



Application Note at Analysis, Inc. in Austin, TX

Digestion of Trace Metals in Soil Using DEENA - an Automated Sample Digestion System

In metal analysis, manual sample preparation often consists of many steps that are both labor intensive and time consuming. DEENA, an automated sample digestion system, alleviates most of the manual sample preparation and reduces the errors associated with sample handling.

The purpose of the application note is to demonstrate that metals in soil samples can be digested by EPA Method 3050 using DEENA.

EPA Method 3050 is a common method for determining trace metals in soils and solid waste. It involves multiple acid digestion steps followed by an organic removal step utilizing concentrated hydrogen peroxide.

Experiment

Standard soil samples (Environmental Resource Associates (ERA) Catalog # 540) were weighed and placed in 50ml disposable digestion vials (SCP Science). Due to the wide range of sample concentrations, several (500, 50, and 5mg) dilutions were prepared. The samples were placed in the rack on DEENA. A method was created in the software (Figure 1) with all the steps corresponding to EPA Method 3050. The block temperature was preset to 120°C. The reagents were DI water, nitric (HNO₃), hydrochloric acid (HCl), and hydrogen peroxide (H₂O₂). The nitric acid and hydrogen peroxide were added incrementally to avoid excessive foaming. The block temperature was set to high values in the method so that the sample achieved the desired temperature (95°C) in the allotted time. The final sample volume was 40 mL.

All steps were carried out by DEENA including adding the reagents, agitating (shaking) the samples, heating and cooling the samples, and filling to the final volume. Samples were then analyzed on a Thermo (TJA) Intrepid Inductively Coupled Plasma Optical Emission Spectrometer (ICP-OES).

Step #	Description	Reagent Name
1	Dispense 5 mL of DI Water	1 Nitric Acid CL304008
2	Dispense 5 mL of Nitric Acid CL304008	2 DI Water
3	Shake for 15 sec at 50 %	3 Hydrochloric Acid CL303489
4	Heat for 15 min at 120 oC	4 Hydrogen Peroxide CL303099
5	Cool for 15 min	5
6	Dispense 5 mL of Nitric Acid CL304008	6
7	Heat for 120 min at 114 oC	7
8	Cool for 15 min	8
9	Dispense 2 mL of DI Water	9
10	Dispense 1 mL of Hydrogen Peroxide CL303099	
11	Heat for 15 min at 113 oC	
12	Cool for 15 min	
13	Dispense 2 mL of Hydrogen Peroxide CL303099	
14	Heat for 105 min at 113 oC	
15	Cool for 15 min	
16	Dispense 5 mL of Hydrochloric Acid CL303489	
17	Heat for 5 min at 124 oC	
18	Cool for 15 min	
19	Dispense 5 mL of Hydrochloric Acid CL303489	
20	Heat for 10 min at 124 oC	
21	Heat for 0.1 min at 20 oC	
22	Cool for 15 min	
23	Fill to 40 mL with DI Water	

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Figure 1: Method in automated sample digestion software

Table 1: Recoveries of metals in soil samples

Element	Certified Value ¹	Performance Acceptance Limits ²		Measured Percent Recovery	Dilution
	(mg/kg)	Low %		%	
Aluminum	9120	53%	147%	120%	800X
Antimony	113	0%	211%	72%	800X
Arsenic	151	77%	123%	107%	8000X
Barium	272	82%	118%	112%	8000X
Boron	139	64%	136%	110%	800X
Cadmium	95.3	80%	120%	104%	800X
Calcium	6850	81%	119%	112%	800X
Chromium	120	81%	119%	114%	800X
Cobalt	67.3	81%	119%	107%	80X
Iron	16700	53%	146%	120%	8000X
Lead	120	78%	121%	108%	80X
Magnesium	2590	78%	122%	113%	800X
Molybdenum	40.8	79%	121%	109%	8000X
Nickel	102	79%	121%	96%	8000X
Selenium	138	78%	122%	112%	8000X
Silver	96.8	66%	133%	114%	800X
Sodium	382	64%	136%	99%	80X
Strontium	77.6	80%	120%	112%	800X
Thallium	170	78%	122%	111%	8000X
Tin	141	70%	130%	108%	80X
Vanadium	129	77%	123%	113%	800X
Zinc	117	79%	121%	100%	8000X

Results and Discussion

Once the samples were digested using DEENA, concentrations were determined for 22 elements in the soil samples using an ICP-OES. Recoveries (%) were calculated and listed in Table 1 along with the Performance Acceptance Limits (provided by ERA as guidelines for acceptable analytical results). As can be seen in Table 1, the measured recoveries (%) for half the elements are between 96% and 111%. And the measured recoveries of all the elements are well within the Performance Acceptance Limits.

1 “Certified Values are equal to the mean recoveries for each parameter as determined in an interlaboratory round robin study using the digestions and analytical techniques listed. All certified values are based on an “as received” basis assuming a 100% solids content” - Excerpt from ERA DataPack™

2 “The Performance Acceptance Limits (PALs™) are listed as guidelines for acceptable analytical results given the limitations of the USEPA methodologies commonly used to determine these parameters and closely approximate the 95% confidence interval. The PALs are based on data generated by your peer laboratories in ERA’s Interlab programs.” - Excerpt from ERA DataPack™



Conclusion



Real and difficult samples such as soil samples can be successfully digested by EPA Method 3050 using DEENA.

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About Thomas Cain

Thomas Cain, Inc. is dedicated and committed to helping analytical laboratories improve their efficiency and simplify their processes by automating their manually repetitive and time consuming tasks.

Our mission is to provide products and services which save time. By listening and identifying true needs, we innovate and provide solutions that have measurable value to our customers.



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