Application Note - The Digestion of SS-1

Introduction:

This study evaluated the effectiveness of the *DigiPREP Jr.* to digest a soil sample, **SCP SCIENCE** SS-1, as a reference material, following USEPA Method 3050b.

Sample Type:

Soil (SCP SCIENCE SS-1 lot SC0063618)

- Sample weight: 0.5 g.
- 4 replicates

Supplies and Reagents:

- 1) DigiPREP Jr. Graphite Block Digestion System*
- 2) DigiPREP Touch Screen Controller*
- 3) DigiPROBE*
- 4) DigiTUBEs, Polypropylene flat bottom tubes, 50 ml*
- 5) DigiFILTERs, 45 µm filter membrane*
- 6) Polypropylene watchglasses*
- 7) Nitric Acid, *Plasma***PURE** grade HNO₃ (70%)
- 8) Hydrochloric Acid, PlasmaPURE grade HCI (37%)
- 9) Hydrogen Peroxide, *PlasmaPURE* grade H₂O₂ (30%)
- 10) Spectro Cirros ICP-OES, Spectro Analytical
- 11) Cyclonic spraychamber*
- 12) Mini X-Flownebulizer*
- 13) Torch with 1.2mm alumina injector*
- 14) Pasteur pipettes
- 15) Rubber bulb
- 16) Analytical balance
- 17) Deionized water*
- 18) Graduated cylinder, Corning

Sample Preparation Procedure:

The sample was weighed directly in the Digitar Digit

The sample was then evaporated to 5 ml and subsequently removed from the block. After cooling to room temperature, 2 ml of deionized water was added and 3 ml of H_2O_2 was added dropwise while manually swirling the tube. The dropwise addition of H_2O_2 was continued until bubbling subsides or until 5 ml of $30\% \, H_2O_2$ had been added. The sample was placed backin the graphite block and heated at 95° C. The volume was then reduced to 5 ml (approx.3h). After cooling to temperature, the sample was normalized to 50 ml with deionized water, filtered with a *Digi*FILTER and analyzed on the ICP.



^{*}Manufactured by SCP SCIENCE.

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DigiPREP Jr Touch Screen Controlled Heating Program:

Stage	Ramp Time (min.)	Temperature (°C)	Hold Time (min.)
1	1	95	10
2	1	95	30
3	1	95	90

Recoveries:

<u>Recoveries:</u>							
Element	DigiPREP Jr. (ppm)	SD (ppm)	Certified Value (ppm)	Conf. interval (ppm)	Tol. Interval (ppm)		
Ag	0.91	0.08	0.88	0.85-0.91	0.72-1.04		
Al	12351	509	12163	11753-12572	9579-14746		
As	19.5	8.0	20.7	19.7-21.8	14.0-27.5		
В	17.1	1.2	26.9	18.5-35.2	0.0-77.8		
Ва	445	7	464	448-480	359-569		
Be	0.45	0.05	0.48	0.43-0.53	0.22-0.74		
Ca	50612	561	50265	49052-51478	42222-53308		
Cd	2.9	0.3	3.2	3.0-3.5	1.8-4.7		
Co	13.3	0.5	12.9	12.5-13.4	10.2-15.7		
Cr	108	8	103	97.9-109	66.6-140		
Cu	399	5	403	393-416	334-472		
Fe	72149	2253	72000	69728-74273	57212-86789		
Hg	0.43	0.06	0.41	0.39-0.43	0.29-0.53		
K	2091	123	2232	2082-2382	1257-3208		
Li	14.2	0.8	14.3	12.9-15.8	6.4-22.3		
Mg	9749	85	9690	9459-9920	8141-11239		
Mn	742	8	737	718-756	605-869		
Mo	7.9	1.4	6.8	6.5-7.2	4.7-9.0		
Na	639	15	650	587-714	235-1066		
Ni	59.4	1.5	59.2	57.9-60.5	50.4-68.0		
Р	1575	34	1552	1518-1589	1329-1775		
Pb	754	11	764	749-779	665-863		
Sb	6.7	1.3	5.5	4.4-6.6	0.0-12.0		
Se	0.86	0.09	0.78	0.64-0.92	0.02-1.54		
Sn	351	8	340	324-357	245-436		
Sr	114	4	114	113-116	106-122		
Ti	522	30	530	473-587	195-865		
V	28.0	1.8	27.2	25.9-28.6	18.8-35.7		
Zn	1140	20	1114	1078-1151	860-1369		



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Discussion/Conclusion:

Most digested elements are within the confidence interval for SS-1. The elements that fell outside of the Confidence Interval (As,B,Cd,Hg,Mo,Sb) are still within the Tolerance Interval. Therefore, the *DigiPREPJr*. is well suited for soil digestion.

It should be noted that, for Mo, the value is still within the Confidence Interval considering the standard deviation. As for B, the recovery is low due to the formation of the volatile boric acid under nitric acid conditions.

References:

USEPA Method 3050b SCP SCIENCE SS-1 certificate of analysis lot SC0063618, May 2010



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