
Cu	707	690	657-723
Cd	34	34	32-36
Ba	106	102	96-108
Li	12	11	9-13
Na	216	217	177-257
Mg	6492	6088	5710-6466
K	2190	1913	1553-2273
Mn	444	425	406-444
Fe	23156	20460	19037-21775
Mo	9	5	4.3-5.7
P	1042	1070	1021-1119
Ti	350	248	186-310
V	19	19	17-21
Ni	242	231	218-244
Zn	7145	6775	6467-7083
Sr	202	202	195-209
Pb	230	233	219-247

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Discussion

The data for the EPA 3050B1 method indicates that all elements, with the exception of Mo, are within 5% Relative Standard Deviation of one another. All the sample recoveries were higher than the certified value for Mo. A more detailed verification of data generated from the ICP-AES indicates the counts obtained with the Mo line at 313,259 varied greatly. Hence, it is recommended that a subsequent study should be undertaken using a different line for Mo.

Overall these results indicate that the Graphite Digestion Block provided a uniform digestion of the SS-1 sample. Data supporting the uniformity of temperature across the block was presented in a previous presentation5. The recovery data presented for each element compare very favorably to results obtained by the EPA in Table 2 of the EPA 3050B1 Method.

The results of the digested samples fell within the Confidence Interval of the SS-1 values for all the elements except, Mg, Fe, Mo, Ti, and Zn. The elements that fell outside the Confidence Interval all had higher recoveries than the certified values.

CONCLUSION:

The results generated from this study indicate that the DigiPREP is well suited for the EPA 3050B1 digestion.

REFERENCE:

1. United States Environmental Protection Agency Method 3050B-Acid Digestion of Sediments, Sludges, and Soils
2. European Standard-EN 13346 Characterization of Sludges and Sediments-Determination of trace elements and phosphorus-Aqua Regia extraction methods-Method B
3. SCP SCIENCE. Certificate of Analysis, Contaminated Soil Certified Reference Material SS-1
4. Peter C. Meier, Richard E. Zünd, Statistical Methods in Analytical Chemistry, 2nd Edition, Wiley Canada.
5. Art Ross, Jean-Pierre Zajac, The Benefits of Temperature Control with a Graphite Block Digestion System, (poster presented at FACSS, October 2002).

DigiPREP

MK-SOIL_DIGESTION-2.0-E

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