

**ACID DIGESTION OF WATERS FOR TOTAL RECOVERABLE  
METALS  
(following EPA METHOD 3005)**

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Approved by

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## 1. SCOPE AND SUMMARY

This method is an acid digestion procedure used to prepare surface and ground water samples for analysis by inductively coupled argon plasma mass spectroscopy (ICP-MS) with reaction cell. The entire sample is acidified at the time of collection with nitric acid. At the time of analysis the sample is heated with acid and reduced in volume. The digestate is filtered and diluted to volume, and is then ready for analysis.

## 2. SAMPLE PREPARATION

### *2.1. INTERFERENCES*

The analyst should be cautioned that this digestion procedure may not be sufficiently vigorous to destroy some metal complexes. Precipitation will cause a lowering of the silver concentration and therefore an inaccurate analysis. High temperatures leading to a boiling of samples will vaporize some volatile analytes like Sb and lead to inaccurate concentration therefore temperatures should be hold below 95 °C.

### *2.2. APPARATUS AND MATERIALS*

All apparatus and bottles are pre-cleaned (see paragraph 2.3).

- a) 50 mL polypropylene centrifuge tubes pre-cleaned
- b) Plastic watch glasses.
- c) Disposable syringe filter (0.45 µm)
- d) Disposable 60 mL syringe with luer lock
- e) Volumetric flasks (polypropylene) 50 and 100 mL
- f) Mod Block with automatic temperature control to maintain temperature of 90-95° C.

### *2.3. REAGENTS*

High purity chemicals shall be used in all tests. All reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

- a) Reagent Water: For all digestion procedures, dilution of acids, and final step of cleaning procedures, water with purity higher than 18 MΩ is used. Reagent water shall be interference free.
  - b) Nitric acid\* (concentrated): OmniTrace Ultra HNO<sub>3</sub> 67-70% (EM-NX0408-7) is used for acidification and preparation of standards and blank solution.
  - c) Hydrochloric acid\* (concentrated), OmniTrace Ultra HCL (EM-HX0608-7) 33-36% is used for acidification and preparation of standards and blank solution.
- \*) Reagent grade acids are used for cleaning

### *2.3. CLEANING PROCEDURE*

- a) Soak in soap water (Micro ®)
- b) Rinse with tap water until all soap is removed

- c) Rinse with distilled water
- d) Fill bottles and flasks with 3% HCl, sonicate at 40 °C for 30 minutes
- e) Rinse with glass distilled water (3x)
- f) Fill bottles and flasks with 3% HNO<sub>3</sub> sonicate at 40 °C for 30 minutes
- g) Rinse with glass distilled water (3x)
- h) Rinse with 18 MΩ water (3x)
- i) Dry in drying oven at 50 °C.

#### 2.4. SAMPLE COLLECTION, PRESERVATION, AND HANDLING

All samples must have been collected using a sampling plan that complies with considerations discussed in Chapter Nine of EPA sampling manual of SW 846 online (<http://www.epa.gov/SW-846/main.htm#table>). All sample containers must be pre-washed with detergents, acids, and water. Only HDPE containers are suitable for trace metals. All samples must be acidified at the time of collection with concentrated HNO<sub>3</sub> omnitrace ultra ® (5 mL/L= 300µL for 60 mL or 625 µL for 125 mL bottles).

#### 2.5. PROCEDURE

Transfer a 50-mL aliquot of well-mixed acidified sample to a 50 mL centrifuge tube and place into Mod Block. Add 2 mL of concentrated HNO<sub>3</sub> and 1mL of concentrated HCl. The sample is covered with a ribbed watch glass (plastic) and heated at 90° C until the volume has been reduced to 30 mL. **CAUTION:** Do not boil.

Turn off the Mod Block and allow cooling. Wash down the tube walls and watch glass with reagent water (18 MΩ). Filter sample into 50 mL volumetric flask to remove silicates and other insoluble material that could clog the nebulizer. Use pre-rinsed 0.45 µm filter attached to disposable 60 mL syringe. Rinse syringe and filter with reagent water into flask. Adjust the final volume to 50 mL with reagent water.

#### 2.6. QUALITY CONTROL

For each analytical batch of samples processed, *blanks* should be carried throughout the entire sample preparation and analytical process. These blanks will be useful in determining if samples are being contaminated.

*Replicate samples* should be processed on a routine basis. A replicate sample is a sample brought through the whole sample preparation and analytical process. Replicate samples will be used to determine precision. The sample load will dictate the frequency, but 5% is recommended.

*Spiked samples* or standard reference materials should be employed to determine accuracy. A spiked sample should be included with each batch.

### 3. METHOD PERFORMANCE

Based on EPA method 200.8, the analytical methods described here allow for the quantification of total elements in samples such as ground waters, surface waters, and drinking water. Total recoverable elements in waste waters, sludges, and soils may also be determined. Analysis of appropriately prepared samples is conducted by ICP-MS. The analytes shown in Table I constitute the usual suite of trace elements included in method 200.8 analyses; however, additional non-trace elements (such as calcium, iron, magnesium, potassium, sodium) may be determined in aqueous matrices as appropriate.

Table I. Typical suite of elements determined by EPA Method 200.8.

	Element Name	Element Symbol	LOD ppb
<b>Trace Elements EPA 200.8 Validated</b>	Aluminum	Al	0.8
	Antimony	Sb	1.5
	Arsenic	As	0.5
	Barium	Ba	0.7
	Beryllium	Be	0.04
	Cadmium	Cd	0.5
	Chromium	Cr	0.8
	Cobalt	Co	2.0
	Copper	Cu	1.5
	Lead	Pb	0.4
	Manganese	Mn	1.4
	Mercury	Hg	
	Molybdenum	Mo	0.8
	Nickel	Ni	0.8
	Selenium	Se	0.8
	Silver	Ag	0.8
	Thallium	Tl	1.0
	Thorium	Th	0.8
	Uranium	U	0.8
Vanadium	V	1.0	
Zinc	Zn	0.7	
<b>Non-Trace Elements</b>	Calcium	Ca	70
	Iron	Fe	20
	Magnesium	Mg	20
	Potassium	K	30
	Sodium	Na	26
	Silicium	Si	

### 3.1 SOLUTIONS AND STANDARDS

All solutions prepared for use in this method must be stored in polyethylene containers – the use of glassware is not acceptable. The useful lifetime of these solutions is no more than about one week.

- Analytical grade deionized water (DI H<sub>2</sub>O)*. Must have a resistivity of 17.5–18.5 MΩ/cm.
- Diluent solution*. Must contain 1.0 % concentrated HNO<sub>3</sub> by volume. Combine 100 mL trace metals analysis grade concentrated HNO<sub>3</sub> in several liters of DI H<sub>2</sub>O, and bring to a total final volume of 10 L with DI H<sub>2</sub>O. Mix thoroughly.
- ICP-MS tuning solution*. Contains 10 ppb Li, Y, Ce, Tl, and Co for instrument tuning and verification of performance. Mixed from single element standards of 1000 ppm concentration: *Preparation of stock solution*: Pipette 1 mL of each element solution into 100 mL volumetric flask and fill with 1% HNO<sub>3</sub> diluent. *Preparation of tuning solution*: Pipette 1 mL of stock solution in 1000 mL flask and fill with 1% HNO<sub>3</sub> diluent.
- Multi-element primary standard mixture*. Contains Fe, K, Ca, Na, and Mg at 1000 ppm and Ag, Al, As, Ba, Be, Cd, Co, Cr, Cu, Mn, Mo, Ni, Pb, Sb, Se, Tl, V, Zn, Th, and U at 10 ppm. Available from Agilent, item number 5183-4688. Standards for Si and P are added as single element standards available from Specs if needed.

- e) *Single-element primary standards.* If elements in addition to those contained in the multi-element primary standard are to be determined, working standard solutions may be supplemented with single-element standards to produce the desired concentrations. Single-element standards of suitable quality for plasma methods are available from a variety of sources.
- f) *Internal standards (ISTD) primary standard mixture.* Contains Li, Sc, Ge, Y, In, Tb, and Bi at 10 ppm for use as internal standards. Available from Agilent, item number 5183-4680.
- g) *Working ISTD solution.* To prepare the working internal standards solution (1 ppm each IS), add 10 mL IS primary standard mixture to approximately 50 mL diluent solution and dilute to a total final volume of 100 mL with diluent solution.
- h) *Erbium primary standard.* Contains Er at 1 ppm for use as a dilution standard. Available from Agilent, item number G1820-60372.
- i) *Working erbium solution.* To prepare the working dilution standard solution (50 ppb Er), add 12.5 mL 1 ppm Er to approximately 100 mL diluent solution and dilute to a total final volume of 250 mL with diluent solution.
- j) *Calibration standards.* To prepare initial calibration (ICAL) solutions, specific volumes of the multi-element primary standard mixture are diluted with diluent solution. The required volumes of multi-element primary standard mixture for preparation of a standard set of ICAL solutions is given below (Table II), followed by the resultant concentration of all analytes at each calibration level (Table III). If additional elements are desired, single-element standards must be used to supplement the preparations described below. In order to obtain the concentrations listed in Table III, the total final volume of each solution must be 250.0 mL. If any of the elements are expected to exceed the calibration range given below, additional levels may be used in order to span the necessary range of concentrations using the multi-element or single-element standards as appropriate.

Table II. Preparation of ICAL standards.

Level	Volume, primary standard mixture ( $\mu\text{L}$ )
1	0.0
2	5.0
3	10.0
4	50.0
5	100.0
6	500.0
7	1000.0

Table III. Typical ICAL Levels.

Analyte(s)	Concentration (ppb) of Analyte at ICAL Level:						
	1	2	3	4	5	6	7
Fe, K, Ca, Na, Mg, Si	Blank	50	100	500.0	1,000.0	5,000.0	10,000
Ag, Al, As, Ba, Be, Cd, Co, Cr, Cu, Hg, Mn, Mo, Ni, Pb, Sb, Se, Tl, V, Zn, Th, and U	Blank	0.5	1.0	5	10.0	50.0	100.0

### 3.2 DAILY PROCEDURES FOR INSTRUMENT SETUP

Prior to operating the ICP-MS, the following steps must be performed on a daily basis. These steps assume that the instrument hardware and software is configured for the 200.8 ICP-MS application. If the configuration is set for another application (i.e., LC-ICP-MS), see Appendix A for detailed instructions regarding setup of the hardware and software for the 200.8 ICP-MS application.

- a) Replace all peristaltic pump tubing. When ready for analysis, clamp the tubing in place on the pumps. Place the lines for uptake of diluent and internal standard solutions into the appropriate vessels (refer to Appendix B for a detailed diagram of the tubing configuration).
- b) Ensure that there is sufficient argon and helium supply.
- c) Verify that the hydrogen generator is functioning properly.
- d) Turn the chiller unit on prior to igniting the plasma.
- e) Verify that all waste lines are in place, and empty waste reservoirs if needed.
- f) Verify that ventilation is operating properly prior to igniting the plasma.
- g) Perform a pulse to analog (P/A) factor tuning for each tune step as follows:

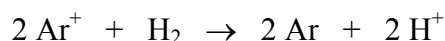
### 3.3 ANALYSIS

Sample analysis is carried out using an Agilent Technologies 7500c ICP-MS equipped with the octopole reaction system. The reaction cell allows a near-vacuum interface between the plasma and the mass spectrometer, or may alternatively be flooded with a reaction or collision gas in order to minimize various interferences. In order to minimize known possible interferences, each element is acquired in one of three segments, or tune steps, of an analysis: no reaction gas (normal mode), He collision, or H<sub>2</sub> reaction.

Elements occurring at mass to charge ratios (m/z) with no anticipated plasma-based or matrix-based interferences are acquired in normal mode. In general, these include the relatively light and relatively heavy elements.

Any element occurring at an m/z with a known matrix-based interference would be acquired in He collision mode. Polyatomic interferences occurring in the plasma as a result of matrix composition will be obstructed by the He gas to a greater degree than an analyte of the same mass owing to the smaller cross-sectional area of the analyte compared to that of the polyatomic interferent. The result is reduced interference with a relatively small reduction in analyte signal, thereby enhancing the signal to noise ratio.

Elements occurring at any m/z with a known argon-based interference are acquired in H<sub>2</sub> reaction mode. Since the plasma is composed of argon, there is inherently a tremendous abundance of Ar<sup>+</sup> ions present in the plasma. These may act as an interferent directly at m/z 40, or may participate in the formation of argon-containing polyatomic ions of the type ArM<sup>+</sup>. Flooding the reaction cell with H<sub>2</sub> results in the following:



Ar<sup>+</sup> is reduced to neutral Ar and therefore does not enter the mass spectrometer or form polyatomic ions. It should be noted that H<sub>2</sub> can act as a collision gas as well, and in fact the collisional obstruction of lighter elements by H<sub>2</sub> is considerable. However, this is far less significant when considering heavier elements. In any case, H<sub>2</sub> acts as a poorer collision gas than He due to H<sub>2</sub>'s relatively small cross-sectional area compared to that of He.

Table IV outlines the tune step in which each element is acquired, which isotope is acquired (if applicable), and internal standard element and isotope being utilized for that analyte. In addition, Tables V-IX outline the remaining acquisition parameters and instrument settings applied during this analysis.

Table IV. Tune step and internal standard m/z for each analyte m/z.

Analyte / m/z	IS / m/z	Tune Step
Be / 9	Li / 6	3 (Normal)
Na / 23	Y / 89	3 (Normal)
Mg / 24	Y / 89	3 (Normal)
Al / 27	Y / 89	3 (Normal)
Si / 29	Sc / 45	3 (Normal)
K / 39	Y / 89	3 (Normal)
Ca / 40	Sc / 45	1 (H <sub>2</sub> )
V / 51	Sc / 45	2 (He)
Cr / 52	Sc / 45	1 (H <sub>2</sub> )
Mn / 55	Y / 89	3 (Normal)
Fe / 56	Y / 89	1 (H <sub>2</sub> )
Co / 59	Y / 89	3 (Normal)
Ni / 60	Y / 89	3 (Normal)
Cu / 63	Y / 89	1 (H <sub>2</sub> )
Zn / 66	Y / 89	3 (Normal)
As / 75	Y / 89	2 (He)
Se / 77	Y / 89	3 (Normal)
Se / 78	Y / 89	1 (H <sub>2</sub> )
Mo / 98	Y / 89	3 (Normal)
Ag / 107	In / 115	3 (Normal)
Cd / 114	In / 115	3 (Normal)
Sb / 121	In / 115	3 (Normal)
Ba / 137	In / 115	3 (Normal)
Tl / 205	Bi / 209	3 (Normal)
Pb / 208	Bi / 209	3 (Normal)
Th / 232	Bi / 209	3 (Normal)
U / 238	Bi / 209	3 (Normal)

Table V. Interference equations.

Mass	Equation
6	$(6)^*1 - (7)^*0.0813$
115	$(115)^*1 - (118)^*0.0149$
208	$(208)^*1 + (207)^*1 + (206)^*1$

Table VI. Acquisition parameters.

<b>Acquisition mode</b>	Spectrum multi-tune
<b>Peak pattern</b>	Full quant
<b>Number of points per mass</b>	3
<b>Integration time per point</b>	0.10 sec
<b>Number of repetitions</b>	3
<b>Total acquisition time</b>	168 sec
<b>Detector</b>	Auto (P/A)
<b>Stabilization time, tune step 1 (tune file: mt_h2.u)</b>	30 sec
<b>Stabilization time, tune step 2 (tune file: mt_he.u)</b>	30 sec
<b>Stabilization time, tune step 3 (tune file: mt_norm.u)</b>	5 sec

Table VII. ISIS peristaltic pump program.

<b>Before Acquisition</b>	Uptake speed	0.70 rps
	Uptake time	20 sec
	Stabilization time (undiluted)	40 sec
	Stabilization time (diluted)	70 sec
<b>After Acquisition (probe rinse)</b>	Rinse speed	0.05 rps
	Rinse time (sample)	5 sec
	Rinse time (standard)	5 sec
<b>After Acquisition (rinse)</b>	Rinse vial	1
	Uptake speed	0.70
	Uptake time	60 sec
	Stabilization speed	0.02 rps
	Stabilization time	0 sec

Table VIII. ISIS autodilution settings.

<b>Dilution factor</b>	20
<b>Correction</b>	On (periodic mode)
<b>Std element</b>	166 amu
<b>Online IS element</b>	159 amu
<b>Dilute all samples</b>	Off

Table IX. Plasma conditions.

<b>RF power</b>	1500 W
<b>S/C Temperature</b>	2°C
<b>Carrier gas flow</b>	1.00 L / min
<b>Makeup gas flow</b>	0.42 L / min
<b>Peri pump speed</b>	0.10 rps
<b>Sample depth</b>	8.4 mm
<b>Torch – H</b>	x.x mm
<b>Torch – V</b>	x.x mm

### 3.4 QUALITY CONTROL

- a) *Internal standard performance.* The response of each internal standard is monitored throughout all standard, analysis, and quality control samples. The initial response in counts per second (cps) for each IS is established when analyzing the calibration blank. All subsequent cps values for each IS must be within the range of 60% - 125% of the initial baseline value. If this condition is not satisfied, the elements quantitated using that IS may not be reported unless the sample is reanalyzed with satisfactory IS recovery.
- b) *Continuing calibration verification (CCV).* For every ten samples, a CCV is analyzed in order to verify the integrity of the instrument calibration. The CCV may be prepared by adding 0.5 mL of the multi-element primary standard mixture and 500 µL of Si solution to 100 mL diluent solution. This results in a concentration of 5000 ppb in Fe, K, Ca, Na, and Mg and a concentration of 50 ppb in Ag, Al, As, Ba, Be, Cd, Co, Cr, Cu, Mn, Mo, Ni, Pb, Sb, Se, Tl, V, Zn, Th, and U. The analytical result for concentration of each element in the CCV must be within 85% - 115% of the actual value. If this condition is not satisfied, the failing element(s) may not be reported in the affected samples. If reanalysis of the CCV does not produce satisfactory results, recalibration may be necessary. Once satisfactory performance of the CCV is achieved, any affected samples may be reanalyzed.

- c) *Continuing calibration blank (CCB).* For every ten samples, a CCB consisting of unsupplemented diluent solution is analyzed in order to determine the presence or absence of unacceptable carryover. If the analytical result obtained for a CCB is greater than 0.5 ppb for trace elements or 50 ppb for non-trace elements, the affected elements may not be quantitated. Reanalysis of the CCB should be performed after rinsing the system with DI H<sub>2</sub>O. Samples may be reanalyzed after acceptable blank levels have been attained.
- d) *Overall performance verification.* An international standard for trace metals in water (NIST 1643) is measured undiluted to verify the overall performance of the trace metal analysis. This performance is tested once a month. The recovery of the standard must be between 85% to 115% for environmental analysis.

Table x: True and measured values for the NIST standard 1643

Element	concentration	Measured n=4	Dev
	µg/kg	µg/kg	%
Ag	7.6	8.4	11
Al	52.0	51.8	0
As	26.7	26.0	-3
Ba	148.0	145.2	-2
Be	34.9	34.0	-3
Cd	22.8	22.5	-1
Co	20.3	20.5	1
Cr	38.6	34.3	-11
Cu	85.2	84.3	-1
Fe	34.3	42.3	23
K	994.0	1035.1	4
Mn	121.5	123.7	2
Mo	46.8	46.8	0
Ni	27.4	27.8	1
Pb	27.9	27.7	-1
Sb	13.8	14.4	5
Se	22.0	18.8	-14
Th	Nd		
Tl	Nd		
U	Nd		
V	13.0	13.5	4
Zn	53.2	52.1	-2
	mg/kg	mg/kg	
Ca	7.0	6.8	3
Mg	5.8	5.9	2
Na	29.4	30.3	3

- e) *Calibration range exceeded.* If any element in a sample is found to exceed the range of calibration, the sample is to be diluted into the appropriate range and reanalyzed.

### 3.5 CALCULATIONS

Recoveries for standard solutions are calculated by dividing the observed value by the expected value. The result is multiplied by 100 to give a percent recovery.

$$\% \text{ recovery} = \frac{V_0}{V_e} \times 100\%$$

$V_0 = \text{Observed Value}$

$V_e = \text{Expected Value}$

The relative percent difference between duplicate samples is calculated as the absolute difference between the sample and the duplicate, divided by the average of the sample and the duplicate, all multiplied by 100.

$$\%RPD = \frac{|S_c - D_c|}{[(S_c + D_c)/2]} \times 100\%$$

$S_c = \text{Observed Sample Concentration}$

$D_c = \text{Observed Duplicate Sample Concentration}$

The limit of detection (LOD) is calculated from the calibration curves using the equation

$$LOD = \frac{3.3 S_{xy}}{m}$$

where ( $S_{xy}$ ) is the standard deviation of the x-intercept and (m) is the slope. For all compounds where a peak is detected but below the limit of detection are reported as <LOD. If no peak is detected in the chromatogram, data is reported as non-detect (ND).

#### 4 Chain of Custody

Chain of custody form is in place in the ASET laboratory. All samples handed to ASET have to be announced in advance. Ideally an electronic sample list with running number and sample name which is unique for each sample sent to ASET lab manager (or analyst in absence). A hardcopy of this sample list is required with each sample batch handed to ASET personnel. PI, date, number and kind of samples and the required storage environment are listed in *Chain of Custody Document*. This document is signed by the person who delivers the samples and person who receives the sample (only ASET personnel is allowed to accept samples).

#### 5 References

- EPA Method 3005, U.S. Environmental Protection Agency, Cincinnati, OH  
 EPA Method 200.8, Revision 5.4, U.S. Environmental Protection Agency, Cincinnati, OH, 1994.  
 Rohrbough, W.G.; et al. Reagent Chemicals, American Chemical Society Specifications, 7th ed.; American Chemical Society: Washington, DC, 1986.  
1985 Annual Book of ASTM Standards, Vol. 11.01; "Standard Specification for Reagent Water"; ASTM: Philadelphia, PA, 1985; D1193-77.

**APPENDIX****A1. REAGENT TRACE ELEMENT CONCENTRATION CERTIFICATES****HCl OmniTrace Ultra\*. 33–36%.**

CAS: 7647-01-0

FW: 36.46

Merck Index: 13.4801

D: 1.20 kg/L

Clear liquid. For low level trace metal analysis. Double-distilled and packaged in ISO Class 4 (FED-STD-209E Class 10/M2.5) cleanroom conditions. Supplied in specially designed, preleached fluoropolymer resin bottles to guarantee product integrity. Certificate of Analysis supplied.

Aluminum	100 ppt max.
Arsenic	100 ppt max.
Barium	100 ppt max.
Beryllium	100 ppt max.
Bismuth	10 ppt max.
Boron	100 ppt max.
Cadmium	10 ppt max.
Calcium	100 ppt max.
Chromium	100 ppt max.
Cobalt	100 ppt max.
Copper	100 ppt max.
Iron	100 ppt max.
Lead	10 ppt max.
Magnesium	100 ppt max.
Manganese	100 ppt max.
Mercury	100 ppt max.
Moybdenum	100 ppt max.
Nickel	100 ppt max.
Potassium	100 ppt max.
Silver	100 ppt max.
Sodium	100 ppt max.
Strontium	100 ppt max.
Sulfates	1 ppm max.
Thallium	10 ppt max.
Thorium	1 ppt max.
Tin	100 ppt max.
Titanium	100 ppt max.
Uranium	1 ppt max.
Vanadium	100 ppt max.
Zinc	100 ppt max.

Nitric Acid OmniTrace Ultra (EM) 67-70%HNO<sub>3</sub> in water

CAS: 7697-37-2

FW: 63.01

Merck Index: 13.6608

D: 1.49 kg/L

Clear liquid. For low level trace metal analysis. Double-distilled and packaged in ISO Class 4 (FED-STD-209E Class 10/M2.5) cleanroom conditions. Supplied in specially designed, preleached fluoropolymer resin bottles to guarantee product integrity. Certificate of Analysis supplied with each shipment.

Aluminum	100 ppt max.
Arsenic	100 ppt max.
Barium	100 ppt max.
Beryllium	100 ppt max.
Bismuth	100 ppt max.
Boron	100 ppt max.
Cadmium	100 ppt max.
Calcium	100 ppt max.
Chlorides	0.5 ppm max.
Chromium	100 ppt max.
Cobalt	100 ppt max.
Copper	100 ppt max.
Iron	100 ppt max.
Lead	10 ppt max.
Magnesium	100 ppt max.
Manganese	100 ppt max.
Mercury	100 ppt max.
Molybdenum	100 ppt max.
Nickel	100 ppt max.
Potassium	100 ppt max.
Silver	10 ppt max.
Sodium	100 ppt max.
Strontium	10 ppt max.
Sulfates	1 ppm max.
Thallium	10 ppt max.
Thorium	1 ppt max.
Tin	100 ppt max.
Titanium	100 ppt max.
Uranium	1 ppt max.
Vanadium	100 ppt max.
Zinc	100 ppt max.
Zirconium	10 ppt max.

## A2 Health and Safety

### A2.0. Safety guide for handling of concentrated acids in this method

**Nitric acid:** Poison, very corrosive

**Hydrochloric acid:** Poison, very corrosive

### A2.1. Chemical Classification / Chemical Hazards / Chemical Labeling / Chemical Storage.

All containers with acids, other than the original container and at any concentrations must be labeled with the following:

Name and concentration of acid  
Preparation date and initials.

All acidic waste with acid concentration  $> 0.1$  N has to be collected in designated waste container. Waste has to be neutralized to pH 7 before discarding.

### A2.2. Personal Protective Equipment (required)

Working with acids of all concentrations, requires goggles, lab coat and nitrile gloves  
Disposable nitrile glove thickness in both cases should be a minimum of 8 mils.

### A2.3. Engineering / Ventilation Controls

All lab work with acids at concentrations  $> 1$  N must be performed in a fume hood. The room must also be equipped with a safety eye wash station / safety shower.

### A2.4. Handling Procedures

Keep all containers closed to minimize hazards due to inhalation vapors / vapors and spills. Leak-proof plastic containers are mandatory when transporting concentrated acid solutions. All glass containers shall be transported in a rubber secondary containment bucket capable of containing 2.5 – 4.0 L to prevent breakage.

When **diluting** pure concentrated acid into water never pure water into concentrated acid. Watch out for heat development.

### A2.5. Exposures / Contact: Skin / Eyes / Inhalation / Contaminated Clothing

Upon exposure to nitric and hydrochloric acids:

**Skin contact** / Symptoms: Corrosive, causes pain, severe skin burns, deep ulcers and discolored skin

**First Aid:** Immediately flush skin with water for at least 15 minutes. Seek medical attention immediately.

**Eye contact** / Symptoms: Corrosive, vapors are irritating and may damage eyes. Contact may cause severe burns and permanent eye damage.

**First Aid:** Immediately flush eyes with water for at least 15 minutes. Get medical attention immediately.

**Inhalation** / Symptoms: May cause coughing, choking, inflammation of nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure and death.

**First Aid:** Remove to fresh air immediately. Get medical attention immediately.

**Ingestion** / Symptoms: swallowing can cause immediate pain and burns of the mouth, throat, esophagus and gastrointestinal tract. May cause nausea, vomiting, and diarrhea, may be fatal.

**First Aid:** Do not induce vomiting! Give large quantities of drinking water or milk if available (but never to an unconscious person). Get medical attention immediately.

**Contaminated clothing** / Symptoms: Causes etching, intense burning, may cause blister formation.

**First Aid:** Remove contaminated clothing and immediately flush affected area with water for at least 15 minutes. Seek medical attention if necessary.

*A2.6. Exposures / Medical Surveillance*

Medical surveillance is required when any of the following occurs:

Any person or employee has eye contact, skin contact, ingestion or inhaling of vapors of concentrated acids;

Any person or employee develops signs and symptoms of overexposure to acids or acidic vapors.

Any person or employee is exposed to acids during an emergency (spill / cleanup).

All arrangements for Medical services / surveillance can be arranged through UAA EHRMS, **Trig Trigiano 786-1351**.

*A2.7. Emergency Phone Numbers:*

Principle Investigator	John Kennish	907-786-1236
ASET LAB MANAGER	Birgit Hagedorn	907-786-1332
ASET Chemical Hygiene Officer	Maury Riner	907-786-1279
UAA EHRMS	Trig Trigiano	907-786-1351
UAA Police		907-786-1120