

Date: 09/04/2024	MA: 3225.0	Title: ICP-MS with Modified Preparation Method and Analysis of Soils with Additional Laboratory QC
Method Source: SFAM01.1	Method: ICP-MS	
Matrix: Soil/Sediment		
Summary of Modification		
The purpose of this modified analysis is to prepare samples by EPA Draft Method 3050C (see below) with additional modified LCS and Matrix Spikes and analyze for the scheduled target analytes by ICP-MS. Unless specifically modified by this modification, all analyses, Quality Control (QC), and reporting requirements specified in the SOW listed in your current EPA agreement remain unchanged and in full force and effect.		
I. Analyte Modifications		Not applicable ☒
II. Calibration and QC Requirements		Not applicable ☐
The Laboratory shall: • Use the Method Detection Limits (MDLs) determined for routine soil analyses (i.e., Method 200.8) to report the results for these analyses. The Laboratory is NOT required to perform an MDL study for Draft Method 3050C. • Prepare and analyze an additional Laboratory Control Sample (LCS) spiked at the CRQL. Percent Recovery limits do NOT apply to this LCS and no corrective actions are required. • Prepare a Matrix Spike spiked at three times the levels specified in the SOW. • Prepare and analyze an additional Matrix Spike sample spiked at five times the levels specified for this Modified Analysis (i.e., 15x the levels specified in the SOW). • Post-Digestion Spike requirements apply to the 5x Matrix Spike only. • Post-Digestion Spike corrective actions apply to Sb.		
III. Preparation and Method Modifications		Not applicable ☐
The Laboratory shall: • Prepare and analyze the sample by EPA Draft Method 3050C as follows: ○ Mix sample thoroughly and transfer 1.00 – 1.50 g to a digestion vessel. ○ Add 10 mL 1:1 HNO₃ and 5 mL 1:1 HCl, heat the sample at 95°C (±3°C) and reflux 10 -15 minutes. ○ Add 5 mL concentrated HNO₃ and reflux for 30 minutes at 95°C (±3°C), repeat until digestion complete. ○ Concentrate sample to 5 mL or reflux without boiling for 2 hours at 95°C (±3°C). ○ Cool sample, add 2mL water and 3 mL 30% H₂O₂. Heat at 95°C (±3°C) and add additional 1 mL aliquots of 30% H₂O₂ until effervescence is minimal. ○ Dilute to 100 mL with water, centrifuge or filter as necessary prior to analysis. • The same sample extracts can be used for ICP-AES analysis. Separate Matrix Spikes and LCS will need to be prepared for both ICP-AES and ICP-MS analyses. • Analyze the samples starting at an initial 5x dilution. Subsequently, dilute samples as necessary to bring the analyte concentrations within the calibration range of the instrument per the SOW. • Method Blanks, both LCSs, and all instrument QC are to be analyzed undiluted.		

IV. Special Reporting Requirements

Not applicable

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The Laboratory shall:

- Ensure the SDG Narrative is updated as stated in the SOW, including any technical and administrative problems encountered and the resolution or corrective actions taken. These problems may include interference problems encountered during analysis, dilutions, re-analyses and/or re-preparations performed, and problems with the analysis of samples. Also include a discussion of any SOW Modified Analyses, including a copy of the approved modification form with the SDG Narrative.
- Initial analysis data are reported with a dilution factor of 1.0 and a final volume of 500 mL, per the SOW.
- Report the additional LCS as "LCSD" in the raw data and in the EDD with QCType "Laboratory_Control_Sample_Duplicate".
- Report the additional Matrix Spike with an "SRE" suffix in the raw data and EDD.
- Report any Post-Digestion Spike of the additional 5x Matrix Spike with an "ARE" suffix.

Date: 09/04/2024	MA: 3226.0	Title: ICP-AES with Modified Preparation Method and Analysis of Soils with Additional Laboratory QC
Method Source: SFAM01.1	Method: ICP-AES	
Matrix: Soil/Sediment		
Summary of Modification		
The purpose of this modified analysis is to prepare samples by EPA Draft Method 3050C (see below) with additional modified LCS and Matrix Spikes and analyze for the scheduled target analytes by ICP-AES. Unless specifically modified by this modification, all analyses, Quality Control (QC), and reporting requirements specified in the SOW listed in your current EPA agreement remain unchanged and in full force and effect.		
I. Analyte Modifications		Not applicable ☒
II. Calibration and QC Requirements		Not applicable ☐
The Laboratory shall: • Use the Method Detection Limits determined for routine soil analyses (i.e., Method 3050B) to report the results for these analyses. The Laboratory is NOT required to perform an MDL study for Draft Method 3050C. • Prepare and analyze an additional Laboratory Control Sample (LCS) spiked at the CRQL. Percent Recovery limits do NOT apply to this LCS and no corrective actions are required. • Prepare a Matrix Spike spiked at two times the levels specified in the SOW. • Post-Digestion Spike requirements apply to the 2x Matrix Spike. • Post-Digestion Spike corrective actions apply to Sb.		
III. Preparation and Method Modifications		Not applicable ☐
The Laboratory shall: • Prepare and analyze the sample by EPA Draft Method 3050C as follows: ○ Mix sample thoroughly and transfer 1.00 – 1.50 g to a digestion vessel. ○ Add 10 mL 1:1 HNO₃ and 5 mL 1:1 HCl, heat the sample at 95°C (±3°C) and reflux 10 -15 minutes. ○ Add 5 mL concentrated HNO₃ and reflux for 30 minutes at 95°C (±3°C), repeat until digestion complete. ○ Concentrate sample to 5 mL or reflux without boiling for 2 hours at 95°C (±3°C). ○ Cool sample, add 2mL water and 3 mL 30% H₂O₂. Heat at 95°C (±3°C) and add additional 1 mL aliquots of 30% H₂O₂ until effervescence is minimal. ○ Dilute to 100 mL with water, centrifuge or filter as necessary prior to analysis. • The same sample extracts can also be used for ICP-MS analysis. Separate Matrix Spikes and LCS will need to be prepared for both ICP-AES and ICP-MS analyses. • Analyze the samples starting at an initial 2x dilution. Subsequently, dilute samples as necessary to bring the analyte concentrations within the calibration range of the instrument per the SOW. • Verify that the dilution was adequate to reduce interferents to within the method calibration range. This can optionally be verified by visual verification of the spectrogram or by analysis of a serial dilution. There are other acceptable means to provide assurance, e.g. some software may automatically provide guidance to the analyst. • Method Blanks, both LCS, and all instrument QC are to be analyzed undiluted.		

IV. Special Reporting Requirements	Not applicable ☐
The Laboratory shall: • Ensure the SDG Narrative is updated as stated in the SOW, including any technical and administrative problems encountered and the resolution or corrective actions taken. These problems may include interference problems encountered during analysis, dilutions, re-analyses and/or re-preparations performed, and problems with the analysis of samples. Also include a discussion of any SOW Modified Analyses, including a copy of the approved modification form with the SDG Narrative. • Initial analysis data are reported with a dilution factor of 2.0 and a final volume of 100 mL, per the SOW. • Report the additional LCS as "LCSD" in the raw data and in the EDD with QCType "Laboratory_Control_Sample_Duplicate". • Ensure that up-to-date Interelement Correction Factors (IECs) are provided with the data package.	

Element, Wavelength and Order	Use?	# IECs	IEC	k1	k2	Calc-in-fit?
As 189.042 {479}	☒	1	Fe	-0.000064	0.000000	No
Ti 190.856 {477}	☒	5	Mo	-0.002450	0.000000	No
			Co	0.002248	0.000000	No
			Ti	-0.000500	0.000000	No
			Mn	0.000370	0.000000	No
			V	-0.012340	0.000000	No
Pb 220.353 {453}	☒	6	Mo	-0.001480	0.000000	No
			Al	-0.000075	0.000000	No
			Cu	0.001400	0.000000	No
			Fe	0.000030	0.000000	No
			Mn	0.000340	0.000000	No
			Ni	0.000630	0.000000	No
Se 196.090 {472}	☒	3	Fe	-0.000308	0.000000	No
			Mn	0.000470	0.000000	No
			Co	-0.000630	0.000000	No
Sb 206.833 {463}	☒	4	Cr	0.010700	0.000000	No
			V	-0.001168	0.000000	No
			Mo	-0.002850	0.000000	No
			Ni	-0.000440	0.000000	No
Al 396.152 { 85}	☒	1	Mo	0.037230	0.000000	No
Ba 493.409 { 68}	☐	None				
Be 234.861 {144}	☒	3	Mo	-0.000320	0.000000	No
			Fe	0.000010	0.000000	No
			Mn	-0.000047	0.000000	No
Cd 214.438 {457}	☒	1	Fe	0.000040	0.000000	No
Ca 373.690 { 90}	☐	None				
Cr 267.716 {126}	☒	1	Mn	0.000160	0.000000	No
Co 228.616 {448}	☒	2	Ti	0.001840	0.000000	No
			Mo	-0.001230	0.000000	No
Cu 324.754 {104}	☒	4	Co	-0.000796	0.000000	No
			Fe	-0.000100	0.000000	No
			Mn	0.000345	0.000000	No
			Ni	0.000895	0.000000	No
Fe 259.837 {130}	☐	None				
Mn 257.610 {131}	☒	1	Ni	0.000897	0.000000	No
Mg 279.079 {121}	☐	None				
Ni 231.604 {446}	☐	None				
Ag 328.068 {103}	☒	3	Fe	-0.000100	0.000000	No
			Mn	0.000146	0.000000	No
			V	-0.000889	0.000000	No
Na 818.326 { 41}	☐	None				
V 292.402 {115}	☒	2	Mo	-0.008480	0.000000	No
			Cr	-0.002220	0.000000	No
Zn 206.200 {464}	☐	None				
Zn 213.856 {158}	☒	1	Ni	0.007280	0.000000	No
K 769.896 { 44}	☐	None				
P 177.495 {490}	☒	2	Ni	0.001640	0.000000	No
			Cu	-0.012530	0.000000	No
B 249.678 {135}	☒	3	Co	0.002880	0.000000	No
			V	-0.002000	0.000000	No
			Fe	-0.001360	0.000000	No
Mo 202.030 {467}	☐	None				
S 182.034 {485}	☒	2	Mo	-0.008000	0.000000	No
			Mn	0.002700	0.000000	No

Element, Wavelength and Order	Use?	# IECs	IEC	k1	k2	Calc-In-fit?
Si 251.611 {134}	☒	2	Mo	0.010520	0.000000	No
			Ti	0.005650	0.000000	No
Sn 189.989 {478}	☐	None				
Ti 336.121 {100}	☒	1	Ni	-0.001000	0.000000	No
Li 670.784 { 50}	☐	None				
Y 224.306 {450}*	☐	None				
Y 360.073 { 94}*	☐	None				
Y 371.030 { 91}*	☐	None				
Y 224.306 {150}*	☐	None				
In 230.606 {446}*	☐	None				
Sr 407.771 { 83}	☐	None				