

Trace Elements by ICP- Mass Spectrometry
EPA 200.8 Revision 5.4 **Page 1 of 3**

Facility Name: _____ VELAP ID _____
 Assessor Name: _____ Analyst Name: _____ Inspection Date _____

Relevant Aspect of Standards	Method Reference	Y	N	N/A	Comments
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Records Examined: SOP Number/ Revision/ Date _____ Analyst: _____
 Sample ID: _____ Date of Sample Preparation: _____ Date of Analysis: _____

If determining dissolved elements, are samples filtered through 0.45 µm pore diameter member filters and then acidified to a pH of <2 with nitric acid ?	8.2				
If determining total recoverable elements in aqueous samples, are samples acidified with nitric acid to a pH of <2 within two weeks of collection?	8.3				
Are samples mixed after preservation, held for at least 16 hours, and the pH rechecked just prior to analysis? If pH has increased to pH>2, additional acid must be added, and samples must be held for another 16 hours and rechecked. <i>ONLY ENFORCED FOR DRINKING WATER per CFR.</i>	8.3, 40 CFR 141.23 k.1				
Are solid samples stored at 4°C until time of analysis?	8.4				
Is the linear dynamic range of the instrument established?	9.2.2				
Is the mean of three analyses of the quality control sample within 10% of the stated value or within the acceptance limits in Table 8 quarterly, whichever is greater?	9.2.3, 7.8				
Is the MDL determined for all analytes annually, for new operators, and after significant hardware changes?	9.2.4				
Are laboratory reagent blanks analyzed with batches of 20 or fewer samples and carried through the entire preparation?	9.3.1, 7.6.2				
Are laboratory fortified blanks analyzed at least once with each batch and verified to have a recovery of 85-115%?	9.3.2, 7.9				
Are the calibration blank and calibration standards analyzed immediately following calibration, after every ten samples, and at the end of the run? Recovery of all analytes must be within ±10% of the calibration.	9.3.4				
Is a laboratory fortified matrix added to at least 10% of routine samples, with an acceptable recovery of 70-130%?	9.4.2, 9.4.3				
Is the instrument warmed up for at least 30 minutes?	10.2.1				

Notes/Comments:

Trace Metals by ICP- Mass Spectrometry
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Relevant Aspect of Standards	Method Reference	Y	N	N/A	Comments
Are mass calibration and resolution checks conducted using tuning solution during warm-up? Mass calibration is adjusted if it has shifted by more than 0.1amu from unit mass.	10.2.1				
For full mass range scans, are a minimum of three internal standards used for internal standardization? These must be present in all samples, standards, and blanks at identical levels.	10.3				
Does the calibration include a calibration blank and two standards prepared with 1% (v/v) nitric acid?	10.4, 7.4.1				
Does rinse time allow for adequate flushing between samples, and are solutions aspirated for 30 seconds prior to data acquisition to allow equilibration?	10.5, 4.1.5, 11.4.6				
For dissolved analytes in ground and surface water, do acid-preserved samples have an appropriate amount of (1+1) nitric acid added to approximate a 1% (v/v) nitric acid solution? [0.4 mL (1+1) HNO ₃ to 20 mL sample.]	11.1.1				
For direct analysis of total recoverable analytes in drinking water, do unfiltered acid-preserved samples have an appropriate amount of (1+1) nitric acid added to approximate a 1% (v/v) nitric acid solution? [0.4 mL (1+1) HNO ₃ to 20 mL sample.]	11.2.1, 11.1.1				
For total recoverable analytes or for preconcentrating drinking water samples prior to analysis, is 2 mL (1+1) HNO ₃ and 1mL (1+1) hydrochloric acid added to 100 mL sample?	11.2.3				
Are sample aliquots evaporated to about 20 mL at a temperature no higher than 85°C?	11.2.3, 11.2.4				
Are samples covered with watch glasses and refluxed for an additional thirty minutes?	11.2.5				
Are samples cooled, diluted to 50 mL, and mixed?	11.2.6				

Notes/Comments:

**Trace Elements in Wastes and Waters by ICP-Mass Spec
EPA 200.8 Revision 5.4**

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For solids, are samples dried to a constant weight at 60°C, in order to calculate percent solids?	11.3.1				
Is 4 mL of (1+1) HNO ₃ and 10 mL of (1+4) HCl added to 1.0 ± 0.01g of dried sample?	11.3.2, 11.3.3				
Are samples covered with a watch glass and refluxed at approximately 95°C for thirty minutes? (Constricted volumetric digestion tubes may alternatively be used on a block digester.)	11.3.3, 11.3.4				
Are samples cooled, diluted to 100 mL, and mixed?	11.3.5				
Is 20 mL of the digested sample transferred to a 100 mL volumetric flask and diluted to volume with reagent water?	11.3.7				
Are samples higher than the linear dynamic range diluted and reanalyzed?	11.4.7				

Notes/Comments: