



## Laboratory Procedure Manual

*Analytes:* **Antimony, Barium, Beryllium, Cadmium, Cesium, Cobalt, Lead, Manganese, Molybdenum, Platinum, Strontium, Thallium, Tin, Tungsten, and Uranium**

*Matrix:* **Urine**

*Method:* **Urine Multi-Element ICP-DRC-MS**  
Renamed from "Inductively Coupled Plasma-Mass Spectrometry (ICP-DRC-MS)"

*Method No:* **3018.6-06**

*Revised:* February 28, 2019

*As performed by:* Inorganic and Radiation Analytical Toxicology  
Division of Laboratory Sciences  
National Center for Environmental Health

*Contact:* Mr. Jeffery M. Jarrett  
Phone: 770-488-7906  
Fax: 770-488-4097  
Email: [JJarrett@cdc.gov](mailto:JJarrett@cdc.gov)

James L. Pirkle, M.D., Ph.D.  
Director, Division of Laboratory Sciences

### **Important Information for Users**

The Centers for Disease Control and Prevention (CDC) periodically refines these laboratory methods. It is the responsibility of the user to contact the person listed on the title page of each write-up before using the analytical method to find out whether any changes have been made and what revisions, if any, have been incorporated.

**Public Release Data Set Information**

This document details the Lab Protocol for testing the items listed in the following table:

This method file describes measurements of UM\_J.

File Name	Variable Name	SAS Label
UM_J UM_J_R	URXUBA	Barium, urine (µg/L)
	URXUCD	Cadmium, urine (µg/L)
	URXUCO	Cobalt, urine (µg/L)
	URXUCS	Cesium, urine (µg/L)
	URXUMN	Manganese, urine (µg/L)
	URXUMO	Molybdenum, urine (µg/L)
	URXUPB	Lead, urine (µg/L)
	URXUSN	Tin, urine (µg/L)
	URXUSR	Strontium, urine (µg/L)
	URXUSB	Antimony, urine (µg/L)
	URXUTL	Thallium, urine (µg/L)
	URXUTU	Tungsten, urine (µg/L)
	URXUUR	Uranium, urine (µg/L)

## 1. Summary of test principle and clinical relevance

### A. Clinical relevance:

These methods are used to achieve rapid and accurate quantification of elements of toxicological and nutritional interest including antimony (Sb), barium (Ba), beryllium (Be), cadmium (Cd), cesium (Cs), cobalt (Co), lead (Pb), manganese (Mn), molybdenum (Mo), platinum (Pt), strontium (Sr), thallium (Tl), tin (Sn), tungsten (W), and uranium (U). Use these methods to screen urine when people are suspected to be acutely exposed to these elements or to evaluate chronic environmental or other non-occupational exposure. [1-4].

### B. Test principle:

Inductively coupled plasma mass spectrometry (ICP-MS) is a multi-element analytical technique capable of trace level elemental analysis [1-4]. When used with dynamic reaction cell technology (DRC), as with cadmium and manganese in this method, the technique is commonly referred to as ICP-DRC-MS. This method is used to measure all 15 elements described here (antimony, barium, beryllium, cadmium, cesium, cobalt, lead, manganese, molybdenum, platinum, strontium, thallium, tin, tungsten, and uranium), or any subgroup of these.

Liquid samples are introduced into the inductively coupled plasma through a nebulizer and spray chamber carried by a flowing argon stream. By coupling radio-frequency power into flowing argon, plasma is created in which the predominant species are positive argon ions and electrons and has a temperature of 6000-8000 K. The sample passes through a region of the plasma and the thermal energy atomizes the sample and then ionizes the atoms. The ions, along with the argon, enter the mass spectrometer through an interface that separates the ICP (at atmospheric pressure, ~760 torr) from the mass spectrometer (operating at a pressure of  $10^{-5}$  torr). The ions pass through a focusing region, the dynamic reaction cell, the quadrupole mass filter, and finally are counted in rapid sequence at the detector allowing individual isotopes of an element to be determined. The dynamic reaction cell operates in one of two modes. In 'standard' mode the cell is not pressurized and ions pass through the cell to the quadrupole mass filter unaffected. In 'DRC' mode the cell is pressurized with a gas which will collide or react with the incoming ions to either eliminate an interfering ion or change the ion of interest to a new mass which is free from interference. In this method the instrument is operated in DRC mode when analyzing for cadmium and manganese, but in standard mode when analyzing for all of the other analytes. When analyzing for cadmium in biomonitoring applications, the reaction cell is pressurized with oxygen. The  $^{98}\text{Mo}^{16}\text{O}^+$  ions which would normally interfere with detection of  $^{114}\text{Cd}$  at  $m/z$  114 react with the oxygen in the cell creating  $^{98}\text{Mo}^{16}\text{O}_2^+$  and  $^{98}\text{Mo}^{16}\text{O}_3^+$  at masses that no longer represent interference to low level  $^{114}\text{Cd}$  analysis. When low level Cd analysis is not the principle purpose of the analysis (i.e. emergency response situations), Cd analysis in vented (standard) mode will reduce analytical time and still yield quantitative results that are suitable for the identification of elevated exposures as long as the results are interpreted with the caveat that the  $^{98}\text{Mo}^{16}\text{O}_2^+$  interference on  $^{114}\text{Cd}$  is not eliminated. The DRC is also pressurized with oxygen gas when analyzing for  $^{55}\text{Mn}$ . The  $^{39}\text{K}^{16}\text{O}^+$  ions which would normally interfere with the detection of  $^{55}\text{Mn}$  at  $m/z$  55 react with the oxygen in the cell and no longer represent interference to  $^{55}\text{Mn}$  analysis. In DRC mode, the voltage applied by the axial field technology (AFT) and

the additional axial push from spectator ions also stable in the DRC bandpass serve to keep the ions moving axially through the pressurized DRC chamber where they would normally slow down due to collisions which result in loss of momentum. Gold is added to the diluent to normalize the spectator ion population in the DRC cell which could otherwise change significantly between low concentration and high concentration samples, a phenomenon called crosstalk [3, 5, 6]. Electrical signals resulting from the detection of ions are processed into digital information that is used to indicate first the intensity of the ions and then the concentration of the element. This method was originally based on the method by Mulligan et al. [7]. The DRC portions of the method are based on work published by Tanner et al. [2, 3]. Urine samples are diluted 1+ 9 with 2% (v/v) concentrated nitric acid. The diluent for the 15 element panel contains iridium (Ir), rhodium (Rh) for multi-internal standardization. Nitric acid is used for the purpose of solubilizing and stabilizing metals in solution. Internal standards are a constant concentration in all blanks, calibrators and samples. Monitoring the instrument signal ratio of a metal to its internal standard allows correction for instrument noise and drift, and sample-to-sample matrix differences.

## 2. Limitations of method; interfering substances and conditions

### A. Interferences addressed by this method:

#### i. Mathematical correction for tin ( $^{114}\text{Sn}$ ) on cadmium ( $^{114}\text{Cd}$ ):

The correction equation  $(-0.026826 * \text{Sn}118)$  is used in the “Equations” tab of the method to correct the counts observed as  $m/z$  114 to exclude counts due to  $^{114}\text{Sn}$ .

#### ii. Molybdenum oxide ( $^{98}\text{Mo}^{16}\text{O}$ ) on cadmium ( $^{114}\text{Cd}$ ):

The dynamic reaction cell is used in this method to eliminate interference from molybdenum oxide ( $^{98}\text{Mo}^{16}\text{O}$ ) onto cadmium at  $m/z$  114 [5]. Oxygen (100%) is the gas used in the dynamic reaction cell for this purpose. When low level Cd analysis is not the principle purpose of the analysis (i.e. emergency response situations), Cd analysis in vented (standard) mode will reduce analytical time and still yield quantitative results that are suitable for the identification of elevated exposures as long as the results are interpreted with the caveat that the  $^{98}\text{Mo}^{16}\text{O}^+$  interference on  $^{114}\text{Cd}$  is not eliminated. The anticipated bias on  $^{114}\text{Cd}$  due to the  $^{98}\text{Mo}^{16}\text{O}^+$  interference is described approximately by Equation 1 [5].

### Equation 1. Anticipated bias on $^{114}\text{Cd}$ due to the $^{98}\text{Mo}^{16}\text{O}^+$ interference.

$$\text{approximate } \mu\text{g/L difference (bias)} = 0.00175[\text{Mo}] - 0.0136$$

#### iii. Potassium oxide ( $^{39}\text{K}^{16}\text{O}$ ) on manganese ( $^{55}\text{Mn}$ ):

The dynamic reaction cell is used in this method to reduce the potassium oxide ( $^{39}\text{K}^{16}\text{O}$ ) interference on manganese at  $m/z$  55. See Section 1.b for an explanation of this process.

### B. Limitations of method:

#### i. Interferences on cobalt ( $^{59}\text{Co}$ ):

Preliminary tests suggest quantitation of cobalt may be positively biased approximately 0.3-0.4  $\mu\text{g/L}$  when the urine sample has elevated levels of calcium. This is expected to be

the result of unresolved polyatomic interferences such as  $^{40}\text{Ca}^{16}\text{O}^3\text{H}$ ,  $^{40}\text{Ca}^{17}\text{O}^2\text{H}$ ,  $^{40}\text{Ca}^{18}\text{O}^1\text{H}$ ,  $^{42}\text{Ca}^{16}\text{O}^1\text{H}$ ,  $^{43}\text{Ca}^{16}\text{O}$ , or  $^{44}\text{Ca}^{14}\text{N}^1\text{H}$ .

**ii. Interferences on cadmium ( $^{114}\text{Cd}$ ):**

Preliminary tests suggest quantitation of cadmium may be negatively biased in the presence of elevated tin by approximately -0.4% of the tin concentration in the urine sample. This occurs due to over-subtraction by the mathematical correction equation described in Section 2.a.i. For a urine sample with 4.14  $\mu\text{g}/\text{L}$  Sn (95<sup>th</sup> percentile level of NHANES 2013-2014), the cadmium concentration may be under reported by approximately 0.02  $\mu\text{g}/\text{L}$ .

**iii. Contamination control**

Accuracy and precision of this method can be critically impacted by the influence of elemental contamination. See Section 3.A regarding contamination control in the pre-analytical processes. Use high purity water and chemicals (See Section 5.E) and pre-rinsed or pre-screened containers in reagent and sample preparation (See Section 6). Occasionally we observe contamination sample preparations due to the Hamilton dilutor components (e.g. valve and syringes), especially if the components are new, or infrequently used. Manganese, especially, can be problematic from the valve of the Hamilton. Monitor instrument and dilutor cleanliness before and during analysis (See Section 8.B.iv) and rinse or replace components identified as causing elevated background levels in the reagent and blank checks.

### **3. Procedures for collecting, storing, and handling specimens; criteria for specimen rejection; specimen accountability and tracking**

**A. Procedures for collecting, storing, and handling specimens:**

Specimen handling conditions, special requirements, and procedures for collection and transport are discussed in the Division of Laboratory Science's (DLS) Policies and Procedures Manual [9]. In general

**i.** No fasting or special diets are required before collection of urine.

**ii.** Use sterile, lot screened collectors for specimen acquisition.

**iii.** Transport:

Transport urine specimens frozen (packed in dry ice during shipment is preferred when possible).

**iv.** Storage:

Once received, store long term at  $\leq -20\text{ }^\circ\text{C}$  until time for analysis. Short-term storage at 2-8  $^\circ\text{C}$  is acceptable. Refreeze at  $\leq -20\text{ }^\circ\text{C}$  portions of the sample that remain after analytical aliquots are withdrawn. Thawing and refreezing samples has not been found to compromise sample results.

**v.** Containers:

Acceptable containers for analytical aliquots include lot screened polypropylene (PP) cryovials or tubes (i.e. 2 to 5 mL cryogenic vial or 15mL centrifuge tube). Avoid colored

plastics and containers containing o-rings when possible due to increased risk of trace element contamination from coloring agents or o-ring materials. Externally threaded containers are preferred because they are less prone to contamination of the specimen and to leaks (internally threaded containers can develop leaks when biological material dries within the threads, compromising resealing).

#### B. Criteria for specimen rejection:

Specimen characteristics that compromise test results are indicated above. Other reasons for rejecting a sample for analysis are listed below. In all cases, request a second urine specimen.

##### i. Low volume:

Optimal amount of urine is 1.8+ mL. The volume of urine used for one analysis is 0.5 mL. Less volume is consumed when only subsets of the 15 elements are analyzed.

##### ii. Contamination:

Improper collection procedures or collection devices can contaminate the urine by contact with dust, dirt, etc.

#### C. Transfer or referral of specimens; procedures for specimen accountability and tracking

Location, status, and final disposition of the specimens will be tracked and records are maintained according to the Division's Policies and Procedures Manual [6] Use only numerical identifiers for samples within the laboratory (e.g., case ID numbers) in order to safeguard confidentiality. Only the medical supervisor (MS) or project coordinator (PC) i.e. non CDC personnel will have access to the personal identifiers.

### 4. Safety precautions

#### A. General Safety:

- i. Observe all safety regulations as detailed in the Laboratory Safety Manual and the Chemical Hygiene Plan.
- ii. Wear gloves, lab coat, and safety glasses while handling reagents, prepared solutions, or urine specimens.
- iii. Stock calibration standard B contains 1% hydrofluoric acid (HF). Latex is not an appropriate protective barrier against HF. When handling these solutions nitrile gloves (or other glove material known to be an adequate protective barrier to HF) are required.
- iv. Observe universal precautions when working with urine.
- v. Exercise special care when handling and dispensing concentrated nitric or hydrochloric acid. Use additional personal protective equipment which protects face, neck, and front of body. Add acid to water. Nitric and hydrochloric acids are caustic chemicals that are capable of causing severe eye and skin damage. **If concentrated acids come in contact with any part of the body, quickly wash the affected area with copious quantities of water for at least 15 minutes.**
- vi. Use secondary containment for containers of biological or corrosive liquids.
- vii. The use of the foot pedal on the benchtop automatic pipette is recommended because it reduces analyst contact with work surfaces that have been in contact with urine and

also keeps the analyst's hands free to hold the specimen cups and autosampler tubes and to wipe off the tip of benchtop automatic pipette.

- viii. There are many potential hazards on an operating ICP-MS instrument including ultraviolet radiation, high voltages, radio-frequency radiation, and high temperatures. This information is detailed in the ICP-MS System Safety Manual.
- ix. Transport and store compressed gas cylinders with proper securing harnesses. For compressed oxygen gas, use regulators which are oil-free. A flash arrestor can be used, but is not required.
- x. Wipe down all work surfaces at the end of the day with disinfectant. Disinfectant may be either daily remake of diluted bleach (1 part household bleach containing 5.25% sodium hypochlorite + 9 parts water) or equivalent disinfectant.

#### **B. Radiation safety:**

Calibration standards used in this method contain  $\mu\text{g/L}$  natural uranium. Staff performing this method in the CDC laboratory must maintain the status of Radiation Worker through the Radiation Safety Office and practice appropriate radiation safety when handling these solutions in accordance to the CDC's license with the Nuclear Regulatory Commission (NRC).

#### **C. Waste disposal:**

- i. Autoclaving: All diluted biological specimens, original biological specimens being disposed, or consumables which come into contact with biological specimens (even diluted or aerosolized). Use sharps containers or special autoclave pans for broken glass / quartz or items which puncture autoclave bags (e.g. pipette tips).
- ii. Other liquid waste:
  - 1. Waste discarded down sink: Do not discard solutions at the sink having a pH lower than 5.0 or higher than 11.5 (limits defined by Dekalb County, GA). Inactivate biological compounds and cellular constituents in mixed chemical and biological waste, such as the waste carboy of the ICP-MS, by adding an approved disinfectant (e.g. household bleach at a 1:100 dilution or equivalent) prior to drain disposal. Flush the sink with copious amounts of water.
  - 2. Waste to be picked up by the CDC radiation safety office: Solutions used in the CDC laboratory having uranium concentrations equal to that of the single element standard, intermediate stock standard, or intermediate working standards.
  - 3. Waste to be picked up by CDC hazardous waste program: Submit request for hazardous waste removal of all other liquid waste generated in the CDC laboratory for this method.

### **5. Instrument and material sources**

#### **A. Sources for ICP-MS instrumentation:**

- i. ICP-MS: Inductively coupled plasma mass spectrometer with dynamic reaction cell technology (ELAN<sup>®</sup> DRC II or NexION) (PerkinElmer Norwalk, CT, [www.perkinelmer.com](http://www.perkinelmer.com) )

- ii. Recirculating chiller / heat exchanger for ICP-MS: Refrigerated chiller (PolyScience 6105PE) or heat exchanger (PolyScience 3370) (PerkinElmer Norwalk, CT, [www.perkinelmer.com](http://www.perkinelmer.com))
- iii. Autosampler: ESI SC4-DX autosampler (Elemental Scientific Inc., Omaha, NE) or equivalent.
- iv. Computer: Computer controller provided or recommended by ICP-MS manufacturer is recommended to ensure proper communication between computer and ICP-MS. Recommend 1-2 Gb RAM and secondary internal hard disk for nightly backups (if network backups are not possible).
- v. FAST sample introduction system (optional): Standard peristaltic pump on ICP-MS replaced by DXi-FAST micro-peristaltic pump / FAST actuator and valve combination unit. For NEXION, like part # DXI-54-P4-F6. If DXi-FAST upgrade on ICP-MS is not used, a separate FAST actuator (built-in option on ESI SC4-DX autosampler or stand-alone FAST actuator) will be necessary to complete the FAST sample introduction system.

## B. Sources for ICP-MS parts & consumables

NOTE: The minimum number of spares recommended before reordering (if owning one instrument) are listed as “# Spares =” in the descriptions below.

- i. Adapter, PEEK: Securely connects 1.6mm O.D. PFA tubing to 0.03” I.D. peristaltic tubing. Composed of three PEEK parts.
  - 1. Female nut for 1.6mm O.D. (1/16”) tubing. Like part P-420 (Upchurch Scientific, Oak Harbor, WA, [www.upchurch.com](http://www.upchurch.com)).
  - 2. PEEK ferrule. Like part P-260x (10pk SuperFlangeless ferrule, Upchurch Scientific, Oak Harbor, WA, [www.upchurch.com](http://www.upchurch.com)).
  - 3. Conical Adapter Body. Like part P-692 (Upchurch Scientific, Oak Harbor, WA, [www.upchurch.com](http://www.upchurch.com)).
- ii. Bottles (for rinse solution): Four liter screw-cap polypropylene container with built-in luer connections (2) designed for use with FAST sample introduction system (like catalog# SC-0305-1, Elemental Scientific Inc., Omaha, NE., [www.elementalscientific.com](http://www.elementalscientific.com)).
- iii. Carboy and cap assembly for waste collection: 10-15 L, polypropylene wide-mouth carboy (100 mm neck size) with handles and no spigot (Like part #7BE-25126, Lab Safety Supply, Janesville, WI, [www.lss.com](http://www.lss.com)) with cap assembly like part # N0690271 (PerkinElmer, Norwalk, CT, [www.perkinelmer.com](http://www.perkinelmer.com)) with tubing connections built into the cap for addition of liquid waste.
- iv. Coolant, for Polyscience chiller or heat exchanger: Only PerkinElmer part # WE01-6558 (PerkinElmer Norwalk, CT, [www.perkinelmer.com](http://www.perkinelmer.com)) is approved for use by PerkinElmer. # Spares = 6.
- v. Cone, hyperskimmer (NexION): PerkinElmer part # W1033995 (PerkinElmer Norwalk, CT, [www.perkinelmer.com](http://www.perkinelmer.com)).
  - 1. Screws (for hyper skimmer cone, NexION): PerkinElmer part # 09919737 (PerkinElmer, Shelton, CT, [www.perkinelmer.com](http://www.perkinelmer.com)) or equivalent. # Spares = 4 screws per instrument.



**vi. Cone, sampler (nickel/platinum):**

1. ELAN ICP-MS: PerkinElmer part # WE021140 / WE027802 (PerkinElmer Norwalk, CT, [www.perkinelmer.com](http://www.perkinelmer.com)) or cross-referenced part number manufactured by Spectron Inc. (Ventura, CA, [www.spectronus.com](http://www.spectronus.com)) or Glass Expansion (Pocasset, MA, [www.geicp.com](http://www.geicp.com)). # Spares = 4.
2. NexION ICP-MS: PerkinElmer part # W1033612 / W1033614 (PerkinElmer Norwalk, CT, [www.perkinelmer.com](http://www.perkinelmer.com)) or cross-referenced part number manufactured by Spectron Inc. (Ventura, CA, [www.spectronus.com](http://www.spectronus.com)) or Glass Expansion (Pocasset, MA, [www.geicp.com](http://www.geicp.com)). # Spares = 4.

**vii. Cone, skimmer (nickel / platinum):**

1. ELAN ICP-MS: PerkinElmer part # WE021137 / WE027803 (PerkinElmer Norwalk, CT, [www.perkinelmer.com](http://www.perkinelmer.com)) or cross-referenced part number manufactured by Spectron Inc. (Ventura, CA, [www.spectronus.com](http://www.spectronus.com)) or Glass Expansion (Pocasset, MA, [www.geicp.com](http://www.geicp.com)). # Spares = 4.
2. NexION ICP-MS: PerkinElmer part # W1026356 / W1026907 (PerkinElmer Norwalk, CT, [www.perkinelmer.com](http://www.perkinelmer.com)) or cross-referenced part number manufactured by Spectron Inc. (Ventura, CA, [www.spectronus.com](http://www.spectronus.com)) or Glass Expansion (Pocasset, MA, [www.geicp.com](http://www.geicp.com)). # Spares = 4.

**viii. Connector (for tubing):** Use to connect 1/8" I.D. PVC tubing to 0.125" I.D peristaltic pump tubing. Use part # 3140715 (PerkinElmer Norwalk, CT, [www.perkinelmer.com](http://www.perkinelmer.com)) or equivalent. # Spares = 4.

**ix. Detector, electron multiplier:** Like part # N8125001 (PerkinElmer Norwalk, CT, [www.perkinelmer.com](http://www.perkinelmer.com)). Available direct from manufacturer (part # 14210, SGE Incorporated, Austin, Texas, <http://www.etpsci.com>) or various distributors. # Spares = 1.

**x. FAST / ESI SC4-DX autosampler accessories:**

1. Valve: CTFE High-flow valve head for SC-FAST (uses ¼-28 fittings). Like part # SC-0599-1010 (Elemental Scientific Inc., Omaha, NE., [www.elementalscientific.com](http://www.elementalscientific.com)).
2. Stator: CTFE Stator for 6 port SC-FAST high flow valve (¼-28 fittings). Like part # SC-0599-1010-01 (Elemental Scientific Inc., Omaha, NE., [www.elementalscientific.com](http://www.elementalscientific.com)).
3. Rotor: Composite rotor for 6 port SC-FAST high flow valve (¼-28 fittings). Like part # SC-0599-1010-05 (Elemental Scientific Inc., Omaha, NE., [www.elementalscientific.com](http://www.elementalscientific.com)).
4. Sample loop: 3 mL Teflon loop with white connector-nuts for high flow valve head, like part # SC-0315-30 (Elemental Scientific Inc., Omaha, NE., [www.elementalscientific.com](http://www.elementalscientific.com)). Subsets of elements can be analyzed using different loop sizes to minimize sample consumption (e.g. 0.5 mL loop for a single element, 1.0 mL loop for 3 element subset, 2.0mL loop for 8 element subset, etc...).

5. Probe, autosampler: Teflon, carbon fiber support, 0.8mm i.d., blue marker, 1/4-28 fittings. Like part number SC-5037-3751 (Elemental Scientific Inc., Omaha, NE., [www.elementalscientific.com](http://www.elementalscientific.com)). # Spares = 2.
  6. Probe, carrier solution: Teflon, carbon fiber support, 0.5mm i.d., orange marker, 1/4-28 fittings. Like part number SC-5037-3501 (Elemental Scientific Inc., Omaha, NE., [www.elementalscientific.com](http://www.elementalscientific.com)). # Spares = 2.
  7. Tubing, carrier solution: 0.5mm i.d. Teflon tubing (orange marker) with red ¼-28 male nut. Connects to high flow FAST valve head, port #2. Like part # SC-0316-0500 (Elemental Scientific Inc., Omaha, NE., [www.elementalscientific.com](http://www.elementalscientific.com)).
  8. Tubing, nebulizer: See “Nebulizer, PolyPro-ST micro flow”
  9. Tubing, rinse station: Teflon tubing and adapters (to attach to back of SC autosampler for filling rinse stations and to attach to rinse containers). Like part # SC-0302-0500, Elemental Scientific Inc., Omaha, NE., [www.elementalscientific.com](http://www.elementalscientific.com)).
  10. Tubing, vacuum: Vacuum line for SC-FAST high flow valve, connects to port #6, black nut for connection to valve head, natural brown color nut on other end for connection to SC autosampler vacuum port. Like part # SC-0321 (Elemental Scientific Inc., Omaha, NE., [www.elementalscientific.com](http://www.elementalscientific.com)).
- xi.** Hose, for connection to recirculator / chiller: Push on hose. I.D. = ½”, O.D. = ¾”. Use part # PB-8 (per inch, Georgia Valve and Fitting, Atlanta, GA, [www.swagelok.com](http://www.swagelok.com)) or equivalent. No spares necessary.
- xii.** Hose, for exhaust of ICP-MS: Available as part of ICP-MS installation kit from PerkinElmer (PerkinElmer Norwalk, CT, [www.perkinelmer.com](http://www.perkinelmer.com)), or equivalent. Available direct from manufacturer as part # S-LP-10 air connector (Thermaflex, Abbeville, SC, [www.thermaflex.net](http://www.thermaflex.net)). # Spares = 10 feet of 4” diameter (ELAN and NexION) and 10 feet of 6” diameter hose (ELAN).
- xiii.** Injector, quartz with ball joint: I.D. = 2.0 mm. PerkinElmer part # WE023948 (PerkinElmer Norwalk, CT, [www.perkinelmer.com](http://www.perkinelmer.com)). Available direct from manufacturer as part # 400-30 (Precision Glass Blowing, Centennial, CO, [www.precisionglassblowing.com](http://www.precisionglassblowing.com)) or from various distributors. # Spares = 2.
- xiv.** Ion lens (ELAN): PerkinElmer part # WE018034 (PerkinElmer Norwalk, CT, [www.perkinelmer.com](http://www.perkinelmer.com)). # Spares = 1.
- xv.** Nebulizer: PolyPro-ST micro flow polypropylene nebulizer with external 1/4-28 threaded connector for liquid delivery, low pressure version or equivalent. Like part # ES-4040-7010 (Elemental Scientific Inc., Omaha, NE., [www.elementalscientific.com](http://www.elementalscientific.com)). # Spares = 1. Comparable nebulizers are acceptable for substitution, however, the nebulizer gas flow rate, sample flush time, read delay time, loop fill time, loop size, urine sample dilution preparation volume, and sample-to-sample carry-over must be evaluated and optimized.
1. Gas connection:

- a. Teflon tubing: 4mm o.d., 2.4mm i.d. Teflon tubing (like part # ES-2502, Elemental Scientific Inc., Omaha, NE., [www.elementalscientific.com](http://www.elementalscientific.com)). # Spares = 1.
  - b. Adapter kit: Plastic adapters to connect Teflon tubing (2.4mm i.d.) to ¼" male Swagelok (compression) port on ICP-DRC-MS. Parts can be obtained as components in a "gas fittings kit for microflow nebulizer", kit part # ES-2501-1000, Elemental Scientific Inc., Omaha, NE., [www.elementalscientific.com](http://www.elementalscientific.com)). # Spares = 1.
2. Liquid connection: Connects nebulizer to port #3 of high flow FAST valve head with green, 1/4- 28 fitting. Like part # SC-0317-0250 (Elemental Scientific Inc., Omaha, NE., [www.elementalscientific.com](http://www.elementalscientific.com)). # Spares = 2.
- xvi.** Nut: (for flanged connections of 1.59mm (1/16") o.d. PFA tubing) Flanged, for 1/16" o.d. tubing, 1/4-28 threads. Use part # P-406x (pkg. of 10, Upchurch Scientific, Oak Harbor, WA, [www.upchurch.com](http://www.upchurch.com)) or equivalent. Use a Teflon-coated Viton o-ring with this nut instead of the stainless steel washer that comes with part # P-406x). # Spares = 10.
- xvii.** Nut and ferrule set, 1/8" Swagelok: Such as part # SS-200-NFSET (stainless steel) or part # B-200-NFSET (brass) (Georgia Valve and Fitting, Atlanta, GA, [www.swagelok.com](http://www.swagelok.com)) or equivalent. For part numbers listed here a quantity of 1 means 1 nut, 1 front ferrule, and 1 back ferrule. Spares = 20.
- xviii.** Nut and ferrule set, 1/4" Swagelok: Such as part # SS-400-NFSET (stainless steel) or part # B-400-NFSET (brass) (Georgia Valve and Fitting, Atlanta, GA, [www.swagelok.com](http://www.swagelok.com)) or equivalent. For part numbers listed here a quantity of 1 means 1 nut, 1 front ferrule, and 1 back ferrule. Spares = 20.
- xix.** Oil for roughing pumps:
1. Welch Directorr Gold: For roughing pumps. Available direct from manufacturer as part # 8995G-15 (1 gallon, Welch Rietschle Thomas, Skokie, IL, [www.welchvacuum.com](http://www.welchvacuum.com)), or equivalent. # Spares = 4.
  2. Fomblin Y14/5 fluid: PerkinElmer part # N8122265 (1 kg bottle, PerkinElmer, Shelton, CT, [www.perkinelmer.com](http://www.perkinelmer.com)) or equivalent. # Spares = 1 per instrument.
- xx.** O-ring (for hyper skimmer cone, NexION): PerkinElmer part # 09902123 (PerkinElmer, Shelton, CT, [www.perkinelmer.com](http://www.perkinelmer.com)) or equivalent. # Spares = 20 o-rings.
- xxi.** O-ring / gasket (for sampler cone):
1. ELAN (o-ring): PerkinElmer part # N8120511 (pkg. of 5, PerkinElmer, Shelton, CT, [www.perkinelmer.com](http://www.perkinelmer.com)) or equivalent. # Spares = 20 o-rings.
  2. NexION (aluminum gasket): PerkinElmer part # WE012989 (PerkinElmer, Shelton, CT, [www.perkinelmer.com](http://www.perkinelmer.com)) or equivalent. # Spares = 20 gaskets.
- xxii.** O-ring (for skimmer cone, ELAN): PerkinElmer part # N8120512 (pkg. of 5, PerkinElmer, Shelton, CT, [www.perkinelmer.com](http://www.perkinelmer.com)) or equivalent. # Spares = 20 o-rings.
- xxiii.** O-ring: (for flanged connections of 1.59mm (1/16") o.d. PFA tubing) Teflon-coated Viton o-ring, i.d. = 1/16", thickness = 1/16", o.d. = 3/16". Such as part # V75-003 (O-rings West, Seattle, WA, [www.oringswest.com](http://www.oringswest.com)) or equivalent. # Spares = 20.
- xxiv.** O-ring: (for injector support).

1. **Internal o-rings:** ID = ¼", OD = 3/8", thickness = 1/16". Need 2 o-rings per injector support setup. PerkinElmer part # N8122008 (PerkinElmer, Shelton, CT, [www.perkinelmer.com](http://www.perkinelmer.com)) or equivalent (such as part # V75-010, O-rings West, Seattle, WA, [www.oringswest.com](http://www.oringswest.com)). # Spares = 20.
  2. **External o-rings:** ID = 3/8", OD = 1/2", thickness = 1/16". Need 2 o-rings for each injector support setup. PerkinElmer part # N8122009 (PerkinElmer, Shelton, CT, [www.perkinelmer.com](http://www.perkinelmer.com)) or equivalent (such as part # V75-012, O-rings West, Seattle, WA, [www.oringswest.com](http://www.oringswest.com)). # Spares = 20.
- xxv.** O-ring (for inside nebulizer port on standard PerkinElmer cyclonic quartz spray chamber for the ELAN): Such as part # 120-56 (Precision Glass Blowing, Centennial, CO, [www.precisionglassblowing.com](http://www.precisionglassblowing.com)). Additional o-rings can sometimes be obtained free of charge or at reduced price when acquired while purchasing spray chambers. # Spares = 20.
- xxvi.** O-ring (for inside of ELAN bayonet torch mount): Part # WE017284 (PerkinElmer, Shelton, CT, [www.perkinelmer.com](http://www.perkinelmer.com)). Do not substitute. The PerkinElmer o-ring is metal impregnated to minimize RF leakage though the torch mount. # Spares = 2.
- xxvii.** Photon Stop (ELAN): PerkinElmer part # WE018278 (PerkinElmer, Shelton, CT, [www.perkinelmer.com](http://www.perkinelmer.com)). # Spares = 1.
- xxviii.** Plugs, quick change for roughing pump oil: These plugs will only work on the roughing pumps which come standard on ELAN DRC II and NexION ICP-MS instruments. These plugs will not fit the Leybold pumps which come standard on ELAN DRC Plus instruments. Part # W1011013 (PerkinElmer, Shelton, CT, [www.perkinelmer.com](http://www.perkinelmer.com)). No spares typically needed.
- xxix.** RF coil: PerkinElmer part # WE02-1816 (PerkinElmer, Shelton, CT, [www.perkinelmer.com](http://www.perkinelmer.com)) or equivalent. # Spares = 2.
- xxx.** Spray chamber, quartz concentric:
1. **ELAN:** PerkinElmer part # WE025221 (PerkinElmer, Shelton, CT, [www.perkinelmer.com](http://www.perkinelmer.com)) or equivalent. Available direct from manufacturer as part # 400-20 (Precision Glass Blowing, Centennial, CO, [www.precisionglassblowing.com](http://www.precisionglassblowing.com)) or from various distributors. # Spares = 2.
  2. **NexION:** PerkinElmer part # N8145013 (PerkinElmer, Shelton, CT, [www.perkinelmer.com](http://www.perkinelmer.com)) or equivalent. # Spares = 2.
- xxxi.** Torch, quartz: PerkinElmer part # N812-2006 (PerkinElmer, Shelton, CT, [www.perkinelmer.com](http://www.perkinelmer.com)) or equivalent. Available direct from manufacturer as part # 400-10 (Precision Glass Blowing, Centennial, CO, [www.precisionglassblowing.com](http://www.precisionglassblowing.com)) or various distributors. # New Spares = 2.
- xxxii.** Tubing, main argon delivery to instrument: I.D. = 1/8", O.D. = ¼". Such as part # C-06500-02 (pkg. of 100ft, polypropylene, Fisher Scientific International, Hampton, NH, [www.fishersci.com](http://www.fishersci.com)) or equivalent. # Spares = 50ft.
- xxxiii.** Tubing, peristaltic, 0.03" i.d. (carrier solution for ESI autosampler): *use either*
1. Standard PVC, 2-stop (black / black) peristaltic pump tubing, i.d. = 0.03". PerkinElmer part # 09908587 (PerkinElmer, Shelton, CT, [www.perkinelmer.com](http://www.perkinelmer.com)) or equivalent. # Spares = 6 packs of 12 tubes.

2. Standard PVC, 3-stop (black/ black/black) peristaltic pump tubing, i.d. 0.76 mm. Spectron part # SC0056 (Spectron, Ventura, CA, [www.spectronus.com](http://www.spectronus.com)) or equivalent. #Spares = 6 packs of 12 tubes. *Use this type of tubing with ESI DXi micro-peristaltic pump.*

**xxxiv.** Tubing, peristaltic, 0.125" i.d. (spray chamber drain): *use either*

1. Standard PVC, 2-stop (black / white) peristaltic pump tubing, i.d. = 0.125" or equivalent. PerkinElmer part # N812-2012 (PerkinElmer, Shelton, CT, [www.perkinelmer.com](http://www.perkinelmer.com)) or equivalent. # Spares = 6 packs of 12 tubes.
2. Standard Santoprene, 3-stop (grey/ grey/ grey) peristaltic pump tubing, i.d. 1.30 mm. Spectron part # SC0311 (Spectron, Ventura, CA, [www.spectronus.com](http://www.spectronus.com)) or equivalent. #Spares = 6 packs of 12 tubes. *Use this type of tubing with ESI DXi micro-peristaltic pump.*

**xxxv.** Tubing, PVC, i.d. = 1/8", o.d. = 3/16". Used to transfer liquid between spray chamber waste port and peristaltic pump and between peristaltic pump and liquid waste jug. Like part # 14-169-7A (pkg. of 50ft, Fisher Scientific International, Hampton, NH, [www.fishersci.com](http://www.fishersci.com)) or equivalent. # Spares = 20ft.

**xxxvi.** Tubing, stainless steel, o.d. = 1/8", wall thickness = 0.028": Used to connect DRC gas cylinders to ICP-MS gas ports. Like part # SS-T2-S-028-20 (20ft, Georgia Valve and Fitting, Atlanta, GA, [www.swagelok.com](http://www.swagelok.com)) or equivalent. Spares = 20ft.

**xxxvii.** Tubing, Teflon, corrugated, 1/4" o.d.: Connects to the auxiliary and plasma gas side-arms of the torch. Part # WE015903 (PerkinElmer, Shelton, CT, [www.perkinelmer.com](http://www.perkinelmer.com)). # Spares = 2.

**xxxviii.** Union elbow, PTFE 1/4" Swagelok (ELAN bayonet mount): Connects argon tubing to torch auxiliary gas sidearm on bayonet mount NEXION ICP-MS instruments. Like part # T-400-9 (Georgia Valve and Fitting, Atlanta, GA, [www.swagelok.com](http://www.swagelok.com)) or equivalent. Spares = 2.

**xxxix.** Union tee, PTFE, 1/4" Swagelok (ELAN bayonet mount): Connects argon tubing to torch plasma gas sidearm and holds igniter inside torch sidearm on bayonet mount NEXION ICP-MS instruments. Like part # T-400-3 (Georgia Valve and Fitting, Atlanta, GA, [www.swagelok.com](http://www.swagelok.com)) or equivalent. Spares = 2.

### C. Sources for ICP-MS maintenance equipment and supplies:

- i. Anemometer: Like digital wind-vane anemometer (Model 840032, SPER Scientific LTD., Scottsdale, AZ, [www.sperscientific.com](http://www.sperscientific.com)) or equivalent. Use to verify adequate exhaust ventilation for ICP-MS (check with hoses fully disconnected).
- ii. Container, to hold acid baths for glassware: Polypropylene or polyethylene containers with lids (must be large enough for torch, injector, or spray chamber submersion). Available from laboratory or home kitchen supply companies. # Spares = 4.
- iii. Cotton swabs: Any vendor. For cleaning of cones and glassware.
- iv. Cutter (for 1/8" o.d. metal tubing): Terry tool with 3 replacement wheels. Like part # TT-1008 (Chrom Tech, Inc., Saint Paul, MN, [www.chromtech.com](http://www.chromtech.com)) or equivalent.
- v. Getter regeneration kit: Part # WE023257 (PerkinElmer, Shelton, CT, [www.perkinelmer.com](http://www.perkinelmer.com)). Use this as needed (at least annually) to clean the getter in the pathway of channel A DRC gas.

- vi.** Magnifying glass: Any 10x<sup>+</sup> pocket loupe for inspection of cones and other ICP-MS parts. Plastic body is preferred for non-corrosion characteristics. Like part # 5BC-42813 (Lab Safety Supply, Janesville, WI, [www.labsafety.com](http://www.labsafety.com)).
- vii.** Pan, for changing roughing pump oil: Like part # 53216 (United States Plastics Corporation, Lima, OH, [www.usplastic.com](http://www.usplastic.com)) or equivalent. # Spares = 1.
- viii.** Screw driver, for ion lens removal: Screw driver with long, flexible shaft, and 2mm ball-Allen end for removal of ion lens screws part # W1010620. Extra 2mm bits, part # W1010598 (PerkinElmer, Shelton, CT, [www.perkinelmer.com](http://www.perkinelmer.com)). This is not necessary if the lens is mounted in a quick-release mount.
- ix.** Ultrasonic bath: Like ULTRASONIK™ Benchtop Cleaners (NEYTECH, Bloomfield, CT, [www.neytech.com](http://www.neytech.com)) or equivalent

**D. Sources for general laboratory consumable supplies:**

- i.** Bar code scanner: Like Xenon 1902 cordless area-imaging scanner (Honeywell International Inc., Morristown, NJ, [www.honeywellaidc.com](http://www.honeywellaidc.com)). For scanning sample IDs during analysis setup. Any bar code scanner capable of reading Code 128 encoding at a 3 mil label density and 2D bar codes can be substituted
- ii.** Carboy (for preparation of urine quality control pool and waste jug for ICP-MS sample introduction system): Polypropylene 10-L carboy (like catalog # 02-960-20C, Fisher Scientific, Pittsburgh, PA, [www.fishersci.com](http://www.fishersci.com)) or equivalent. Carboys with spouts are not advised due to potential for leaking.
- iii.** Containers for diluent and rinse solution: Two liter Teflon™ containers (like catalog# 02-923-30E, Fisher Scientific, Pittsburgh, PA., [www.fishersci.com](http://www.fishersci.com)) and 4L polypropylene jugs (like catalog# 02-960-10A, Fisher Scientific, Pittsburgh, PA, [www.fishersci.com](http://www.fishersci.com)) have both been used. Acid rinse before use.
- iv.** Cups for urine collection: Like polypropylene 4.5 oz cup, catalog # 354013 (Becton Dickinson Labware, Franklin Lakes, NJ, [www.bd.com](http://www.bd.com)) or equivalent. Each lot of cups used must be lot screened (tested to be free of trace metal contamination). Colorless plastics tend to have lowest trace metal contamination.
- v.** Flask, volumetric:
- vi.** 100mL volumetric flasks (like catalog # 40000100, Thermo Scientific, Fisher Scientific, Pittsburgh, PA., [www.fishersci.com](http://www.fishersci.com)). Plastic or glass is acceptable.
- vii.** 200mL volumetric flask (like catalog # 40000200, Thermo Scientific, Fisher Scientific, Pittsburgh, PA., [www.fishersci.com](http://www.fishersci.com)). Plastic or glass is acceptable.
- viii.** 500mL volumetric flask (like catalog # 40000500, Thermo Scientific, Fisher Scientific, Pittsburgh, PA., [www.fishersci.com](http://www.fishersci.com)). Plastic or glass is acceptable.
- ix.** 1L volumetric flask (like catalog # 40001000, Thermo Scientific, Fisher Scientific, Pittsburgh, PA., [www.fishersci.com](http://www.fishersci.com)). Plastic or glass is acceptable.
- x.** Gloves: Powder-free, low particulate nitrile (like Best CleaN-DEX™ 100% nitrile gloves, any vendor), or equivalent. Use only nitrile for handling calibration stock standard solution B which contains 1% HF.
- xi.** Paper towels: For general lab use, any low-lint paper wipes such as KIMWIPES®EX-L Delicate Task Wipers or KAYDRY®EX-L Delicate Task Wipers (Kimberly-Clark Professional, Atlanta, GA, [www.kcprofessional.com](http://www.kcprofessional.com)). For sensitive applications in cleanrooms, use a wipe designed for cleanrooms such as the Econowipe or Wetwipe (Liberty, East Berlin, CT, [www.liberty-ind.com](http://www.liberty-ind.com)).

- xii.** Pipette, benchtop automatic (for preparation of urine dilutions to be analyzed): Like the Microlab 625 advanced dual syringe diluter (Hamilton, Reno, NV, <http://www.hamilton.com/>) equipped with a 10.0 mL left syringe, a 1.0 mL right syringe, a 12 gauge Concorde CT probe dispense tip, the Microlab cable management system and a foot pedal. PEEK valves like part # 60676-01 (left) and part # 60675-01 (right) may reduce metal (e.g. manganese) background in prepared samples. Alternatives are acceptable, including the Micromedic Digiflex™ (Titertek, Huntsville, AL, <http://www.titertek.com/>) equipped with 10.0-mL dispensing syringe, 2 mL sampling syringe, 0.75-mm tip, and foot pedal.
- xiii.** Pipettes (for preparation of intermediate stock working standards & other reagents): Like Picus® NxT electronic, single-channel pipettes (Sartorius AG, Göttingen, Germany, [www.sartorius.com](http://www.sartorius.com)). 5-120 µL (catalog # LH-745041), 10-300 µL (catalog #LH-745061), 50-1000 µL (catalog #LH-745081), 100-5000 µL (catalog #LH-745101).
- xiv.** Tubes for sample analysis (for autosampler): Like polypropylene 15-mL conical tubes, BD Falcon model #352097 (Becton Dickinson Labware, Franklin Lakes, NJ, [www.bd.com](http://www.bd.com)), or equivalent. Clear plastics tend to have lowest trace metal contamination. Blue colored caps have also been used successfully for this method.
- xv.** Tubes for storage of intermediate working stock standards: Like polypropylene 50-mL conical tubes, BD Falcon model #352098 (Becton Dickinson Labware, Franklin Lakes, NJ, [www.bd.com](http://www.bd.com)), or equivalent. For use in storage of intermediate working stock standards. Clear plastics tend to have lowest trace metal contamination. Blue colored caps have also been used successfully for this method.
- xvi.** Vortexer: Like MV-1 Mini Vortexer (VWR, West Chester, PA, [www.vwr.com](http://www.vwr.com)). Used for vortexing urine specimens before removing an aliquot for analysis. Equivalent item can be substituted.
- xvii.** Water purification system: Like NANOpure Diamond Ultrapure Water System (Barnstead International, Dubuque, Iowa, [www.barnstead.com](http://www.barnstead.com)), or equivalent. For ultra-pure water ( $\geq 18$  Mohm-cm) used in reagent and dilution preparations.

#### **E. Sources of chemicals, gases, and regulators:**

- i.** Acid, hydrochloric acid: Double Distilled, 30-38% (GFS Chemicals Inc. Columbus, OH, [www.gfschemicals.com](http://www.gfschemicals.com)). This is referred to as “concentrated” hydrochloric acid in this method write-up. For use in preparation of intermediate working stock standards. Equivalent acid products which meet or exceed the trace metals purity are acceptable substitutions.
- ii.** Acid, nitric acid: Double distilled grade, 70% (GFS Chemicals Inc. Columbus, OH, [www.gfschemicals.com](http://www.gfschemicals.com)). For use in diluent, intermediate working stock standards, and QC pool preparations. This is referred to as “concentrated” nitric acid in this method write-up. Equivalent acid products which meet or exceed the trace metals purity are acceptable substitutions.
- iii.** Acid, nitric acid: Environmental Grade, 70% (GFS Chemicals Inc. Columbus, OH, [www.gfschemicals.com](http://www.gfschemicals.com)). For use in rinse solution. This is referred to as “concentrated” nitric acid in this method write-up. Equivalent acid products which meet or exceed the trace metals purity are acceptable substitutions.
- iv.** Argon gas (for plasma & nebulizer) and Regulator: High purity argon (>99.999% purity, Specialty Gases Southeast, Atlanta, GA, [www.sgsgas.com](http://www.sgsgas.com)) for torch and nebulizer.



Minimum tank source is a dewar of liquid argon (180-250L). Bulk tank (1500+L is preferred).

1. Regulator for argon (at dewar): Stainless steel, single stage, specially cleaned regulator with 3000 psig max inlet, 0-200 outlet pressure range, CGA 580 cylinder connector, and needle valve shutoff on delivery side terminating in a ¼” Swagelok connector. Part number “KPRCGRF415A2/AG10-AR1” (Georgia Valve and Fitting, Atlanta, GA, [www.swagelok.com](http://www.swagelok.com)), or equivalent. # Spares = 1.
  2. Regulator for argon (between bulk tank and PerkinElmer filter regulator): Single Stage 316SS Regulator, with 0-300 psi Inlet Gauge, 0-200 psi Outlet Gauge, Outlet Spring Range, 0-250 psi, ¼” Swagelok Inlet Connection, ¼ turn Shut off Valve on Outlet with ¼” Swagelok Connection and Teflon Seals. Part number KPR1GRF412A20000-AR1 (Georgia Valve and Fitting, Atlanta, GA, [www.swagelok.com](http://www.swagelok.com)), or equivalent. # Spares = 1.
  3. Regulator for argon (filter regulator on back of ICP-MS):
    - a. ELAN: Argon regulator filter kit. Catalog number N812-0508 (PerkinElmer, Shelton, CT, [www.perkinelmer.com](http://www.perkinelmer.com)).
    - b. NexION: Argon regulator filter kit. Catalog number N8145023 (PerkinElmer, Shelton, CT, [www.perkinelmer.com](http://www.perkinelmer.com)).
- v. Disinfectant, for work surfaces: Diluted bleach (1 part household bleach containing 5.25% sodium hypochlorite+ 9 parts water), re-made daily or equivalent disinfectant.
- vi. Oxygen: Oxygen (“Research Grade 5.0”, 99.9999% purity, equivalent, or higher purity) for DRC channel B. Like part # OX R33A (Airgas South, Atlanta, GA, [www.airgas.com](http://www.airgas.com)).
1. Regulator for oxygen: Stainless steel, two stage regulator for use with high purity oxygen (cleaned to be free of all oils). Maximum inlet pressure 3600-5000 psi. Inlet gauge pressure 0-5000 psi (no oil in gauge). Maximum delivery pressure 50–100 psi with a 0-30 psi outlet gauge (no oil in gauge). CGA 540 cylinder connector on inlet side and an angle pattern (90 degree) stainless steel needle valve on the delivery side terminating in a 1/8” stainless steel Swagelok connector. Like part # GEORG/KCYCFR/ORS2/540 (Georgia Valve and Fitting, Atlanta, GA, [www.swagelok.com](http://www.swagelok.com)), or equivalent.
- vii. Standard, dual detector: Like item # SM-2107-052 (High Purity Standards, Charleston, SC, <http://www.hps.net/>).
- viii. Standard, iridium: Like 1,000 mg/L iridium, item # PLIR3-2Y (SPEX Industries, Inc., Edison, NJ, [www.spexcsp.com](http://www.spexcsp.com)), or equivalent. Used as an internal standard in diluent. Standard must be traceable to the National Institute for Standards and Technology.
- ix. Standard, multi-element stock calibration standard: Item numbers “SM-2107-037 solution A” and “SM-2107-037 solution B” (High Purity Standards, Charleston, SC, <http://www.hps.net/>). This is a set of custom mixes (see Table 3 in Appendix C for concentrations). Both are needed to cover all analytes of method 3018. These solutions are diluted to prepare the intermediate stock working standards, which are in turn diluted to prepare the working calibrators. This solution can be prepared in-house from NIST traceable single element stock solutions if necessary.



- x. Standard, rhodium: Like 1,000 mg/L, item # PLRH3-2Y. (SPEX Industries, Inc., Edison, NJ, [www.spexcsp.com](http://www.spexcsp.com)), or equivalent. Used as an internal standard in diluent. Standard must be traceable to the National Institute for Standards and Technology.
- xi. Standard, single element stock standards for preparation of urine quality control pools: National Institute of Standards and Technology (NIST) Standard Reference Materials (SRMs) 3105a (Be), 3113 (Co), 3132 (Mn), 3134 (Mo), 3108 (Cd), 3102a (Sb), 3111a (Cs), 3104a (Ba), 3163 (W), 3128 (Pb), 3140 (Pt), 3161a (Sn), 3156a (Sr), 3158 (Tl), and 3164 (U) (National Institute of Standards and Technology (NIST), Office of Standard Reference Materials, Gaithersburg, MD, [www.nist.gov](http://www.nist.gov)). Other sources of standards can be used if they are NIST traceable.
- xii. Triton X-100™ surfactant: Like “Baker Analyzed” TritonX-100™ (J.T. Baker Chemical Co., [www.jtbaker.com](http://www.jtbaker.com)), or equivalent.
- xiii. Standard, gold: Like 10,000 µg/mL, cat # 10M21-2. (High Purity Standards, Charleston, SC, [www.highpuritystandards.com](http://www.highpuritystandards.com)), or equivalent. Used in diluent and rinse solution. Standard must be traceable to the National Institute for Standards and Technology

## 6. Preparation of reagent and materials

### A. Intermediate internal standard mixture:

- i. Purpose: Preparation of single intermediate solution containing internal standards will simplify the addition of the internal standards into the final diluent solution. This solution can be purchased rather than prepared
- ii. Preparation: To prepare 200 mL of 2% (v/v) HNO<sub>3</sub>, 40 µg/mL Ir and Rh intermediate internal standard solution:
  1. If not previously dedicated to this purpose, acid wash a 200 mL volumetric flask (PP, PMP, or Teflon™). For example, with 2% (v/v) HNO<sub>3</sub> and ≥18 Mohm·cm water (at least 3 times each) and verify cleanliness through analysis of rinsate.
  2. Partially fill the 200 mL volumetric flask with ≥18 Mohm·cm water (approximately 100-150 mL).
  3. Carefully add 4 mL of concentrated nitric acid. Mix into solution.
  4. Add 8,000 µg of rhodium (e.g. 8 mL of 1,000 µg/mL Rh stock standard).
  5. Add 8,000 µg of iridium (e.g. 8 mL of 1,000 µg/mL Ir stock standard).
  6. Fill to mark (200mL) and mix thoroughly.
  7. Store at ambient temperature and label appropriately. Expiration is 1 year from date of preparation.

### B. Intermediate Triton X-100™ solution:

- i. Purpose: To avoid the time-consuming process of dissolving Triton X-100 on a daily basis for use in rinse solution, prepare an intermediate solution for daily use.
- ii. Preparation: To prepare 2L of 2% Triton X-100™ in 5% (v/v) HNO<sub>3</sub>:
  1. If not previously dedicated to this purpose, acid wash a 2 L bottle (PP, PMP, or Teflon™). For example, with 2% (v/v) HNO<sub>3</sub> and >18 Mohm·cm water (at least 3 times each) and verify cleanliness through analysis of rinsate.
  2. Partially fill the bottle with ≥18 Mohm·cm water (approximately 1-1.5 L).

3. Add 40 mL of Triton X-100™ and stir until completely dissolved. Use a Teflon™ stir bar and stir plate if necessary (acid wash stir bar before use).
4. Carefully add 100 mL of concentrated nitric acid.
5. Fill to 2 L and stir thoroughly.
6. Store at ambient temperature and label appropriately. Expiration is 1 year from date of preparation.

**C. Diluent and carrier:**

- i. Purpose: All samples (blanks, calibrators, QC, or patient samples) are combined with the diluent during the sample preparation step before analysis. This is where the internal standards are added which during the analysis will compensate for instrumental variations on the analyte signal. If using the FAST sample introduction system, the diluent is also used as the carrier solution.
- ii. Preparation: To prepare 2 L of an aqueous solution of 10 microgram/L internal standards and 500 µg/L gold in 2% (v/v) nitric acid:
  1. If not previously dedicated to this purpose, acid wash a 2 L container (PP, PMP, or Teflon™). For example, with 2% (v/v) HNO<sub>3</sub> and >18 Mohm·cm water (at least 3 times each) and verify cleanliness through analysis of rinsate.
  2. Partially fill the 2 L container with ≥18 Mohm·cm water (~2/3 full).
  3. Carefully add 40 mL concentrated nitric acid and mix.
  4. Add spike of internal standard solution (to use other concentrations or volumes, adjust the volumes proportionally).
  5. Add 500 µL of the 40 µg/mL Rh and Ir internal standard solution.
  6. Add 100 µL of the 10,000 µg/mL gold standard.
  7. Make up to volume (2 L) with ≥18 Mohm·cm water.
  8. Store at ambient temperature and label appropriately. Expiration is 1 year from date of preparation.

**D. ICP-MS rinse solution:**

- i. Purpose: Pump this solution into the sample introduction system between samples to prevent carry-over of the analytes of interest from one sample measurement to the next.
- ii. Preparation: To Prepare 4 L of 0.002% Triton X-100™, 5% (v/v) nitric acid solution and 500 µg/L gold:
  1. If not previously dedicated to this purpose, acid wash a 4 L container (PP, PMP, or Teflon™). For example, with 5% (v/v) HNO<sub>3</sub> and ≥18 Mohm·cm water (at least 3 times each) and verify cleanliness through analysis of rinsate.
  2. Partially fill the bottle with ≥18 Mohm·cm water (approximately 2-3 L).
  3. Add 4 mL of the 2% Triton X-100™ / 5% (v/v) nitric-acid intermediate stock solution and mix well.
  4. Carefully add 200 mL of concentrated nitric acid and mix well.

5. Add 200  $\mu\text{L}$  of the 10,000  $\mu\text{g}/\text{mL}$  gold.
6. Fill to 4 L using  $\geq 18$  Mohm-cm water.
7. Store at ambient temperature and label appropriately. Expiration is 1 year from date of preparation.

#### E. Standards, calibrators, and QC:

##### i. Multi-element stock calibration standard

1. Purpose: These two master solutions will be diluted to prepare intermediate working calibrators.
2. Preparation: Prepared by external vendor or in-house from NIST-traceable standards.
  - a. Solution A: 5%  $\text{HNO}_3$  solution containing Ba, Be, Cd, Cs, Co, Pb, Mn, Sr, Tl, and U. Concentrations are listed in Table 3 of Appendix C.
  - b. Solution B: 5%  $\text{HNO}_3$ , 1% HF, 0.5% HCl solution containing Sb, Mo, Pt, Sn, and W. Concentrations are listed in Table 3 of Appendix C.
3. Storage: Store at ambient temperature and label appropriately. Expiration is determined by manufacturer or is 1 year after the container is opened (whichever comes first).

##### ii. Intermediate multi-element stock calibration standard

1. Purpose: The two stock standards are combined into a single intermediate stock calibration standard preparation.
2. Preparation: To prepare standards in 2% (v/v) nitric acid and 1% (v/v) hydrochloric acid which have final concentrations listed in Table 4 of Appendix C:
  - a. If not previously dedicated to this purpose, acid wash a 100 mL PP, PMP, or Teflon™ volumetric flask. For example, with a 2% (v/v)  $\text{HNO}_3$  / 1% (v/v) HCl solution and  $\geq 18$  Mohm-cm water (at least 3 times each) followed by verifying cleanliness through analysis of rinsate. Dedicate to purpose.
  - b. Partially fill the 100 mL volumetric flask with the 2% (v/v) nitric acid and 1% (v/v) hydrochloric acid prepared in Section 6.e.iii (50-75% full).
  - c. Using the volume listed in Table 4 of Appendix C, pipette the appropriate volume of the multi-element stock calibration standard solutions (both A and B) into the volumetric flask. Dilute to the volumetric mark with the 2% (v/v) nitric acid and 1% (v/v) hydrochloric acid using a pipette for the final drops. Mix each solution thoroughly. Final concentrations are listed in Table 4 of Appendix C.
  - d. Once mixed, transfer to an acid-cleaned, labeled, 50 mL container (PP, PMP, or Teflon™) for storage.
  - e. Label appropriately and store at ambient temperature. Expiration is 1 year from the date of preparation.

##### iii. Multi-element intermediate working calibration standards

1. **Purpose:** Six multi-element standards (S0 plus 5 spiked standards) used each day of analysis to prepare the final working calibrators.
2. **Preparation:** To prepare multi-element standards S0-S5 in 2% (v/v) HNO<sub>3</sub>, 1% (v/v) HCl according to the volumes and concentrations listed in Table 5 of Appendix C:
  - a. **Cleaning flasks:** If not previously dedicated to this purpose, acid wash PP, PMP, or Teflon™ volumetric flasks. For example, with a 2% (v/v) HNO<sub>3</sub> / 1% (v/v) HCl solution and ≥18 Mohm·cm water (at least 3 times each) followed by verifying cleanliness through analysis of rinsate. Dedicate to purpose.
  - b. **2% (v/v) HNO<sub>3</sub> & 1% (v/v) HCl diluent (S0) preparation:** In a cleaned 2L volumetric flask, add 1-1.5L ≥18 Mohm·cm water, 40 mL high purity concentrated HNO<sub>3</sub>, and 20 mL high purity concentrated HCl. Fill to the mark and mix thoroughly. Use this diluent to fill the remaining volumetric flasks during preparation of the intermediate working calibration standards.
  - c. **Dilutions & storage:**
    - i. Fill two acid-cleaned 50-mL containers (PP, PMP, or Teflon™) with the HNO<sub>3</sub> and HCl diluent. Label appropriately (including “S0”) and store at ambient temperature. Expiration is 1 year from the date of preparation.
    - ii. Partially fill the volumetric flasks with the HNO<sub>3</sub> & HCl diluent (50-75% full).
    - iii. Using the volumes listed in Table 5 of Appendix C, pipette the appropriate volume of the multi-element stock calibration standard or the multi-element intermediate stock calibrator solutions (both A and B) into each of the volumetric flasks. Dilute each to the volumetric mark with the HNO<sub>3</sub> & HCl diluent using a pipette for the final drops. Mix each solution thoroughly. Final concentrations are listed in Table 5 of Appendix C.
    - iv. Once mixed, transfer to acid-cleaned, labeled, 50-mL containers (PP, PMP, or Teflon™) for storage.
    - v. Store at ambient temperature and label appropriately. Expiration is 1 year from date of preparation.

#### iv. Working multi-element calibrators

1. **Purpose:** The working multi-element calibrators will be analyzed in each run to provide a signal-to-concentration response curve for each analyte in the method. The concentration of an analyte in a patient urine sample dilution is determined by comparing the observed signal from the dilution of the patient urine sample to the response curve from the working multi-element calibrators.
2. **Preparation:** Make dilutions of the intermediate working calibration standards (S0-S5) immediately prior to analysis by combining with base urine (Section 6.e.v) and diluent (Section 6.c) using a benchtop automatic pipette. See Table 8 of Appendix C and Section 8.b.ii for details of sample preparation. Expiration of

capped dilutions is 3 days from preparation (see Appendix B, test for time between preparation and analysis).

**v. Base urine**

1. **Purpose:** This urine pool material will be mixed with the intermediate working calibrators just prior to analysis to matrix-match the calibration curve to the urine matrix of the unknown samples.
2. **Contents:** A mixture of multiple urine sources collected from anonymous donors are used to approximate an average urine matrix.
3. **Preparation and storage:**
  - a. Collect urine anonymously by placing screened containers and collection cups in the restrooms with a sign stating the reason the specimens are being collected, the name of the investigator to contact for additional information, and requesting that people provide a urine specimen (see supervisor regarding potential Institutional Review Board, IRB, requirements).
  - b. Once collected, analyze to ensure that concentrations of the analytes in this method are relatively low, so as to not interfere with the proper measurement of calibrators (see Table 2 in Appendix C for suggested maximum base urine concentrations).
  - c. Once screened, mix the urine collections together in a larger container (polypropylene (PP), polymethylpentene (PMP), or Teflon™) which has been acid washed. For example, with 2% (v/v) HNO<sub>3</sub> and ≥18 Mohm·cm water (at least 3 times each) and verify cleanliness through analysis of rinsate. Add large Teflon™ stir bar and stir for 30+ minutes.
  - d. Label appropriately and store long-term in smaller volume tubes (e.g. 50-mL acid-washed or lot screened polypropylene tubes) at ≤ -20 °C. Expiration date is 3 years from the date of preparation.

**vi. Internal quality control materials (“Bench” QC)**

1. **Purpose:** Internal (or “bench”) quality control (QC) materials are used to evaluate the accuracy and precision of the analysis process, and to determine if the analytical system is “in control” (is producing results that are acceptably accurate and precise). They are included in the beginning and at the end of each analytical run.
2. **Content:** The internal (or “bench”) quality control (QC) materials used in this method are pooled human urine, acidified to 1% (v/v) HNO<sub>3</sub>, and spiked, if necessary, to reach a desired concentration. The analyte concentrations are in the low-normal concentration range (“low QC”) and high-normal concentration range (“high QC”).
3. **Preparation & storage:** Quality control materials can be either prepared by and purchased from an external laboratory or prepared within the CDC laboratories. Quality control must always be traceable to the National Institute for Standards and Technology (NIST). The CDC laboratory currently prepares its own bench QC materials using the following procedures:

- a. Collection of urine: Collect urine anonymously by placing screened containers and / or collection cups in the restrooms with a sign stating the reason the specimens are being collected, the name of the investigator to contact for additional information, and requesting that people provide a urine specimen. Volume of urine to collect is dependent on the desired pool size. This write-up will assume a 10-L pool size for both the low and high bench QC.
- b. Screening urine: Screen collected samples for metal content before mixing together to make separate pools that will be spiked to low, high, and elevated levels. Samples can be screened individually or after combining several together (reduces number of analyses).
  - i. Keep urine refrigerated whenever possible to minimize microbial growth.
  - ii. Because this is only a quick screen of the metal content, the number of replicates in the urine method can be reduced to one in order to reduce analysis time.
  - iii. Spike analyte concentrations for the low bench QC pool in the low-normal population range. Spike analyte concentrations for the high bench QC pool less than some preselected target concentration values in the high normal population range. See the National Report on Human Exposure to Environmental Chemicals for estimations of the normal population ranges for metals (<http://www.cdc.gov/exposurereport/>).
- c. Combining collected urine: Be attentive not to combine only diluted matrix urine samples into the low pool and only concentrated matrix urine samples into the pool for high and elevated QC. The goal is for combining samples is to approach an 'average' matrix for each pool.
  - i. Graduate four acid-washed 10-L carboys (PP or PMP) in 0.5 L increments (two will be used for decanting into).
  - ii. Combine collected urine samples into separate acid-washed 10-L carboys (PP or PMP), according to their concentrations, for the low high, and elevated bench QC pools.
  - iii. Mix each urine pool using large acid washed, Teflon™ coated stir bars and large stir plates. Keep urine refrigerated whenever possible.
  - iv. Acidify each urine pool to 1% (v/v) HNO<sub>3</sub> by adding the appropriate volume of concentrated HNO<sub>3</sub>. Stir for 30+ min on large stir plates.
- d. Settling out of solids:
  - i. Refrigerate the urine (no stirring) for 1-3 days to allow for settling out of solids.
  - ii. For each urine pool, decant the urine into another of the acid-washed 10-L carboys to remove the urine from the solids settled out on the bottom of the carboy.
  - iii. Repeat steps (i) and (ii) until minimal solids are left at the bottom of the carboy after sitting overnight.
- e. Spiking of urine
  - i. Analyze a sample of each urine pool. Record these results for future recovery calculations.

- ii. Use these results to determine target analyte concentrations possible for the pools
  - iii. Calculate the volume of single element standards needed to spike each pool to the desired concentrations.
  - iv. While stirring the pools on large stir plates, spike each pool with calculated volumes of single element standards (all spiking standards used must be traceable to NIST).
  - v. Continue to stir pools for 30+ minutes after spiking, then reanalyze.
  - vi. Repeat steps 4 and 5 until all analytes reach target concentrations keeping track of the total volume of spiking solution added to each urine pool.
- f. Dispensing and storage of urine
- i. Container types: Dispense urine into smaller, lot screened containers (e.g. 2 mL cryovials). This allows for one vial of QC to be used in only a small number of analyses, reducing chances of contamination due to long-term use of the same container.
  - ii. Labels: Place labels on vials after dispensing and capping if the vials are originally bagged separately from the caps. This minimizes the chance for contamination during the process. Include at least the name of QC pool (text and bar code), date of preparation, and a vial number on the labels.
  - iii. Dispensing: Dispensing can be accomplished most easily using a benchtop automatic pipette in continuous cycling dispense mode. Complete this process in a clean environment (i.e., a class 100 cleanroom area or hood). Allow urine pool to reach ambient temperature before dispensing (to prevent temperature gradients possibly causing concentration gradients across the large number of vials being dispensed and to prevent condensation problems during labeling of vials). Replace the tubing attached to the dispensing syringe (left when looking at front of benchtop automatic pipette) with a length of clean Teflon™ tubing long enough to reach into the bottom of the 10L carboy while it is sitting on the stir plate. Check cleanliness of benchtop automatic pipette before use by analyzing 1-2% (v/v) HNO<sub>3</sub> which has been flushed through the benchtop automatic pipette with a portion of the same solution which has not been through the benchtop automatic pipette. Approximately one hour before dispensing begins, with the large stir plate close to the left side of the benchtop automatic pipette, begin stirring the urine pool to be dispensed. Also during this time, flush the benchtop automatic pipette with urine from the pool to be dispensed. Place the ends of the tubing attached to both the sample and dispensing syringes into the carboy of urine so that urine won't be used up during this process. Be sure to secure both ends of tubing in the carboy with Parafilm so they will not come out during the flushing process. After dispensing the urine into the vials, cap the vials and label them. Placing labels on vials after capping minimizes the chance for contamination during the process.

- iv. Homogeneity testing: After dispensing, check homogeneity of analyte concentrations in pool aliquots by analysis of vials selected from across those dispensed. Seek guidance from a statistician regarding the number of vials needed for homogeneity analysis.
- v. Storage: Store long-term according the same storing and handling criteria described in Section 3.

## F. Optimization solutions:

### i. DRC optimization (cell gas flow rate and RPq):

1. Purpose: For periodic testing of the DRC cell parameters. Procedure requires at a minimum a blank (i), an analyte solution (ii), a blank with interference (iii), and an analyte and interference containing solution (iv).
2. Content: Diluent in this section refers to sample diluent (10 µg/L internal standards and 500 µg/L gold in 2% (v/v) nitric acid) described in Section 6c.
  - a. Solutions for testing elimination of  $^{98}\text{Mo}^{16}\text{O}$  interference on  $^{114}\text{Cd}$ :
    - i. Base urine in diluent (1 + 9)
    - ii. Base urine in diluent (1 + 9) + 0.24 µg/L Cd
    - iii. Base urine in diluent (1 + 9) + 300 µg/L Mo
    - iv. Base urine in diluent (1 + 9) + 0.24 µg/L Cd + 300 µg/L Mo
  - b. Solutions for testing elimination of  $^{39}\text{K}^{16}\text{O}$  interference on  $^{55}\text{Mn}$ :
    - i. Base urine in diluent (1 + 9)
    - ii. Base urine in diluent (1 + 9) + 0.3 µg/L Mn
    - iii. Base urine in diluent (1 + 9) + 400 µg/L K
    - iv. Base urine in diluent (1 + 9) + 0.3 µg/L Mn + 400 µg/L K
3. Preparation & storage: Prepare different volumes by adding proportionally larger or smaller volumes of solution constituents. Interference concentrations can be prepared higher as needed by adjusting the volume of this spike. Keep interference spike volume small (<0.3 mL) using a high concentration stock solution (i.e. 1000 mg/L). Analyte concentrations can be made higher if needed for sensitivity reasons by preparing a higher concentration calibrator.
  - a. Solutions for testing elimination of  $^{98}\text{Mo}^{16}\text{O}$  interference on  $^{114}\text{Cd}$ :
    - i. Base urine in diluent (1 + 9).

In a 50 mL lot screened or acid-washed polypropylene tube, prepare a 50 mL portion of working calibrator 0 as described in Table 8 in Appendix C (multiply volumes by 5).

- ii. Base urine in diluent (1 + 9) + 0.24 µg/L Cd

In a 50 mL lot screened or acid-washed polypropylene tube, prepare a 50 mL portion of working calibrator 2 as described in Table 8 in Appendix C (multiply volumes by 5).



iii. Base urine in diluent (1 + 9) + 300 µg/L Mo

In a 50 mL lot screened or acid-washed polypropylene tube, prepare a 50 mL portion of working calibrator 0 as described in Table 8 in Appendix C (multiply volumes by 5).

Add 0.015 mL of 1000 mg/L Mo.

iv. Base urine in diluent (1 + 9) + 0.24 µg/L Cd + 300 µg/L Mo

In a 50 mL lot screened or acid-washed polypropylene tube, prepare a 50 mL portion of working calibrator 2 as described in Table 8 in Appendix C (multiply volumes by 5).

Add 0.015 mL of 1000 mg/L Mo.

b. Solutions for testing elimination of  $^{39}\text{K}^{16}\text{O}$  interference on  $^{55}\text{Mn}$ :

i. Base urine in diluent (1 + 9)

In a 50 mL lot screened or acid-washed polypropylene tube, prepare a 50 mL portion of working calibrator 0 as described in Table 8 in Appendix C (multiply volumes by 5).

ii. Base urine in diluent (1 + 9) + 0.3 µg/L Mn

In a 50 mL lot screened or acid-washed polypropylene tube, prepare a 50 mL portion of working calibrator 2 as described in Table 8 in Appendix C (multiply volumes by 5).

iii. Base urine in diluent (1 + 9) + 400 µg/L K

In a 50 mL lot screened or acid-washed polypropylene tube, prepare a 50 mL portion of working calibrator 0 as described in Table 8 in Appendix C (multiply volumes by 5).

Add 0.02 mL of 1000 mg/L K

iv. Base urine in diluent (1 + 9) + 0.3 µg/L Mn + 400 µg/L K

In a 50 mL lot screened or acid-washed polypropylene tube, prepare a 50 mL portion of working calibrator 2 as described in Table 8 in Appendix C (multiply volumes by 5).

Add 0.02 mL of 1000 mg/L K.

Store at ambient temperature and prepare as needed. Label appropriately, i.e. "Store at ambient temperature", preparation date, expiration date one year from preparation date, and preparer's initials.

ii. DRC optimization (axial field voltage):

1. Purpose: Use as necessary to verify the elimination of crosstalk phenomenon. The crosstalk phenomenon will present itself as increased sensitivity for an ion of interest in the presence of greater population of spectator ions in the DRC cell. Elimination is verified when the ratio of the intensity for iridium in standard 5 versus standard 0 is the same ( $1.00 \pm 0.05$ ).
2. Content: Working calibrators 0 and 5 (see Section 6.e.iv).
3. Preparation and Storage: See section 6.e.iv.

iii. For dual detector calibration:

1. Purpose: Use as necessary to perform the dual detector calibration if any element exceeds 1,000,000 cps for calibrator 5 (typically Sr).
2. Content: Dilutions of single element or special mix stock standards in 2% (v/v) HNO<sub>3</sub>. Recommended elements include: Ba, Cs, Co, Pb, Mo, Sr, Sn. Other elements can be added as required for optimal instrument performance (esp. if measured intensities approach 500,000 cps in highest working calibrator).
3. Preparation & storage: To prepare elements of interest at 200 µg/L in 2% (v/v) HNO<sub>3</sub>:
  - a. Partially fill a 50 mL lot screened or acid-washed polypropylene tube with 2% (v/v) HNO<sub>3</sub>,
  - b. Add a 0.1 mL of 100 µg/mL special mix standard.
  - c. Add 0.01 mL of any additional 1,000 µg/mL single element stock standard desired to be added.
  - d. Dilute to the 50 mL mark with 2% (v/v) HNO<sub>3</sub>.
  - e. Label appropriately and store at ambient temperature. Expiration date is one year from preparation date.

## 7. Analytical instrumentation setup

(See Section 5 for details on hardware used, including sources)

### A. Instrumentation and equipment setup:

#### i. Configuration for liquid handling

1. FAST valve setup: See Appendix B, Figure 1 for diagram and Section 5.b "FAST / ESI SC4-DX autosampler accessories" for source information.

- a. Port 1: sample loop (white nut).
  - b. Port 2: 0.5 mm ID probe (red nut) for carrier solution.
  - c. Port 3: nebulizer line (green nut) for transfer of liquid to nebulizer.
  - d. Port 4: sample loop (white nut).
  - e. Port 5: 0.8 mm ID probe (blue nut) for diluted samples.
  - f. Port 6: vacuum line (black nut).
2. Carrier solution uptake: Use peristaltic pump to control uptake flow rate of carrier solution to the SC-FAST valve. Use of a 'peristaltic to Teflon tubing adapter' for prevents damage to small i.d. tubing when making connections (see consumables descriptions in Section 5.b).
  3. Spray chamber waste removal: Use of a 'peristaltic to Teflon tubing adapter' prevents damage to small i.d. tubing when making connections (see consumables descriptions in Section 5.b).
    - a. Between spray chamber and peristaltic tubing:
      - i. Spray chambers with threaded connection: Use vendor-supplied threaded connector on base of chamber, connecting tubing directly to peristaltic pump tubing through a PEEK adapter or directly.
      - ii. Spray chambers without threaded connection: Use of a specialized push-on connectors available from various vendors (like UFT-075 from Glass Expansion, Pocasset, MA) are preferred for safety reasons to direct connection of PVC tubing (e.g. 1/8" i.d. x 1/4" o.d.).
    - b. Between peristaltic pump tubing and waste container: Connect 1/8" i.d. x 1/4" o.d. PVC tubing to the white / black peristaltic pump tubing using a tubing connector (PerkinElmer item # B3140715). Place the free end of the PVC tubing through the lid of the waste jug (be sure it is secure). Place the waste container in a deep secondary containment tray in case of overflow.
  4. Rinse solution for autosampler:
    - a. Rinse solution jug: Leave one of the caps on the top of the rinse jug loose to allow air venting into the jug as liquid is removed. Otherwise the jug will collapse on itself as the liquid is removed and a vacuum is created inside. Use secondary containment tray.
    - b. Rinse solution uptake to autosampler rinse station: Use tubing of different lengths and inner diameters between the rinse solution container and the autosampler rinse station to control uptake rate of rinse solution. These can be obtained from the autosampler manufacturer, their distributors, or custom built in the lab. Optimize these factors along with fill time in the software so that waste of rinse solution is minimized and rinse station does not go empty.
    - c. Autosampler rinse station waste removal: Gravity drain of waste to the waste container is sufficient. Use minimum drain tubing to make this connection. If this tube is too long, the rinse station will not drain properly.

## ii. Gas delivery and regulation

1. ICP-MS modifications:
  - a. Plastic tubing between mass flow controllers and dynamic reaction cell have been replaced with stainless steel. Stainless steel tubing is preferred between the reaction gas cylinder / regulator and the back of the ICP-MS instrument.
  - b. A second mass flow controller is used (channel B) that does not send the DRC gas through a 'getter'.
2. Argon gas: Used for various ICP-MS functions including plasma and nebulizer.
  - a. Regulator for argon source (if a dewar): Set delivery pressure of this regulator at least 10 psi higher than the delivery pressure of the step-down regulator to allow for pressure drop across tubing that stretches to the instrument.
  - b. Step down regulator (if source of argon is a bulk tank): Place this single stage regulator in the lab so that incoming argon pressure can be monitored and adjusted. Set delivery pressure to 10 psig above the delivery pressure of the filter regulator on the ICP-MS.
  - c. Filter Regulator at ICP-MS: Single stage "argon regulator filter kit" supplied with the ICP-DRC-MS. Set the delivery pressure depending on the instrument setup:
    - i. ELAN with a 0-60psi gauge on the filter regulator: 52±1 psi when plasma is running (need 0-150 psi regulator if using a PolyPro or PFA nebulizer made by Elemental Scientific Inc).
    - ii. ELAN or NexION with a 0-150psi gauge on the filter regulator: 90-100 psi when plasma is running.
3. Oxygen (99.999+%) gas: Used for dynamic reaction cell interference removal from cadmium and manganese isotopes.
  - a. Connect to DRC channel B.
  - b. Set the delivery pressure of regulator to 5-7 psig when gas is flowing.

iii. Chiller / Heat Exchanger: If using refrigerated chiller, set temperature control to approximately 18 °C.

#### **B. Parameters for Instrument and Method:**

See Tables and Figures in Appendix C for a complete listing of the instrument and method parameters and software screen shots.

## **8. The run: quality, execution, evaluation, and reporting**

### **A. Bench QC, reference materials and calibration verification:**

#### **i. Bench "QC":**

Analysis of bench QC permits assessment of methodological imprecision, determination of whether the analytical system is 'in control' during the run, and assessment of time-associated trends. Bench QC pool analyte concentrations in this method span the analyte concentration range of the calibrators. Before QC materials can be used in the QC process,

they quality control limits are calculated based on concentration results obtained in at least 20 separate runs (see Section 11.c). In each analytical run the analyst will test each of the bench QC materials two times, subjecting them to the complete analytical process. Bench QC pool samples are analyzed first in the run after the calibrators but before any patient samples are analyzed. This permits making judgments on calibration linearity and blank levels prior to analysis of patient samples. The second analysis of the bench QC pools is done after analysis of all patient samples in the run (typically 20-30 patient samples total when analyzing for all elements in the method) to ensure analytical performance has not degraded across the time of the run. If more patient samples are analyzed on the same calibration curve after the second run of the bench QC, all bench QC must be reanalyzed before and after the additional samples. For example, the schemes shown in Table 6 in Appendix C are both acceptable ways to analyze multiple consecutive “runs”.

**ii. Reference materials:**

Use standard reference material (SRM, e.g. SRM 2668 levels 1 and 2) from the National Institute of Standards and Technology (NIST) to verify method accuracy. Use previously characterized samples from proficiency testing program or commercially-produced reference materials when NIST SRMs are unavailable.

**iii. Calibration verification:**

The test system is calibrated as part of each analytical run with NIST-traceable calibration standards. These calibrators, along with the QCs and blanks, are used to verify that the test system is performing properly.

**B. Perform, evaluate and report a run:**

**i. Starting the equipment for a run:**

1. Power on the computer, printer, and autosampler, and instrument computer controller.
2. Peristaltic pump: Set proper tension on peristaltic pump tubing.
3. Software: Start software for the ICP-MS and autosampler control.
4. Daily pre-ignition maintenance checks: Perform and document daily maintenance checks (e.g., Ar supply pressure, interface components cleanliness and positioning, interface pump oil condition, vacuum pressure, etc.).
5. Place probe in adequate volume of carrier or rinse solution: If using an ESI FAST, manually place carrier probe into carrier solution. If not, send the autosampler probe to a rinse solution (e.g. autosampler rinse station).
6. Start the plasma
7. Start the peristaltic pump: Start the pump running slowly, making sure that the rotational direction is correct for the way the tubing is set up.

8. Warm-up time: Allow warm-up time suggested by the manufacturer for the ICP-MS (e.g. RF generator) after igniting the plasma. There will be another warm-up time (or “stability time”) for the DRC later in this procedure.
9. Daily performance check: Perform and document a daily performance check and any optimizations necessary. Save new parameters to the “default.tun” and “default.dac” files.
10. DRC Stability time: Best analyte-to-internal standard ratio stability is typically observed after 1-1.5 hours of analysis of urine samples using the DRC mode method (~12 measurements of the 15 element panel). Prepare 50mL<sup>+</sup> of a calibrator (e.g. standard 2) to be analyzed repeatedly before the beginning of the run to achieve a stable analyte-to-internal standard ratio. Time to reach stability is instrument-specific and learned from performance of runs. See Table 7 in Appendix C for example of setup in the Samples / Batch window and Table 8 in Appendix C for details of making a working standard.
11. Readying the instrument for quick-start analysis: Leave the plasma running to eliminate the need for an initial instrument warm-up period and / or a DRC stabilization period as long as appropriate planning is made for sufficient solution supply and waste collection. Analysis of conditioning samples (diluted urine matrix) can also be scheduled to occur at roughly a predetermined time. Accomplish this by setting up multiple sample analyses with extended rinse times (e.g. one 15 element analysis with a 1400s rinse time will take approximately 30 minutes to complete). Initial samples would be non-matrix, while final samples would be diluted matrix for conditioning. If running a DRC-only method during these scheduled analyses, the ICP-MS will remain in DRC-mode for approximately 45 minutes without depressurizing the cell.
12. Software setup for Analysis: Verify & set up the correct files and data directories for your analysis (See Table 1 in Appendix C for defaults). Update the software to reflect the current sample set. Use a bar code scanner to input data whenever possible. See Table 1 in Appendix C for times and speeds. There are two method files for this one method (see Table 1 in Appendix C). It is necessary to use both to accomplish each run because the current PerkinElmer software will not allow for more than one blank per method file. The ONLY DIFFERENCE between these two files is on the Sampling tab where one lists the autosampler positions of the urine blank and urine calibrators (the “urblk” method file) and the other lists the autosampler position of the aqueous blank (the “aqblk” method file). The ONLY TIME when it matters which of these files is used is when the measurement action *includes* “Run blank” or “Run standards”. When the measurement action is only ‘run sample’, it does not matter whether the “urblk” or “aqblk” method file is used. Analysts typically follow the pattern below, however, for the sake of consistency and as a reminder of which blank must be used for which type of sample. See Table 7 in Appendix C. Use to analyze the initial urine blank (blank for the calibration curve), the urine calibrators, and the urine blank checks at the very beginning of the run. The urine blank method defines the autosampler location of the urine blank and the urine calibrators. The “aqblk” method file

must be used to analyze all QC materials and patient samples. The aqueous blank method defines the aqueous blank in autosampler location.

**ii. Preparation of samples for analysis (See Table 8 in Appendix C)**

**i. Thaw urine samples; allow them to reach ambient temperature.**

1. Prepare the following solutions into pre-labeled containers benchtop automatic pipette or other volumetric sample transfer device. See Table 8 in Appendix C for a summary. Prepare samples in the cleanest environment available to prevent trace element contamination and an environment which provides personnel protection (e.g. Class II, Type A/B3 biological safety cabinet).
  - a. *Aqueous Blank*: Prepare at least two aqueous blanks. One will be the actual reagent blank for patient and QC samples and the other will be a backup (“Aqueous Blank Check”) in case the original aqueous blank is unusable.
  - b. *Calibrators*: Prepare the working calibrators (S0-S5). Prepare S0 in triplicate. One of these S0 preparations will be the zero standard (urine blank) for the calibrators; the other two will be analyzed twice after the last calibrator to collect run blank data that can be used to verify and document washout after the calibrators.
  - c. *Patient & QC Samples*: Before taking an aliquot for analysis, homogenize the sample (e.g. vortex for 3-5 seconds, or invert 5-10 times). After preparation, mix and cover diluted samples. Place prepared dilutions on the autosampler of the ICP-MS in the order corresponding to the sequence setup in the ICP-MS software. Ambient temperature storage of original samples for the work day is acceptable.

**iii. Start the analysis using the ICP-MS software.**

**iv. Monitor the analysis in real-time as much as possible. If necessary, leave the run to complete itself unattended as long as appropriate planning is made for either overnight operation or Auto Stop (see below). Monitor the analysis for the following:**

1. *Verify proper operation of the instrument* (proper loop filling, sample reaching nebulizer in correct timing, autosampler arm moving properly, etc . . .)
2. *Verify that background signal from instrument and reagents are low.* Helpful checks when diagnosing high background problems include:
  - a. *Water* ( $\geq 18$  Mohm·cm) to be used in Aq Blank Checks and dilutions.
  - b. Diluent before and after being flushed through the benchtop automatic pipette. If contamination is observed from the pipette, flush the pipette with  $\geq 500$  mL of nitric acid solution ( $\leq 5\%$  (v/v)  $\text{HNO}_3$ ) and retest.
  - c. Comparison with other instruments.
3. *Verify analyte / internal standard ratio stability*: The net intensity (analyte / internal standard ratio) of the measurements made while stabilizing the UCT can be evaluated to determine the readiness of the system to begin analysis.

Continual trending in this ratio indicates that unwanted instrument drift will occur within the run.

4. *Evaluate the Axial Field Voltage (AFV) optimized value.* Monitor the change between S0 and S5 for measured intensities (cps) of the internal standard iridium in DRC mode. If the percent difference between the iridium intensities is greater than 5% (especially if greater in S5), then run the axial field voltage optimization. See Section 6.f.ii for preparation of optimization solutions.
5. *Verify calibration curves* meet  $R^2$  requirements (minimum of 0.98, typically 0.99 to 1.000).
6. *Verify bench QC results are within the acceptable limits.* If an analyte result for the beginning QC material(s) falls outside of the  $\pm 3SD$  limits, then the steps below are recommended. If these steps do not result in correction of the out-of-control values for QC materials, consult the supervisor for other appropriate corrective actions.
  - a. Evaluate the blank results.
  - b. Evaluate the reproducibility of the 3 replicates within the measurements.
  - c. Evaluate the consistency of the internal standard across the measurements (esp. the calibrators).
  - d. Evaluate calibration curves. If a particular calibrator is obviously in error, it can be re-analyzed as a sample (old or new dilution) and incorporated into the curve through data reprocessing as a calibrator. As a last resort, a single calibration point per analyte between and including S2 and S4 can be removed from the curve. Follow up on repeated problems with calibrators with appropriate corrective actions (e.g. re-preparation of intermediate working standards or troubleshooting instrument parameters).
  - e. Prepare a fresh dilution of the failing QC material (same vial) and reanalyze it to see if the QC dilution was not properly made.
  - f. Prepare a fresh dilution of the failing QC material (unused vial) and analyze it to see if the QC vial had become compromised.
  - g. Prepare and analyze new working calibrators.
  - h. Test a different preparation of intermediate working calibration standards.
7. Verify good precision among replicates of each measurement.
8. *Verify consistent measured intensities of the internal standards.* Some sample-to-sample variations are to be expected, however, intensities drifting continuously in one direction resulting in failing results for ending QC indicate the instrument needs additional pre-conditioning before the run or environmental conditions are changing too much around the instrument.
9. *Verify elevated patient results.* Refer to Figure 17 in Appendix C for flowchart.



- a. Confirming an elevated concentration: Repeat for confirmation any sample having a concentration greater than the 1UB threshold. See Table 10 in Appendix C.
- b. Dilution of a sample to within the calibration range: Repeat in duplicate with extra dilution any sample having a concentration greater than the highest calibrator to bring the observed result within the concentration range of the calibrators. See Table 9 of Appendix C for high calibrator concentrations and validated extra dilutions.
- c. Confirming proper washout after an elevated sample: When monitoring the analysis in real-time, if an observed sample concentration is greater than the highest concentration tested for washout (see Table 10 in Appendix C), do the following to verify that the run is still in control for low concentration samples before proceeding with analysis.
  - i. Stop run following elevated sample
  - ii. Verify that the run is still in control for lower concentration samples before proceeding with analysis. Analyze 2 urine blank checks followed by a low bench QC washout check. If the low bench QC wash check is not in control (within  $\pm 3SD$  limits), repeat these 3 check samples until washout is verified before proceeding with analysis.

Example:

3018 UrBlkChk Wash1

3018 UrBlkChk Wash2

LUXXXXX Wash

- iii. If the run is not verified in-control for low concentration samples before the next samples are analyzed, see Section 8.b.vii.2. for directions.

- v. Overnight operation or using auto stop: Ensure sufficient solution supply and waste collection during unattended operation. Turn on the AutoStop feature of the ICP-MS software. Delay the shutdown at least 10 minutes (use peristaltic pump speed approximately that of the method wash) to rinse the sample introduction system of urine matrix before turning off the plasma. It will be necessary to replace the sample peristaltic pump tubing the next day since it will have been clamped shut overnight. Enable "Auto Start/Stop" is on the "AutoStop" tab of the Instrument window.
  1. ELAN specifics: Enable "Auto Start / Stop" is on the "AutoStop" tab of the Instrument window.
  2. NexION specifics: Enable AutoStop in on the Run List window. Select "Batch Completed" for Stop Criteria.
- vi. Records of results: Run results will be documented after each run in both electronic and paper form.
  1. Electronic records: Transfer data electronically to the laboratory information system. When keyboard entry must be used, proofread transcribed data after entry.

- a. Export data from the ICP-MS software using “original conditions” or files and folders used during the analysis. Use descriptive report filenames (e.g. 2005-0714a\_group55.txt). In the ELAN or NexION software under “Report Format” (METHOD window, REPORT tab) choose the “Use Separator” option, and under the “File Write” Section choose “Append.”
  - b. Move the generated .TXT data file to the appropriate subdirectory on the network drive where exported data are stored prior to import to the laboratory information management system.
  - c. Import the instrument file into the laboratory information system with appropriate documentation (e.g. instrument ID, analyst, calibration standards lot number, and run or sample specific comments).
2. Paper records: Run sheets must be documented with
- a. Analyst initials
  - b. Instrument ID
  - c. Date of analysis and run # for the day
- vii. Analyst evaluation of bench QC run results**: After completing a run, and importing the results into the laboratory information system, evaluate the run bench QC according to laboratory QC rules[6]. The QC limits are based on the average and standard deviation of the beginning and ending analyses of each of the bench QC pools, so it will not be possible to know if the run is in control until statistically reviewed.
1. Rules for bench quality control evaluation: The following are the CDC DLS QC rules for two QC pools per run with two or more QC results per pool.
    - a. If both QC run means are within  $2S_m$  limits and individual results are within  $2S_i$  limits, then accept the run.
    - b. If one of the two QC run means is outside a  $2S_m$  limit - reject run if:
      - i. Extreme Outlier – Run mean is beyond the characterization mean  $\pm 4S_m$
      - ii. 3S Rule - Run mean is outside a  $3S_m$  limit
      - iii. 2S Rule – Two or more of the run means are outside the same  $2S_m$  limit
      - iv. 10 X-bar Rule – Current and previous 9 run means are on same side of the characterization mean
    - c. If one of the 4 QC individual results is outside a  $2S_i$  limit - reject run in the cases stated below. Note: Since runs have multiple results per pool for 2 pools, the R 4S rule is applied within runs only.  $S_i$  = Standard deviation of individual results.  $S_m$  = Standard deviation of the run means.  $S_w$  = Within-run standard deviation.
      - i. Extreme Outlier – One individual result is beyond the characterization mean  $\pm 4S_i$
      - ii. R 4S Rule – Within-run ranges for all pools in the same run exceed  $4S_w$  (i.e., 95% range limit)

2. Implications of QC failures: If the DLS SAS program declares the run “out of control” for any analyte, use the following to determine the implications on usability of the data from the run.
  - a. 13 – 15 elements in the run
    - i. 1, 2 or 3 analytes “out of control”: ONLY the analytes which were “out of control” are invalid for reporting from the run.
    - ii. 4 or more analytes “out of control”: All results, regardless of analyte, are invalid for reporting from the run.
  - b. 4 – 12 elements in the run
    - i. 1 or 2 analytes “out of control”: ONLY the analytes which were “out of control” are invalid for reporting from the run.
    - ii. 3 or more analytes “out of control”: All results, regardless of analyte, are invalid for reporting from the run.
  - c. 3 elements in the run
    - i. 1 analyte “out of control”: ONLY the analyte which was “out of control” is invalid for reporting from the run.
    - ii. 2 or more analytes “out of control”: All results, regardless of analyte, are invalid for reporting from the run.
  - d. 1 – 2 elements in the run: ONLY the analyte which was “out of control” is invalid for reporting from the run.

**viii.** Analyst evaluation of patient results:

1. Elevated concentrations: Refer to Figure 17 in Appendix C for flowchart.

- a. Results greater than the first upper boundary (1UB): Confirm by repeat analysis of a new sample preparation concentrations observed greater than the “first upper boundary” (defined in the laboratory database as the “1UB”). Report the first analytically valid result, as long as the confirmation is within 10%. Continue repeat analysis until a concentration can be confirmed.
  - b. Results greater than the second upper boundary (2UB): Report any patient results confirmed to be greater than the second upper boundary (2UB) as an “elevated result”.
  - c. Results greater than highest calibrator: Samples that exceed the high calibrator must be prepared with minimum extra dilution in duplicate to bring the observed result within the calibration range ( $\leq S5$ ). Report the first analytically valid result (i.e. the first one within the calibration range), as long as the confirmation is within 10%. Continue repeat analysis until a concentration can be confirmed.
  - d. Concentrations requiring verification of washout: Following a result greater than the highest concentrations tested for washout (see Table 10 of Appendix C) do the following:
    - i. If the run was determined to be in-control for low concentration samples before the next samples were analyzed, no further action is required.
    - ii. If the run was not determined to be in-control for low concentration samples before the next samples were analyzed confirm by re-analysis the results for the 2 samples immediately following the elevated sample. Report the results if they confirm the initial results within  $\pm 10\%$  or  $\pm 3SD$  of the low bench QC, whichever is greater.
2. Unacceptable reproducibility: If the range of the three replicate readings (maximum replicate concentration value - minimum replicate concentration value) for a single sample analysis is greater than the range maximum criteria listed in Table 10 in Appendix C **and** the range of the three replicate readings is greater than 10% of the observed concentration, do not use the measurement for reporting. Repeat the analysis of the sample.
- ix. Submitting final work for review: All analyses must undergo quality control and quality assurance review. After appropriately documenting the run in the laboratory information system (e.g. sample and run QC, and run and sample comments), inform the first level reviewer of the completed work and submit any printed documentation.

## 9. Routine equipment maintenance and data backups

### A. Equipment maintenance:

Analysts are expected to regularly evaluate the need for, and when necessary perform, cleaning, replacement, or re-positioning of components in ICP-MS the sample introduction system, interface, ion optics region, and equipment required resources (e.g. autosampler, exhaust, compressed gases, and coolant). Frequency of equipment maintenance will be dependent on instrument throughput. Maintenance activities will be documented in the instrument logbook.

- i. Parameter optimizations: Analysts are expected to optimize instrument parameters.
- ii. Dual detector calibration: Perform dual detector calibration regularly (weekly or monthly) for any element exceeding 1,000,000 cps for calibrator 5. This is typically only Sr. The dual detector calibration solution is described in Section 6.f.iii.

**B. DRC optimizations:**

DRC conditions (cell gas flow rate and RPq value) can be verified by analyzing the DRC optimization solutions (see Section 6.f.i) as needed to ensure proper reduction of potential ICP-MS interferences.

**C. Data backup:**

Data on the instrument computer will be backed up via two backup routines. Files used and produced by the ICP-MS in analyzing samples will be backed up and kept a minimum of three years after analysis.

- i. Daily backups to secondary hard drive: Program automatic backups of the relevant computer files to occur each night onto a secondary hard drive to prevent loss of data from failure of primary hard drive.
- ii. Periodic backup: Backup relevant computer files periodically (e.g. 5 – 10 runs) either to secondary hard drive which is remote to the laboratory or to removable media which will be placed remote to the laboratory for retrieval in the case of catastrophic data loss inside the lab.

## 10. Reporting thresholds

**A. Reportable range of test results:**

Urine element concentrations are reportable in the range between the method LOD and the high calibrator times the maximum permitted extra dilution (see Table 9 of Appendix C). Above the high calibrator, extra dilutions are made of the urine sample to bring the observed concentration within the calibration range.

**B. Reference Range (normal range):**

In this method the 95% reference ranges (see Table 11 in Appendix C) for these elements in urine fall within the range of the calibrators.

**C. Action Levels:**

Due to the uncertainty of the health implications of elevated concentrations of many of the elements determined with this method, there is no routine notification for elevated levels of every analyte determined with this method. The present NRC standard for workplace removal is 15 µg/L of U in urine [7]. Other action levels for reporting to supervising physicians are determined on a study-by-study basis.

## 11. Method calculations

### A. Method limit of detection (LOD):

The method detection limits for elements in urine specimens are defined as 3 times  $S_0$ , where  $S_0$  is the estimate of the standard deviation at zero analyte concentration.  $S_0$  is taken as the y-intercept of a linear or 2<sup>nd</sup> order polynomial regression of standard deviation versus concentration (4 concentration levels of the analytes in urine each measured 60 times across at least a 2-month timeframe). Method LODs are re-evaluated periodically.

### B. Method limit of quantitation (LOQ):

The Division of Laboratory Sciences does not currently utilize limits of quantitation in regards to reporting limits [6].

### C. QC limits:

Before QC materials can be used in the QC process, they quality control limits are calculated based on concentration results obtained in at least 20 separate runs. It is preferable to perform separate analyses on separate days and using multiple calibrator lot numbers, instruments, and analysts to best mimic real-life variability. The statistical calculations are performed using the SAS program developed for the Division of Laboratory Sciences.

## 12. Alternate methods for performing test and storing specimens if test system fails:

If the analytical system fails, setup analysis on other ICP-MS instrument, if available. If no other instrument is available, store the specimens at  $\leq -20$  °C until the analytical system can be restored to functionality.

## 13. Method Performance Documentation

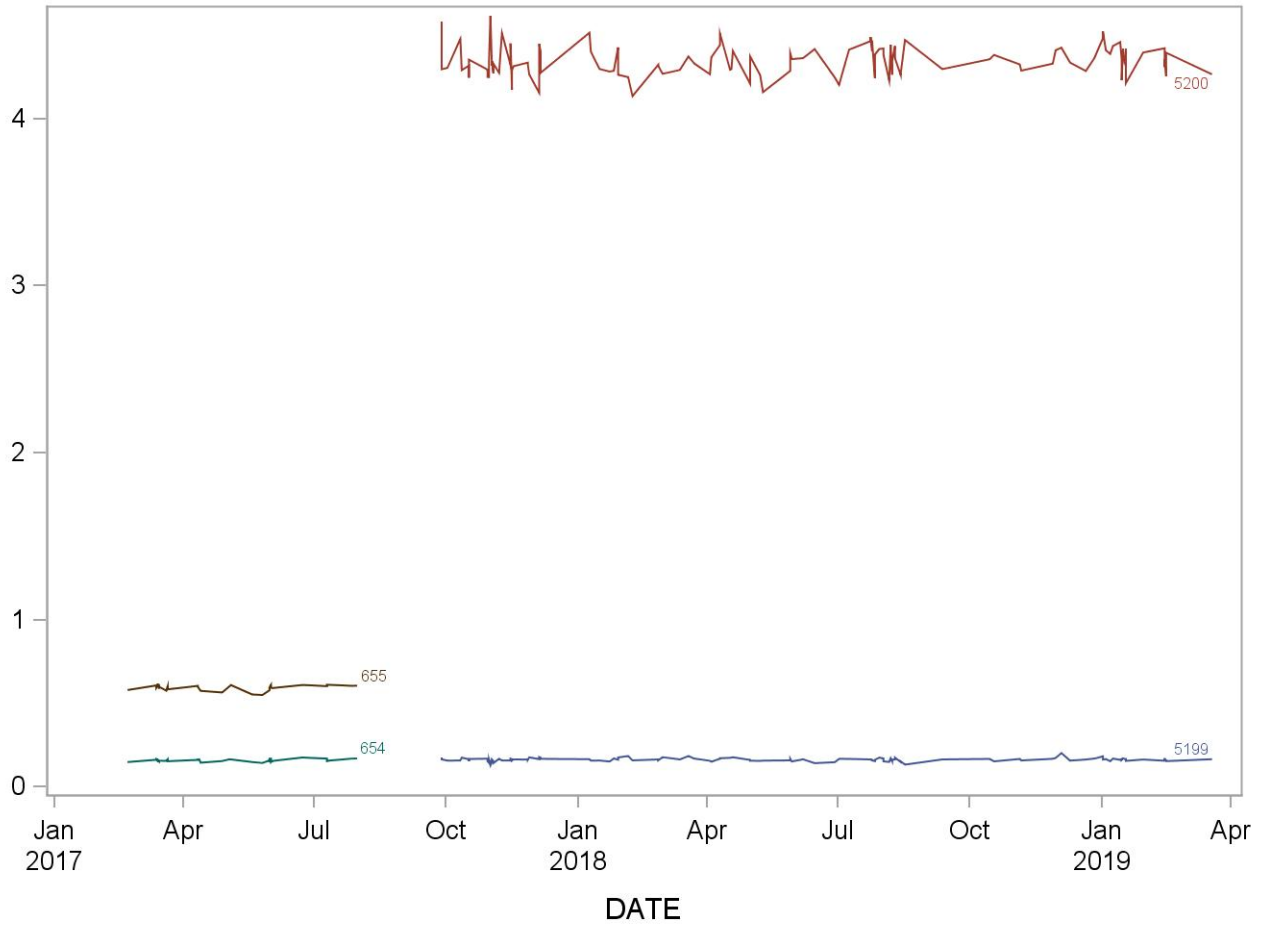
Method performance documentation for this method including accuracy, precision, sensitivity, specificity and stability is provided in Appendix A of this method documentation. **The signatures of the branch chief and director of the Division of Laboratory Sciences on the first page of this procedure denote that the method performance is fit for the intended use of the method.**

## 14. Summary Statistics and QC Charts

Please see following pages

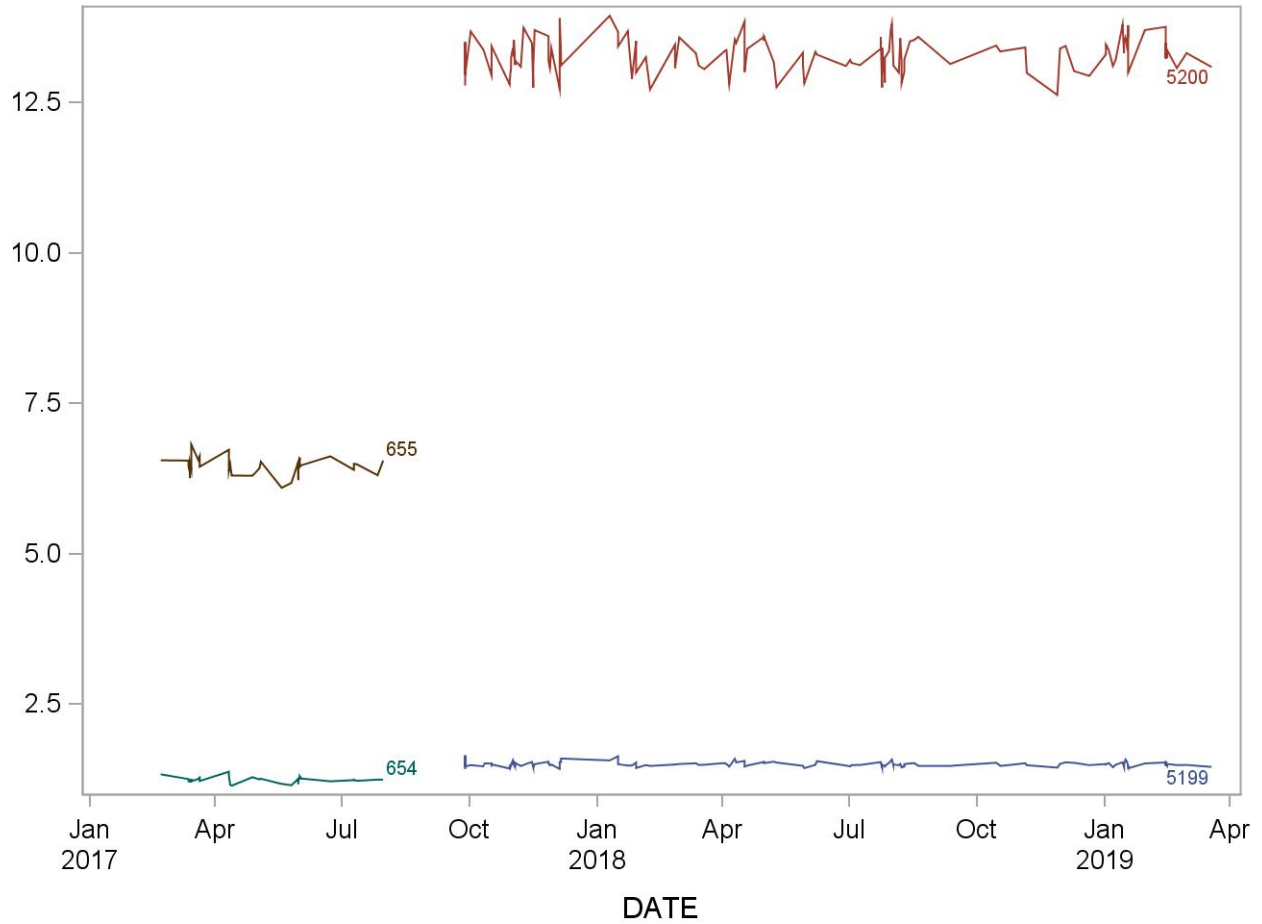
**2017-2018 Summary Statistics and QC Chart for Antimony, urine (ug/L)**

Lot	N	Start Date	End Date	Mean	Standard Deviation	Coefficient of Variation
654	35	21FEB17	31JUL17	0.1568	0.0081	5.2
655	35	21FEB17	31JUL17	0.5917	0.0167	2.8
5199	117	28SEP17	19MAR19	0.1609	0.0101	6.3
5200	117	28SEP17	19MAR19	4.3450	0.0893	2.1



### 2017-2018 Summary Statistics and QC Chart for Barium, urine (ug/L)

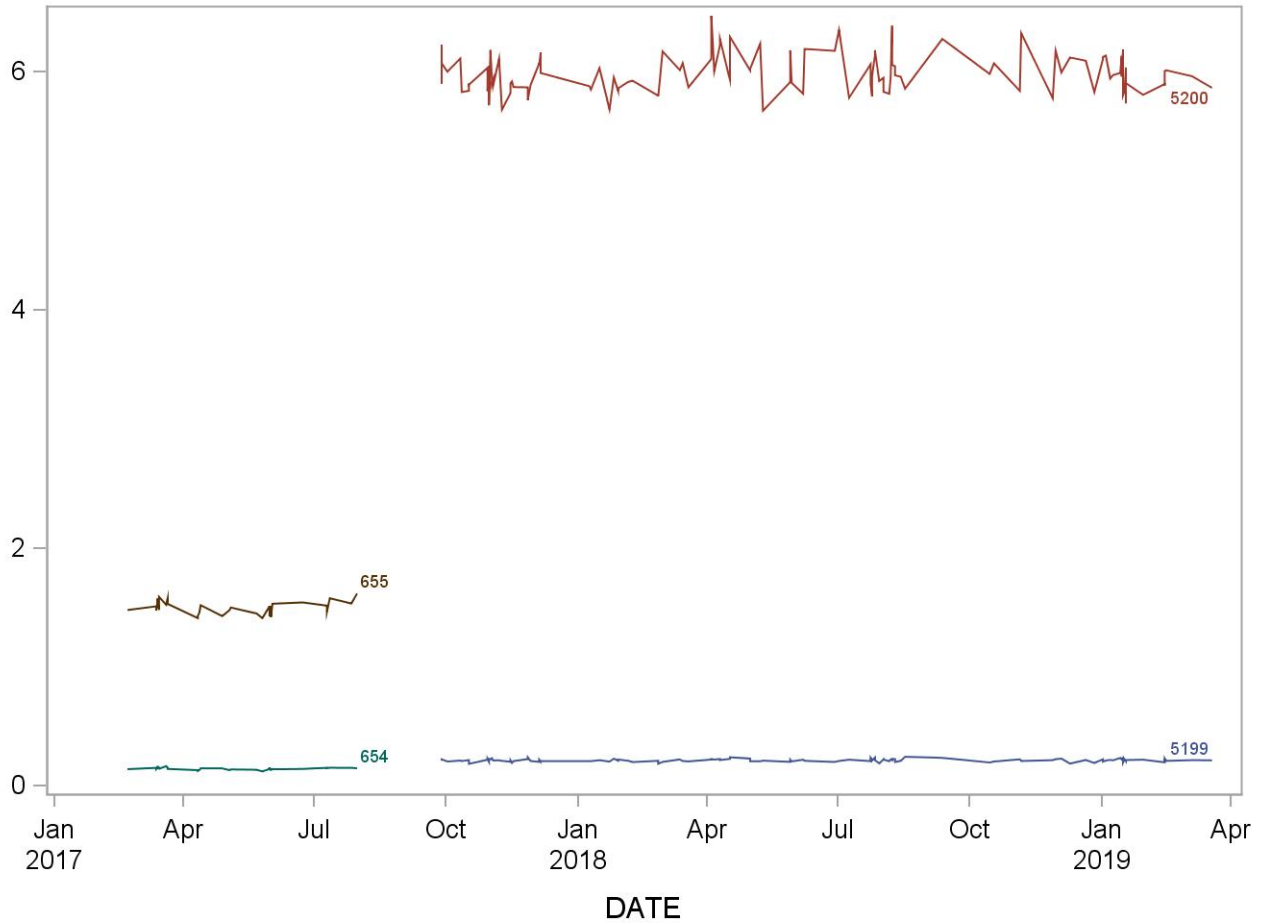
Lot	N	Start Date	End Date	Mean	Standard Deviation	Coefficient of Variation
654	34	21FEB17	31JUL17	1.2350	0.0488	4.0
655	34	21FEB17	31JUL17	6.4517	0.1515	2.3
5199	122	28SEP17	19MAR19	1.4941	0.0397	2.7
5200	122	28SEP17	19MAR19	13.2900	0.2910	2.2





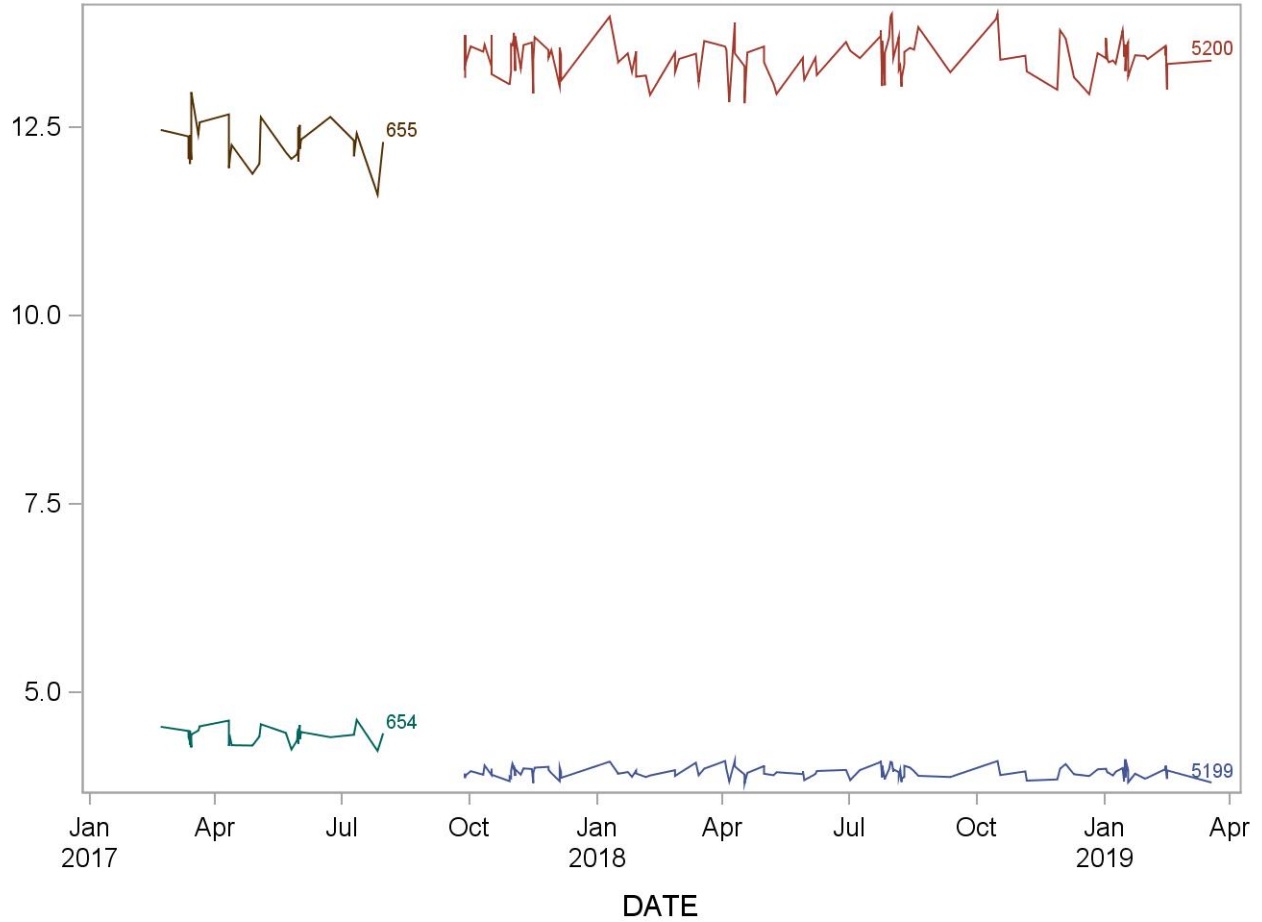
### 2017-2018 Summary Statistics and QC Chart for Cadmium, urine (ug/L)

Lot	N	Start Date	End Date	Mean	Standard Deviation	Coefficient of Variation
654	34	21FEB17	31JUL17	0.1420	0.0096	6.7
655	34	21FEB17	31JUL17	1.5001	0.0561	3.7
5199	116	28SEP17	19MAR19	0.2133	0.0119	5.6
5200	116	28SEP17	19MAR19	5.9841	0.1588	2.7



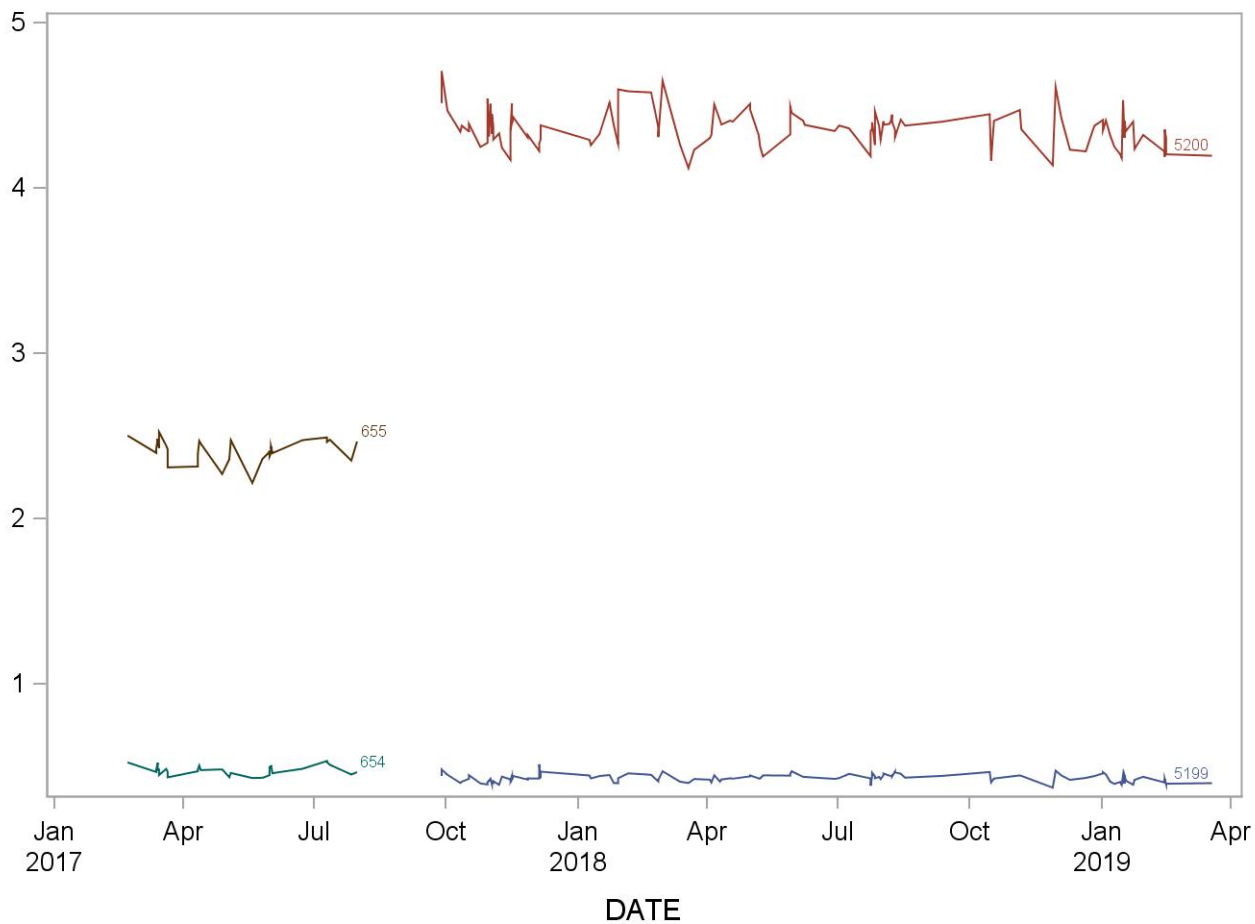
**2017-2018 Summary Statistics and QC Chart for Cesium, urine (ug/L)**

Lot	N	Start Date	End Date	Mean	Standard Deviation	Coefficient of Variation
654	35	21FEB17	31JUL17	4.4271	0.1075	2.4
655	35	21FEB17	31JUL17	12.2788	0.2690	2.2
5199	125	28SEP17	19MAR19	3.9387	0.0756	1.9
5200	125	28SEP17	19MAR19	13.4256	0.2545	1.9



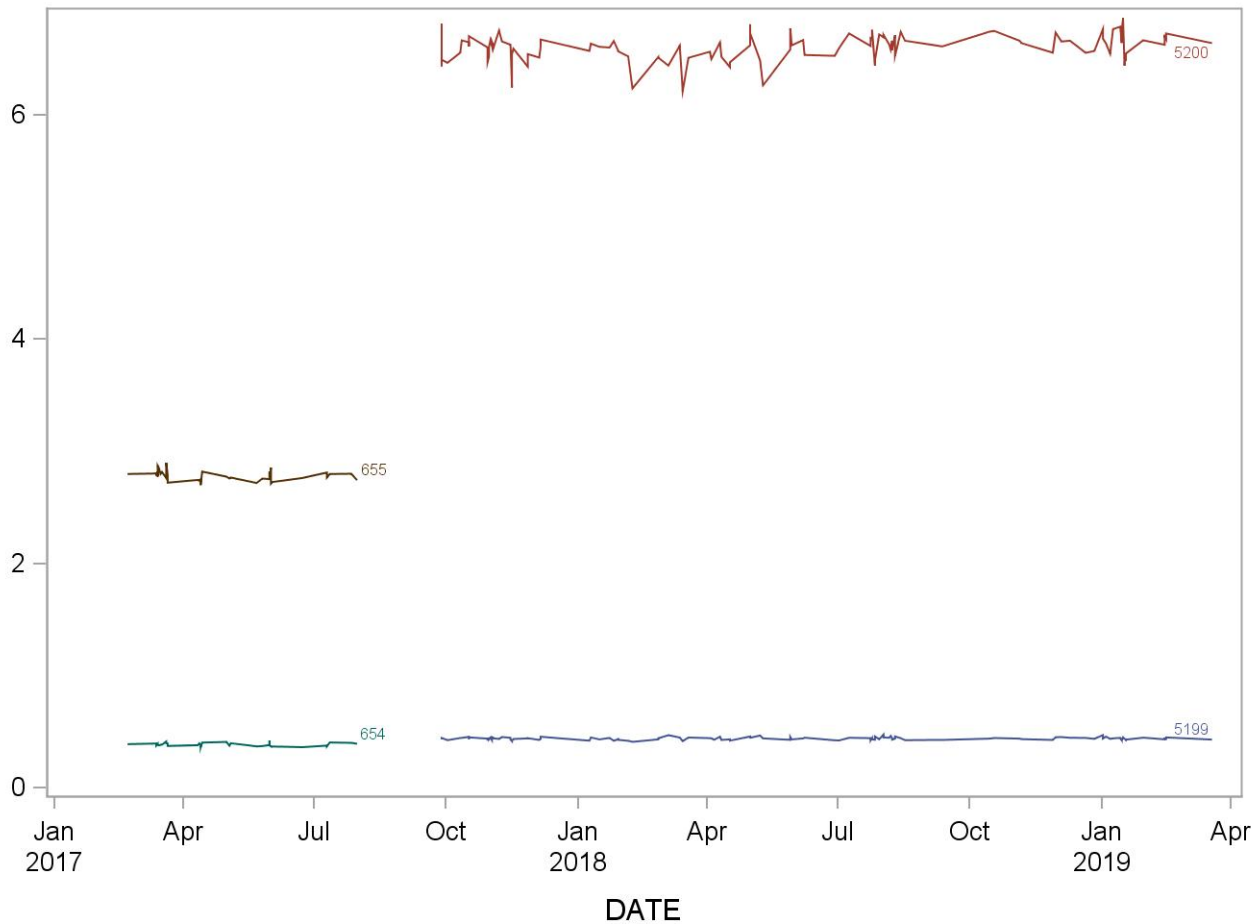
### 2017-2018 Summary Statistics and QC Chart for Cobalt, urine (ug/L)

Lot	N	Start Date	End Date	Mean	Standard Deviation	Coefficient of Variation
654	34	21FEB17	31JUL17	0.4817	0.0288	6.0
655	34	21FEB17	31JUL17	2.4173	0.0684	2.8
5199	118	28SEP17	19MAR19	0.4323	0.0243	5.6
5200	118	28SEP17	19MAR19	4.3606	0.1132	2.6



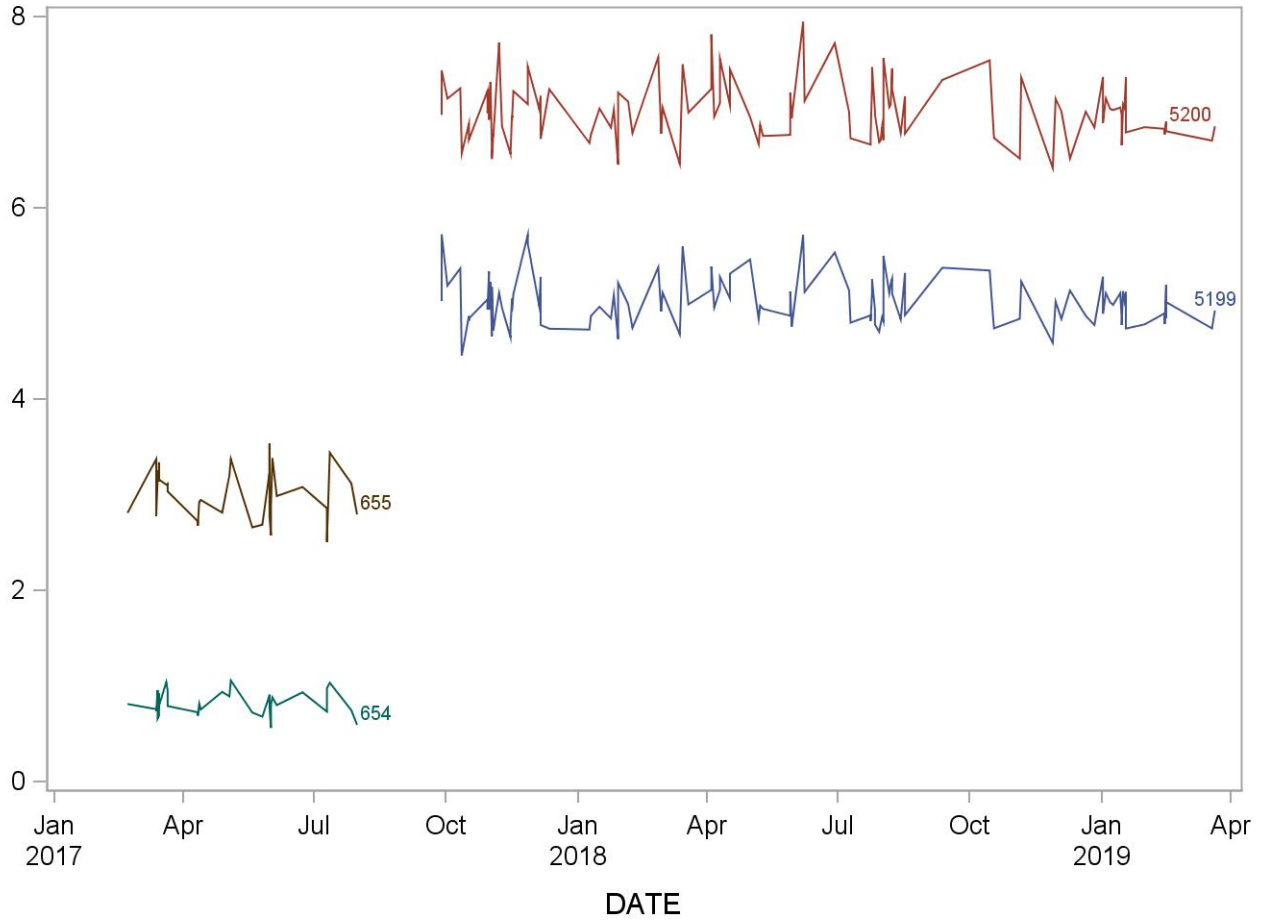
### 2017-2018 Summary Statistics and QC Chart for Lead, urine (ug/L)

Lot	N	Start Date	End Date	Mean	Standard Deviation	Coefficient of Variation
654	36	21FEB17	31JUL17	0.3841	0.0148	3.9
655	36	21FEB17	31JUL17	2.7774	0.0453	1.6
5199	115	28SEP17	19MAR19	0.4375	0.0124	2.8
5200	115	28SEP17	19MAR19	6.5984	0.1165	1.8



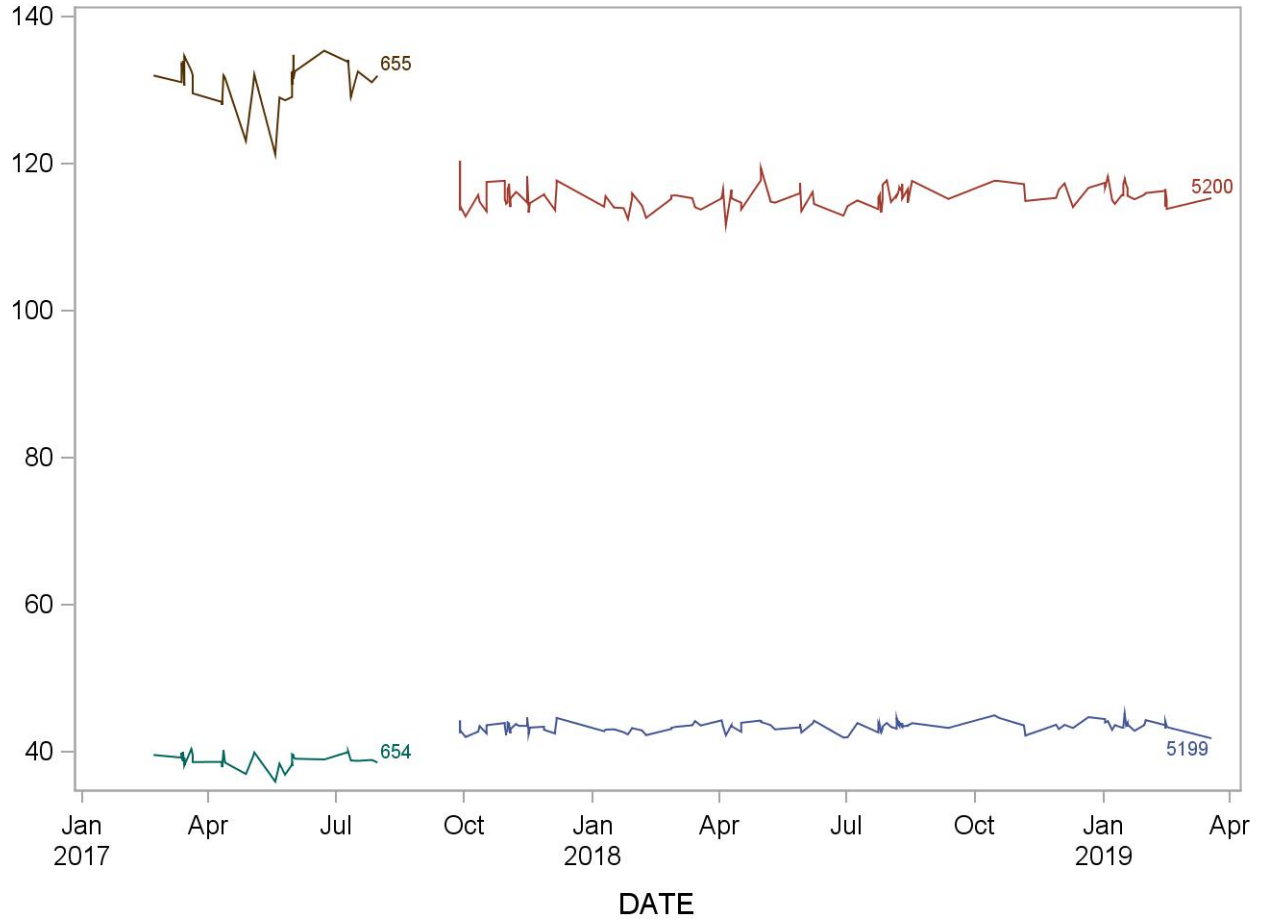
**2017-2018 Summary Statistics and QC Chart for Manganese, urine (ug/L)**

Lot	N	Start Date	End Date	Mean	Standard Deviation	Coefficient of Variation
654	35	21FEB17	31JUL17	0.8220	0.1240	15.1
655	35	21FEB17	31JUL17	3.0168	0.2670	8.9
5199	116	28SEP17	21MAR19	5.0165	0.2563	5.1
5200	116	28SEP17	21MAR19	7.0029	0.3115	4.4



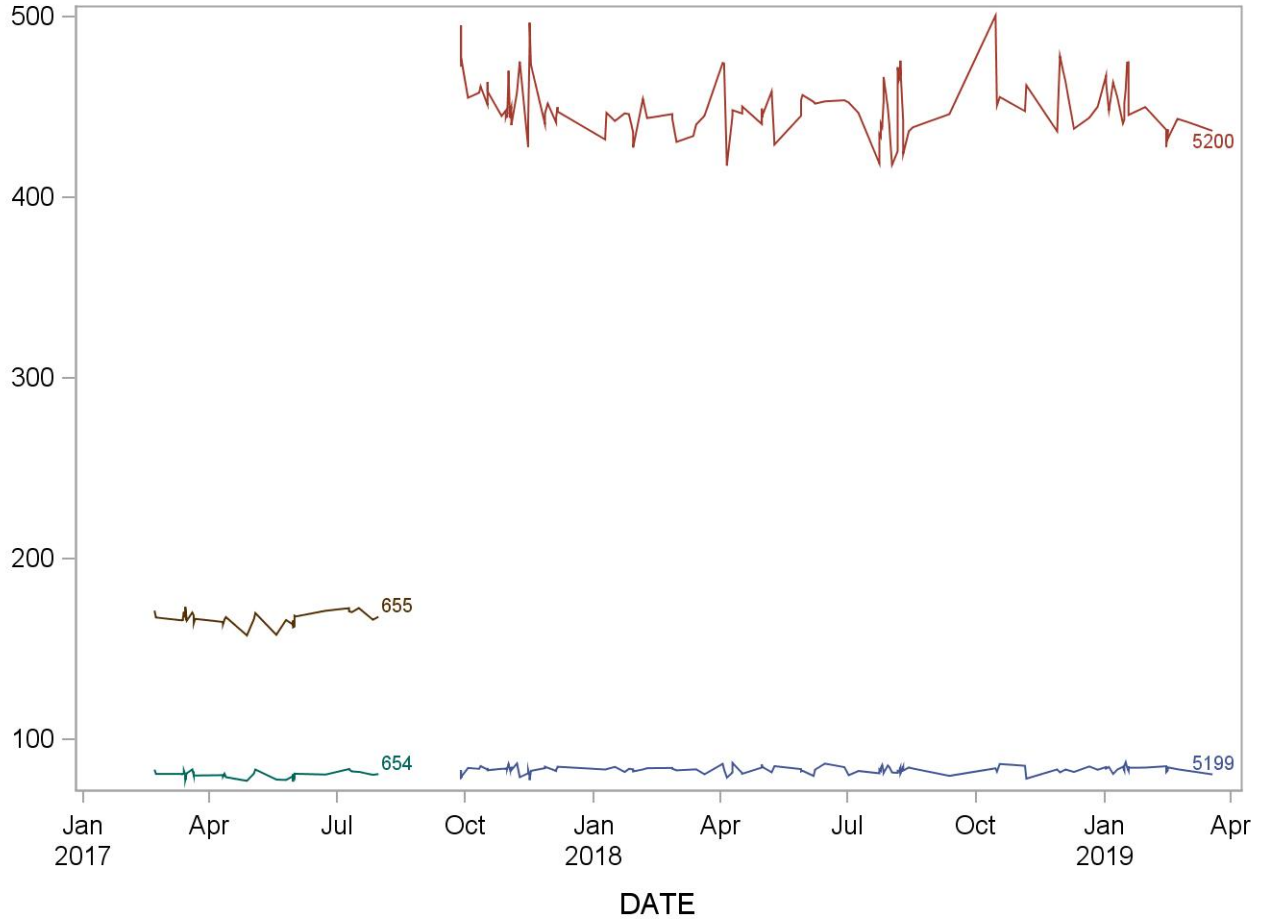
**2017-2018 Summary Statistics and QC Chart for Molybdenum, urine (ug/L)**

Lot	N	Start Date	End Date	Mean	Standard Deviation	Coefficient of Variation
654	37	21FEB17	31JUL17	38.893	0.961	2.5
655	37	21FEB17	31JUL17	131.301	2.919	2.2
5199	120	28SEP17	19MAR19	43.381	0.672	1.5
5200	120	28SEP17	19MAR19	115.563	1.517	1.3



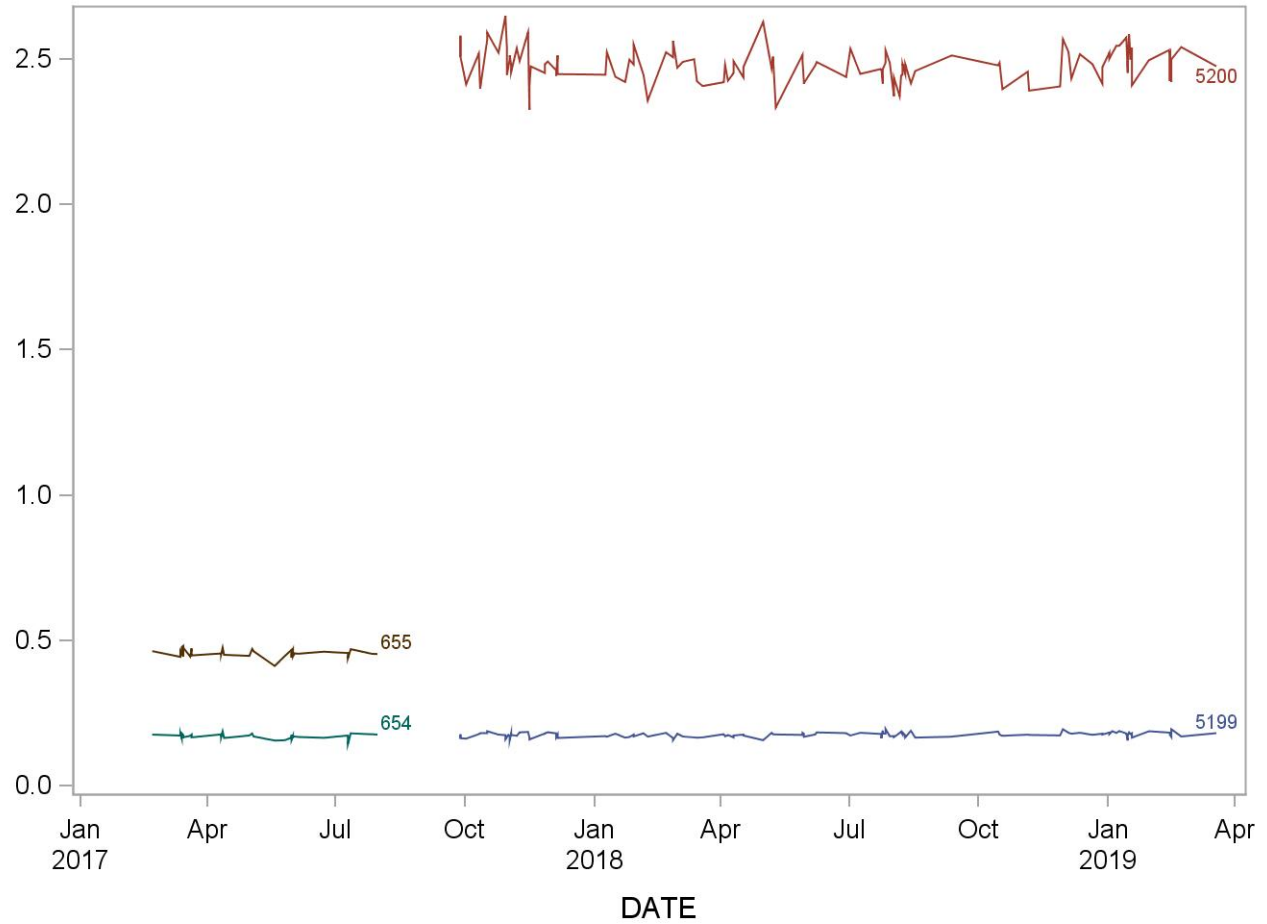
**2017-2018 Summary Statistics and QC Chart for Strontium, urine (ug/L)**

Lot	N	Start Date	End Date	Mean	Standard Deviation	Coefficient of Variation
654	39	21FEB17	31JUL17	80.664	1.759	2.2
655	39	21FEB17	31JUL17	167.258	3.643	2.2
5199	124	28SEP17	19MAR19	83.283	1.888	2.3
5200	124	28SEP17	19MAR19	449.821	15.638	3.5



### 2017-2018 Summary Statistics and QC Chart for Thallium, urine (ug/L)

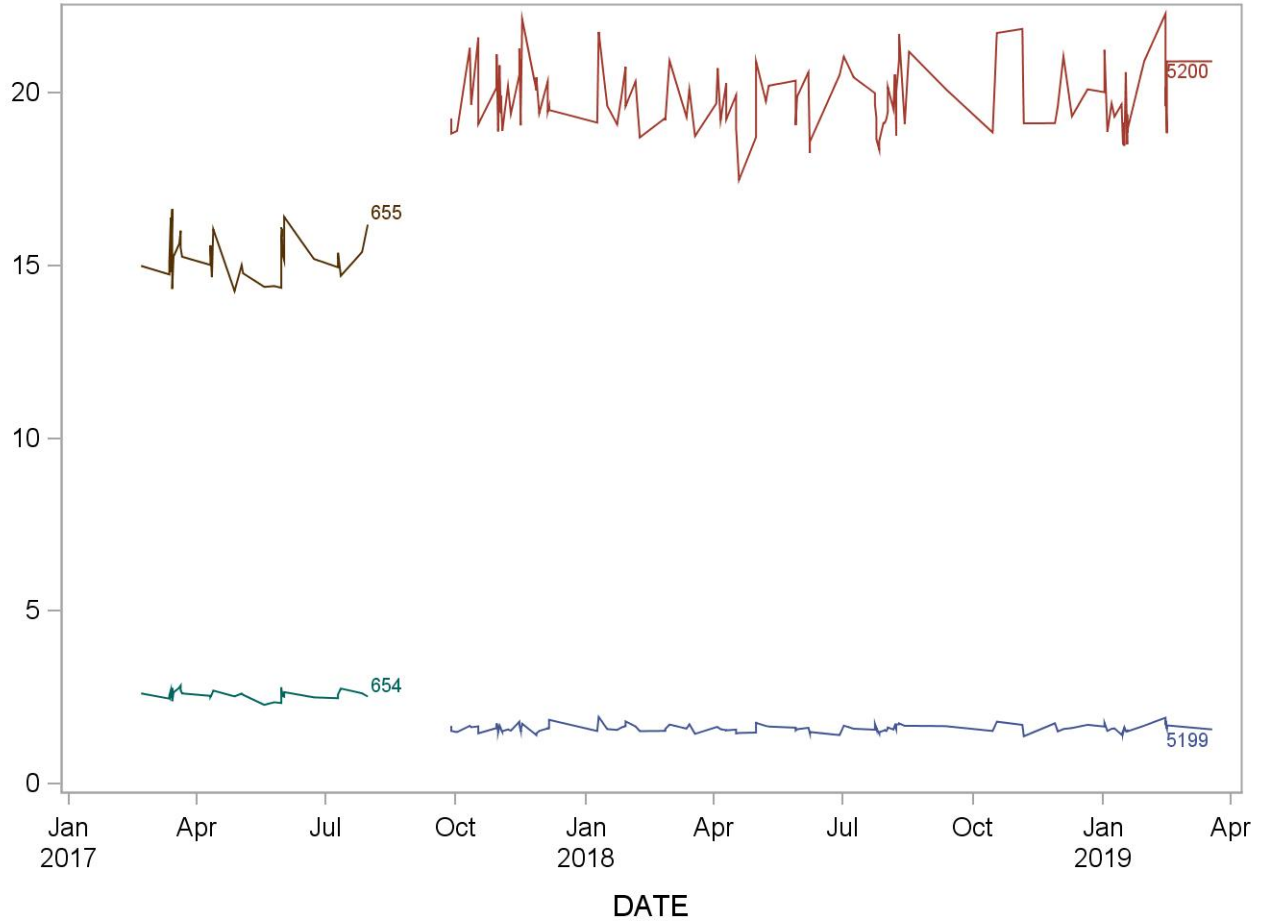
Lot	N	Start Date	End Date	Mean	Standard Deviation	Coefficient of Variation
654	36	21FEB17	31JUL17	0.1693	0.0084	5.0
655	36	21FEB17	31JUL17	0.4559	0.0133	2.9
5199	122	28SEP17	19MAR19	0.1748	0.0084	4.8
5200	122	28SEP17	19MAR19	2.4795	0.0588	2.4





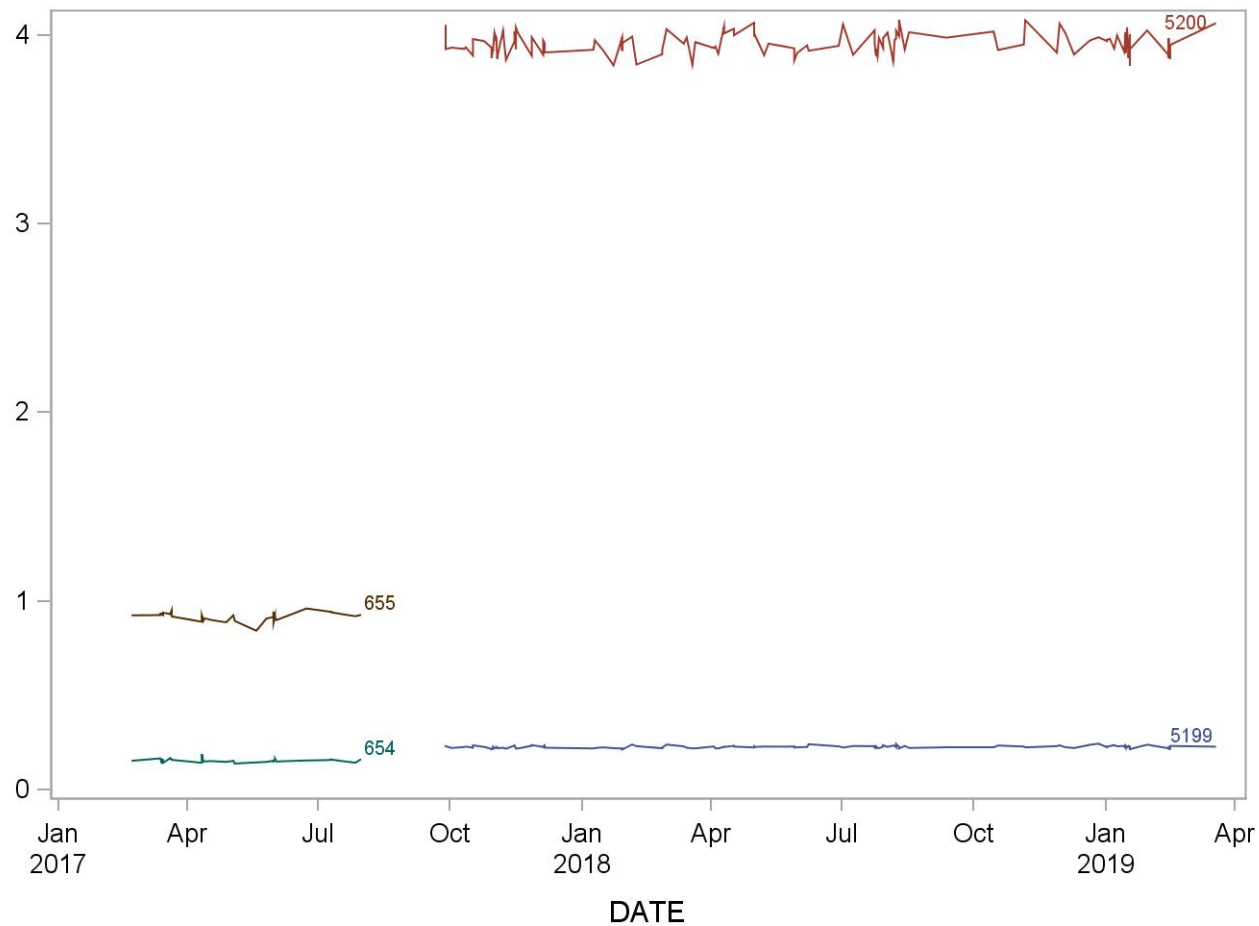
**2017-2018 Summary Statistics and QC Chart for Tin, urine (ug/L)**

Lot	N	Start Date	End Date	Mean	Standard Deviation	Coefficient of Variation
654	35	21FEB17	31JUL17	2.589	0.132	5.1
655	35	21FEB17	31JUL17	15.317	0.670	4.4
5199	119	28SEP17	19MAR19	1.604	0.108	6.7
5200	119	28SEP17	19MAR19	19.827	0.961	4.8



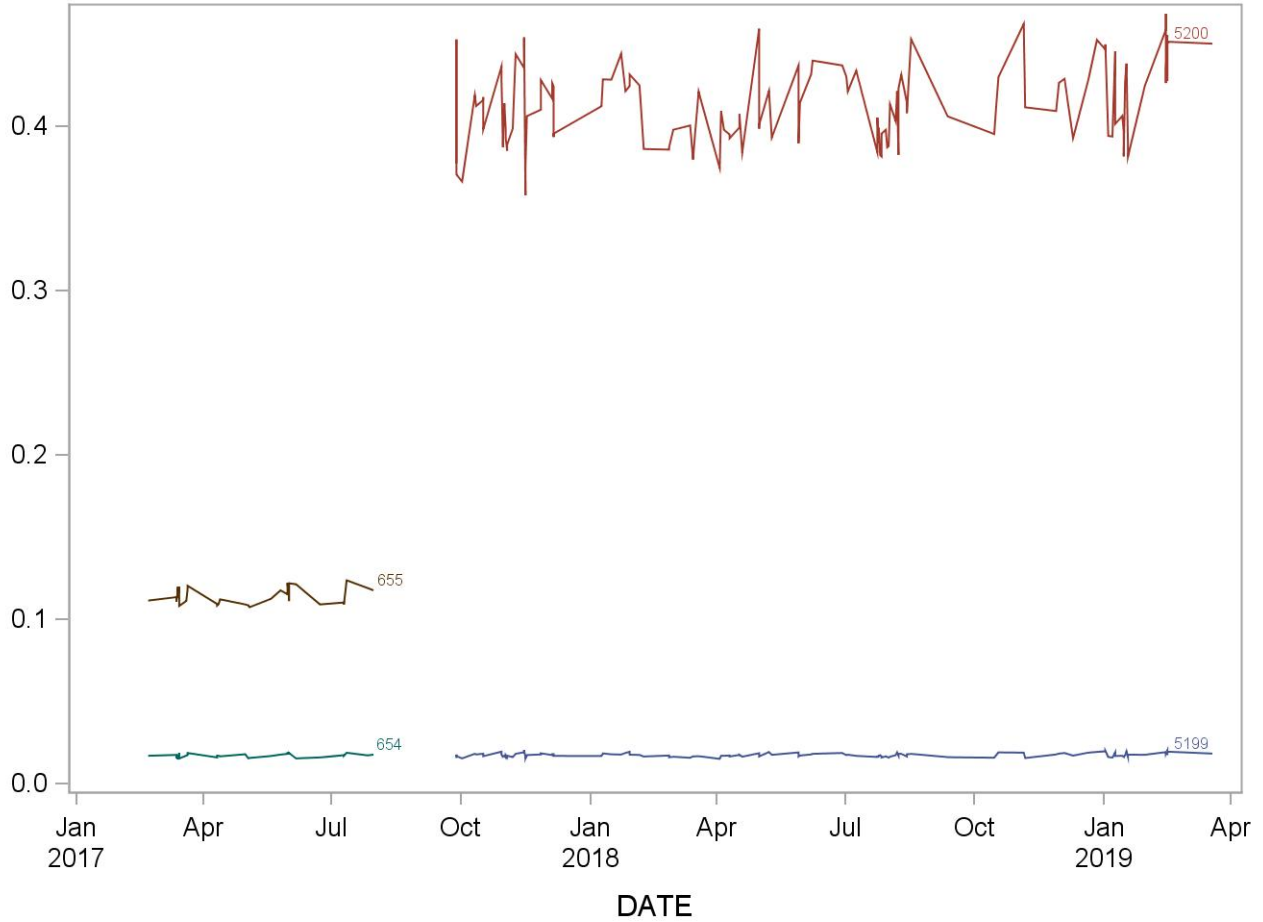
### 2017-2018 Summary Statistics and QC Chart for Tungsten, urine (ug/L)

Lot	N	Start Date	End Date	Mean	Standard Deviation	Coefficient of Variation
654	35	21FEB17	31JUL17	0.1542	0.0097	6.3
655	35	21FEB17	31JUL17	0.9189	0.0229	2.5
5199	117	28SEP17	19MAR19	0.2262	0.0066	2.9
5200	117	28SEP17	19MAR19	3.9535	0.0575	1.5



**2017-2018 Summary Statistics and QC Chart for Uranium, urine (ug/L)**

Lot	N	Start Date	End Date	Mean	Standard Deviation	Coefficient of Variation
654	34	21FEB17	31JUL17	0.01712	0.00116	6.8
655	34	21FEB17	31JUL17	0.11458	0.00513	4.5
5199	122	28SEP17	19MAR19	0.01743	0.00115	6.6
5200	122	28SEP17	19MAR19	0.41212	0.02405	5.8



## 15. Appendix A. Method Performance Documentation

### A. Accuracy

#### i. Antimony

<b>Accuracy compared to Reference Material</b>														
Mean concentration should be within $\pm 15\%$ of the nominal value except at 3*LOD, where it should be within $\pm 20\%$														
Method name:		Urine Multi-Element ICP-DRC-MS												
Method #:		3018												
Matrix:		Urine												
Units:		$\mu\text{g/L}$												
Reference material:		SRM 2668 L1, SRM 2668 L2 10x, SRM 2668 L2 4x												
Analyte:		antimony												
Reference material	Replicate	Nominal value	Measured concentration								Mean	SD	CV (%)	Difference from nominal value (%)
			Day 1	Day 2	Day 3	Day 4	Day 5							
Level 1	1	0.242	0.27	0.22	0.23	0.24	0.22	0.24	0.02	7.99	-2.5			
	2		0.27	0.22	0.23	0.25	0.22							
Level 2	1	2.24	2.2	2.1	2.1	2.3	2.2	2.20	0.08	3.65	-2.0			
	2		2.2	2.1	2.1	2.3	2.2							
Level 3	1	5.6	5.4	5.3	5.4	5.6	5.8	5.48	0.16	2.97	-2.1			
	2		5.4	5.4	5.4	5.6	5.7							

#### ii. Barium

<b>Accuracy compared to Reference Material</b>											
Mean concentration should be within $\pm 15\%$ of the nominal value except at 3*LOD, where it should be within $\pm 20\%$											
Method name:		Urine Multi-Element ICP-DRC-MS									
Method #:		3018									
Matrix:		Urine									
Units:		$\mu\text{g/L}$									
Reference material:		SRM 2668 L1 2x, SRM 2668 L1, SRM 2668 L2 20x									
Analyte:		barium									
Reference material	Replicate	Nominal value	Measured concentration					Mean	SD	CV (%)	Difference from nominal value (%)
			Day 1	Day 2	Day 3	Day 4	Day 5				
Level 1	1	0.98	0.94	0.86	0.91	1.0	1.3	0.97	0.13	13.59	-0.6
	2		0.93	0.88	0.91	1.0	0.95				
Level 2	1	1.96	1.9	1.7	1.8	1.9	1.9	1.9	0.06	3.22	-5.5
	2		1.9	1.8	1.9	1.9	1.8				
Level 3	1	12.73	12	12	11	14	12	12.3	0.87	7.06	-3.0
	2		12	12	11	14	13				

### iii. Beryllium

#### Accuracy compared to Reference Material

Mean concentration should be within  $\pm 15\%$  of the nominal value except at 3\*LOD, where it should be within  $\pm 20\%$

Method name: Urine Multi-Element ICP-DRC-MS  
 Method #: 3018  
 Matrix: Urine  
 Units:  $\mu\text{g/L}$   
 Reference material: SRM 2668 L1 2x, SRM 2668 L1, SRM 2668 L2 10x  
 Analyte: beryllium

Reference material	Replicate	Nominal value	Measured concentration								Difference from nominal value (%)
			Day 1	Day 2	Day 3	Day 4	Day 5	Mean	SD	CV (%)	
Level 1	1	0.5365	0.57	0.53	0.55	0.58	0.57	0.56	0.03	4.80	3.6
	2		0.57	0.51	0.52	0.59	0.57				
Level 2	1	1.073	1.2	1.0	1.0	1.1	1.1	1.09	0.05	4.34	2.0
	2		1.2	1.0	1.1	1.1	1.1				
Level 3	1	5.45	5.4	5.3	5.2	5.8	5.5	5.47	0.24	4.45	0.4
	2		5.5	5.3	5.3	5.9	5.5				

### iv. Cadmium

#### Accuracy compared to Reference Material

Mean concentration should be within  $\pm 15\%$  of the nominal value except at 3\*LOD, where it should be within  $\pm 20\%$

Method name: Urine Multi-Element ICP-DRC-MS  
 Method #: 3018  
 Matrix: Urine  
 Units:  $\mu\text{g/L}$   
 Reference material: SRM 2668 L1 2x, SRM 2668 L1, SRM 2668 L2 4x  
 Analyte: cadmium

Reference material	Replicate	Nominal value	Measured concentration								Difference from nominal value (%)
			Day 1	Day 2	Day 3	Day 4	Day 5	Mean	SD	CV (%)	
Level 1	1	0.528	0.54	0.54	0.51	0.57	0.51	0.54	0.02	4.28	2.3
	2		0.55	0.52	0.53	0.55	0.58				
Level 2	1	1.056	1.1	1.1	1.1	1.1	1.0	1.07	0.02	1.91	1.2
	2		1.1	1.1	1.1	1.0	1.1				
Level 3	1	4.1	4.0	3.9	4.0	4.2	4.2	4.05	0.11	2.77	-1.2
	2		4.0	3.9	4.0	4.1	4.1				

v. Cesium

**Accuracy compared to Reference Material**

Mean concentration should be within ±15% of the nominal value except at 3\*LOD, where it should be within ± 20%

Method name: Urine Multi-Element ICP-DRC-MS  
 Method #: 3018  
 Matrix: Urine  
 Units: µg/L  
 Reference material: SRM 2668 L1 2x, SRM 2668 L1, SRM 2668 L2 20x  
 Analyte: cesium

Reference material	Replicate	Nominal value	Measured concentration								Difference from nominal value (%)
			Day 1	Day 2	Day 3	Day 4	Day 5	Mean	SD	CV (%)	
Level 1	1	2.45	2.4	2.4	2.4	2.6	2.5	2.46	0.07	2.97	0.6
	2		2.4	2.4	2.5	2.6	2.5				
Level 2	1	4.9	4.9	4.7	5.0	5.0	4.9	4.90	0.10	2.09	0.1
	2		4.9	4.7	5.1	4.9	5.0				
Level 3	1	11.05	11	11	10	12	11	11.01	0.62	5.60	-0.3
	2		11	11	10	12	11				

vi. Cobalt

**Accuracy compared to Reference Material**

Mean concentration should be within ±15% of the nominal value except at 3\*LOD, where it should be within ± 20%

Method name: Urine Multi-Element ICP-DRC-MS  
 Method #: 3018  
 Matrix: Urine  
 Units: µg/L  
 Reference material: SRM 2668 L1 2x, SRM 2668 L1, SRM 2668 L2 10x  
 Analyte: cobalt

Reference material	Replicate	Nominal value	Measured concentration								Difference from nominal value (%)
			Day 1	Day 2	Day 3	Day 4	Day 5	Mean	SD	CV (%)	
Level 1	1	0.408	0.45	0.43	0.40	0.43	0.45	0.43	0.02	5.29	6.1
	2		0.46	0.43	0.39	0.45	0.45				
Level 2	1	0.816	0.88	0.83	0.80	0.88	0.84	0.84	0.03	3.79	3.4
	2		0.89	0.85	0.80	0.84	0.84				
Level 3	1	5.18	5.2	5.2	5.1	5.7	5.1	5.24	0.23	4.40	1.2
	2		5.2	5.2	5.0	5.6	5.1				

**vii. Lead**

**Accuracy compared to Reference Material**

Mean concentration should be within ±15% of the nominal value except at 3\*LOD, where it should be within ± 20%

Method name: Urine Multi-Element ICP-DRC-MS  
 Method #: 3018  
 Matrix: Urine  
 Units: µg/L  
 Reference material: SRM 2668 L1 2x, SRM 2668 L1, SRM 2668 L2 20x  
 Analyte: lead

Reference material	Replicate	Nominal value	Measured concentration								Difference from nominal value (%)
			Day 1	Day 2	Day 3	Day 4	Day 5	Mean	SD	CV (%)	
Level 1	1	0.617	0.60	0.59	0.60	0.65	0.61	0.61	0.02	3.45	-0.9
	2		0.60	0.61	0.59	0.63	0.64				
Level 2	1	1.234	1.2	1.2	1.2	1.3	1.2	1.22	0.02	1.96	-1.0
	2		1.2	1.2	1.3	1.2	1.2				
Level 3	1	6.895	6.2	6.3	6.5	7.5	6.5	6.63	0.52	7.79	-3.8
	2		6.4	6.3	6.5	7.7	6.5				

**viii. Manganese**

**Accuracy compared to Reference Material**

Mean concentration should be within ±15% of the nominal value except at 3\*LOD, where it should be within ± 20%

Method name: Urine Multi-Element ICP-DRC-MS  
 Method #: 3018  
 Matrix: Urine  
 Units: µg/L  
 Reference material: SRM 2668 L1 2x, SRM 2668 L1, SRM 2668 L2 10x  
 Analyte: manganese

Reference material	Replicate	Nominal value	Measured concentration								Difference from nominal value (%)
			Day 1	Day 2	Day 3	Day 4	Day 5	Mean	SD	CV (%)	
Level 1	1	0.54	0.63	0.59	0.53	0.53	0.60	0.57	0.05	8.27	4.7
	2		0.55	0.61	0.54	0.48	0.60				
Level 2	1	1.08	1.3	1.1	1.2	1.2	1.1	1.23	0.16	13.27	14.1
	2		1.2	1.2	1.7	1.2	1.1				
Level 3	1	4.76	4.9	5.2	5.2	5.3	4.8	5.05	0.24	4.73	6.1
	2		4.8	5.1	5.1	5.4	4.7				

### ix. Molybdenum

#### Accuracy compared to Reference Material

Mean concentration should be within  $\pm 15\%$  of the nominal value except at 3\*LOD, where it should be within  $\pm 20\%$

Method name: Urine Multi-Element ICP-DRC-MS  
 Method #: 3018  
 Matrix: Urine  
 Units:  $\mu\text{g/L}$   
 Reference material: SRM 2668 L1 4x, SRM 2668 L1, SRM 2668 L2 10x  
 Analyte: molybdenum

Reference material	Replicate	Nominal value	Measured concentration								Difference from nominal value (%)
			Day 1	Day 2	Day 3	Day 4	Day 5	Mean	SD	CV (%)	
Level 1	1	12.9	13	13	13	14	13	13.03	0.56	4.34	1.0
	2		13	13	13	14	13				
Level 2	1	51.6	51	50	50	52	50	50.14	0.81	1.61	-2.8
	2		51	50	50	49	49				
Level 3	1	168.7	165	167	166	186	165	169.68	8.52	5.02	0.6
	2		165	167	166	186	165				

### x. Platinum

#### Accuracy compared to Reference Material

Mean concentration should be within  $\pm 15\%$  of the nominal value except at 3\*LOD, where it should be within  $\pm 20\%$

Method name: Urine Multi-Element ICP-DRC-MS  
 Method #: 3018  
 Matrix: Urine  
 Units:  $\mu\text{g/L}$   
 Reference material: SRM 2668 L1 4x, SRM 2668 L1 2x, SRM 2668 L1  
 Analyte: platinum

Reference material	Replicate	Nominal value	Measured concentration								Difference from nominal value (%)
			Day 1	Day 2	Day 3	Day 4	Day 5	Mean	SD	CV (%)	
Level 1	1	0.26	0.22	0.25	0.26	0.26	0.19	0.23	0.03	11.26	-10.2
	2		0.22	0.25	0.24	0.25	0.19				
Level 2	1	0.52	0.46	0.51	0.49	0.53	0.39	0.47	0.05	9.62	-8.7
	2		0.46	0.50	0.50	0.51	0.41				
Level 3	1	1.04	0.89	0.88	1.00	0.95	0.87	0.91	0.05	5.39	-12.7
	2		0.87	0.86	0.96	0.93	0.87				



### xi. Strontium

#### Accuracy compared to Reference Material

Mean concentration should be within  $\pm 15\%$  of the nominal value except at 3\*LOD, where it should be within  $\pm 20\%$

Method name: Urine Multi-Element ICP-DRC-MS  
 Method #: 3018  
 Matrix: Urine  
 Units:  $\mu\text{g/L}$   
 Reference material: Seronorm L-1 4x, Seronorm L-1 2x, Seronorm L-1  
 Analyte: strontium

Reference material	Replicate	Nominal value	Measured concentration								Difference from nominal value (%)
			Day 1	Day 2	Day 3	Day 4	Day 5	Mean	SD	CV (%)	
Level 1	1	29.75	30	21	29	35	29	28.78	4.50	15.63	-3.3
	2		29	21	29	35	29				
Level 2	1	59.5	58	43	55	64	58	55.57	7.22	13.00	-6.6
	2		58	43	56	64	57				
Level 3	1	119	116	115	113	113	109	112.81	2.88	2.56	-5.2
	2		115	116	108	114	110				

### xii. Tin

#### Accuracy compared to Reference Material

Mean concentration should be within  $\pm 15\%$  of the nominal value except at 3\*LOD, where it should be within  $\pm 20\%$

Method name: Urine Multi-Element ICP-DRC-MS  
 Method #: 3018  
 Matrix: Urine  
 Units:  $\mu\text{g/L}$   
 Reference material: SRM 2668 L1 2x, SRM 2668 L1, SRM 2668 L2 10x  
 Analyte: tin

Reference material	Replicate	Nominal value	Measured concentration								Difference from nominal value (%)
			Day 1	Day 2	Day 3	Day 4	Day 5	Mean	SD	CV (%)	
Level 1	1	0.845	0.71	0.84	0.77	0.76	0.64	0.75	0.07	8.87	-11.4
	2		0.75	0.82	0.78	0.77	0.65				
Level 2	1	1.69	1.5	1.8	1.5	1.6	1.3	1.53	0.16	10.50	-9.6
	2		1.5	1.7	1.5	1.6	1.3				
Level 3	1	17.1	15	16	16	19	14	16.11	1.64	10.21	-5.8
	2		15	16	16	19	14				

### xiii. Thallium

#### Accuracy compared to Reference Material

Mean concentration should be within  $\pm 15\%$  of the nominal value except at 3\*LOD, where it should be within  $\pm 20\%$

Method name: Urine Multi-Element ICP-DRC-MS  
 Method #: 3018  
 Matrix: Urine  
 Units:  $\mu\text{g/L}$   
 Reference material: SRM 2668 L1 2x, SRM 2668 L1, SRM 2668 L2 40x  
 Analyte: thallium

Reference material	Replicate	Nominal value	Measured concentration								Difference from nominal value (%)
			Day 1	Day 2	Day 3	Day 4	Day 5	Mean	SD	CV (%)	
Level 1	1	0.3595	0.36	0.40	0.34	0.38	0.36	0.36	0.02	4.82	0.9
	2		0.35	0.37	0.34	0.37	0.36				
Level 2	1	0.719	0.71	0.72	0.71	0.74	0.73	0.72	0.01	1.63	0.2
	2		0.72	0.73	0.71	0.70	0.72				
Level 3	1	2.88	2.6	2.9	2.9	3.0	2.7	2.83	0.16	5.50	-1.8
	2		2.7	2.9	2.9	3.1	2.7				

### xiv. Tungsten

#### Accuracy compared to Reference Material

Mean concentration should be within  $\pm 15\%$  of the nominal value except at 3\*LOD, where it should be within  $\pm 20\%$

Method name: Urine Multi-Element ICP-DRC-MS  
 Method #: 3018  
 Matrix: Urine  
 Units:  $\mu\text{g/L}$   
 Reference material: SRM 2668 L1 2x, SRM 2668 L1, SRM 2668 L2 20x  
 Analyte: tungsten

Reference material	Replicate	Nominal value	Measured concentration								Difference from nominal value (%)
			Day 1	Day 2	Day 3	Day 4	Day 5	Mean	SD	CV (%)	
Level 1	1	0.626	0.59	0.62	0.62	0.64	0.63	0.61	0.02	2.85	-2.2
	2		0.61	0.59	0.60	0.64	0.61				
Level 2	1	1.252	1.2	1.2	1.2	1.2	1.2	1.21	0.01	1.23	-3.1
	2		1.2	1.2	1.2	1.2	1.2				
Level 3	1	3.125	2.8	3.0	3.0	3.5	3.0	3.08	0.23	7.42	-1.4
	2		2.9	3.0	3.0	3.5	3.1				

**xv. Uranium**

**Accuracy compared to Reference Material**

Mean concentration should be within  $\pm 15\%$  of the nominal value except at 3\*LOD, where it should be within  $\pm 20\%$

Method name: Urine Multi-Element ICP-DRC-MS  
Method #: 3018  
Matrix: Urine  
Units:  $\mu\text{g/L}$   
Reference material: SRM 2668 L1 2x, SRM 2668 L1, SRM 2668 L2 40x  
Analyte: uranium

Reference material	Replicate	Nominal value	Measured concentration								Difference from nominal value (%)
			Day 1	Day 2	Day 3	Day 4	Day 5	Mean	SD	CV (%)	
Level 1	1	0.017	0.015	0.017	0.016	0.019	0.016	0.02	0.00	9.35	-4.2
	2		0.014	0.017	0.016	0.018	0.015				
Level 2	1	0.034	0.033	0.036	0.031	0.039	0.031	0.03	0.00	7.67	0.3
	2		0.035	0.036	0.033	0.036	0.032				
Level 3	1	0.33425	0.27	0.32	0.32	0.36	0.27	0.31	0.04	12.35	-7.6
	2		0.27	0.31	0.31	0.38	0.27				

## B. Precision

### i. Antimony

<b>Precision</b>						
Total relative standard deviation should be $\leq 15\%$ (CV $\leq 15\%$ )						
Method name:	Urine Multi-Element ICP-DRC-MS					
Method #:	3018					
Matrix:	Urine					
Units:	$\mu\text{g/L}$					
Analyte:	antimony					
<b>Quality material 1</b>						
Run	Result 1	Result 2	Mean	SS 1	SS 2	2*mean^2
1	0.15	0.15	0.15	1E-08	1E-08	0.04792608
2	0.16	0.15	0.16	4.9E-05	4.9E-05	0.04948658
3	0.18	0.17	0.17	3.08025E-05	3.08025E-05	0.058584645
4	0.16	0.18	0.17	3.90625E-05	3.90625E-05	0.057223445
5	0.16	0.16	0.16	3.4225E-06	3.4225E-06	0.051809805
6	0.16	0.16	0.16	4E-06	4E-06	0.05268258
7	0.15	0.14	0.15	1.156E-05	1.156E-05	0.04339458
8	0.16	0.17	0.16	9.9225E-06	9.9225E-06	0.053366445
9	0.16	0.15	0.16	1.764E-05	1.764E-05	0.049298
10	0.16	0.15	0.16	1.296E-05	1.296E-05	0.04848498
<b>Grand sum</b>	<b>3.198</b>	<b>Grand mean</b>	<b>0.1599</b>			
	<b>Sum squares</b>	<b>Mean Sq Error</b>	<b>Std Dev</b>	<b>Rel Std Dev (%)</b>		
<b>Within Run</b>	0.00035676	0.000035676	0.005972939	3.74		
<b>Between Run</b>	0.00089694	9.966E-05	0.005656147	3.54		
<b>Total</b>	0.0012537		0.008226056	<b>5.14</b>		
<b>Quality material 2</b>						
Run	Result 1	Result 2	Mean	SS 1	SS 2	2*mean^2
1	4.4	4.5	4.45	3.721E-05	3.721E-05	39.69226802
2	4.4	4.3	4.35	0.00225625	0.00225625	37.80499058
3	4.3	4.2	4.28	0.000897003	0.000897002	36.60684613
4	4.4	4.5	4.43	0.00073441	0.00073441	39.21791048
5	4.4	4.2	4.30	0.002899822	0.002899823	36.95506421
6	4.5	4.4	4.44	0.00027556	0.00027556	39.48760712
7	4.2	4.2	4.21	0.00132496	0.00132496	35.46335762
8	4.5	4.6	4.54	0.000189063	0.000189062	41.30950513
9	4.3	4.4	4.36	0.00062001	0.00062001	38.01222432
10	4.4	4.4	4.43	3.025E-05	3.025E-05	39.29588552
<b>Grand sum</b>	<b>87.5977</b>	<b>Grand mean</b>	<b>4.379885</b>			
	<b>Sum squares</b>	<b>Mean Sq Error</b>	<b>Std Dev</b>	<b>Rel Std Dev</b>		
<b>Within Run</b>	0.018529075	0.001852908	0.043045412	0.98		
<b>Between Run</b>	0.177806851	0.019756317	0.094613448	2.16		
<b>Total</b>	0.196335926		0.103945236	<b>2.37</b>		

ii. Barium

<b>Precision</b>						
Total relative standard deviation should be $\leq 15\%$ (CV $\leq 15\%$ )						
Method name:	Urine Multi-Element ICP-DRC-MS					
Method #:	3018					
Matrix:	Urine					
Units:	$\mu\text{g/L}$					
Analyte:	barium					
<b>Quality material 1</b>						
Run	Result 1	Result 2	Mean	SS 1	SS 2	2*mean <sup>2</sup>
1	1.5	1.5	1.54	1.521E-05	1.521E-05	4.76354978
2	1.5	1.5	1.47	7.14025E-05	7.14025E-05	4.315628205
3	1.5	1.4	1.45	9.12025E-05	9.12025E-05	4.231431405
4	1.5	1.5	1.49	0.000205923	0.000205923	4.458993845
5	1.5	1.5	1.49	1.521E-05	1.521E-05	4.422338
6	1.5	1.5	1.50	0.000287303	0.000287303	4.484114045
7	1.5	1.4	1.45	0.00412164	0.00412164	4.19224968
8	1.5	1.5	1.49	4.83025E-05	4.83025E-05	4.411935125
9	1.5	1.5	1.50	0.00010404	0.00010404	4.4910045
10	1.5	1.5	1.48	7.0225E-06	7.0225E-06	4.388203125
<b>Grand sum</b>	29.7142	<b>Grand mean</b>	1.48571			
	<b>Sum squares</b>	<b>Mean Sq Error</b>	<b>Std Dev</b>	<b>Rel Std Dev (%)</b>		
<b>Within Run</b>	0.00993451	0.000993451	0.031519058	2.12		
<b>Between Run</b>	0.012763628	0.001418181	0.014572747	0.98		
<b>Total</b>	0.022698138		0.034724861	<b>2.34</b>		
<b>Quality material 2</b>						
Run	Result 1	Result 2	Mean	SS 1	SS 2	2*mean <sup>2</sup>
1	14	14	13.72	0.003862623	0.003862623	376.6606704
2	13	13	13.04	0.000351563	0.000351562	339.8928426
3	13	13	13.26	0.00719104	0.00719104	351.5597345
4	13	14	13.51	0.00651249	0.00651249	365.0077767
5	13	13	13.17	0.00077284	0.00077284	346.6923784
6	13	13	13.36	0.00066564	0.00066564	357.086088
7	13	13	12.87	0.00219961	0.00219961	331.3664705
8	14	13	13.37	0.017516523	0.017516522	357.6822818
9	13	13	13.27	0.029635623	0.029635623	352.4061164
10	13	14	13.55	0.021830063	0.021830063	367.066803
<b>Grand sum</b>	266.2439	<b>Grand mean</b>	13.312195			
	<b>Sum squares</b>	<b>Mean Sq Error</b>	<b>Std Dev</b>	<b>Rel Std Dev</b>		
<b>Within Run</b>	0.181076025	0.018107603	0.134564492	1.01		
<b>Between Run</b>	1.130448124	0.125605347	0.23183803	1.74		
<b>Total</b>	1.311524149		0.268060581	<b>2.01</b>		

iii. Beryllium

<b>Precision</b>						
Total relative standard deviation should be $\leq 15\%$ (CV $\leq 15\%$ )						
Method name:	Urine Multi-Element ICP-DRC-MS					
Method #:	3018					
Matrix:	Urine					
Units:	$\mu\text{g/L}$					
Analyte:	beryllium					
<b>Quality material 1</b>						
Run	Result 1	Result 2	Mean	SS 1	SS 2	2*mean^2
1	0.43	0.42	0.43	3.025E-05	3.025E-05	0.36159008
2	0.43	0.45	0.44	8.649E-05	8.649E-05	0.38386322
3	0.42	0.41	0.42	1.98025E-05	1.98025E-05	0.350870645
4	0.42	0.43	0.43	7.5625E-06	7.5625E-06	0.365085125
5	0.42	0.42	0.42	6.25E-08	6.25E-08	0.346528125
6	0.43	0.44	0.43	1.33225E-05	1.33225E-05	0.376278125
7	0.43	0.44	0.44	1.26025E-05	1.26025E-05	0.379581845
8	0.41	0.41	0.41	7.5625E-06	7.5625E-06	0.336282005
9	0.43	0.45	0.44	0.00010816	0.00010816	0.37914632
10	0.42	0.43	0.43	1.12225E-05	1.12225E-05	0.363889805
<b>Grand sum</b>	8.5341	<b>Grand mean</b>	0.426705			
	<b>Sum squares</b>	<b>Mean Sq Error</b>	<b>Std Dev</b>	<b>Rel Std Dev (%)</b>		
<b>Within Run</b>	0.000594075	5.94075E-05	0.007707626	1.81		
<b>Between Run</b>	0.001572154	0.000174684	0.00759198	1.78		
<b>Total</b>	0.002166229		0.010818765	<b>2.54</b>		
<b>Quality material 2</b>						
Run	Result 1	Result 2	Mean	SS 1	SS 2	2*mean^2
1	7.2	7.4	7.34	0.009594203	0.009594203	107.67341
2	7.1	7.4	7.29	0.01901641	0.01901641	106.1482781
3	7.0	7.1	7.08	0.003950123	0.003950123	100.2230662
4	7.1	7.2	7.13	0.00502681	0.00502681	101.7822049
5	7.0	7.0	7.01	9.31225E-05	9.31225E-05	98.30964421
6	7.3	7.4	7.36	0.00154449	0.00154449	108.368642
7	7.1	7.4	7.25	0.017596023	0.017596023	105.2598932
8	7.0	7.0	7.01	0.000855563	0.000855563	98.36574061
9	7.2	7.5	7.33	0.029532423	0.029532423	107.4270162
10	7.1	7.4	7.27	0.02996361	0.02996361	105.702892
<b>Grand sum</b>	144.1478	<b>Grand mean</b>	7.20739			
	<b>Sum squares</b>	<b>Mean Sq Error</b>	<b>Std Dev</b>	<b>Rel Std Dev</b>		
<b>Within Run</b>	0.23434555	0.023434555	0.15308349	2.12		
<b>Between Run</b>	0.331375248	0.036819472	0.081807448	1.14		
<b>Total</b>	0.565720798		0.17357135	<b>2.41</b>		

iv. Cadmium

<b>Precision</b>						
Total relative standard deviation should be $\leq 15\%$ ( $CV \leq 15\%$ )						
Method name:	Urine Multi-Element ICP-DRC-MS					
Method #:	3018					
Matrix:	Urine					
Units:	$\mu\text{g/L}$					
Analyte:	cadmium					
<b>Quality material 1</b>						
Run	Result 1	Result 2	Mean	SS 1	SS 2	2*mean^2
1	0.22	0.23	0.229	0.00004	0.00004	0.10484
2	0.24	0.24	0.236	0.00000	0.00000	0.11182
3	0.20	0.21	0.206	0.00001	0.00001	0.08491
4	0.21	0.23	0.220	0.00007	0.00007	0.09706
5	0.22	0.21	0.213	0.00003	0.00003	0.09087
6	0.20	0.24	0.220	0.00038	0.00038	0.09702
7	0.22	0.19	0.207	0.00018	0.00018	0.08553
8	0.21	0.21	0.213	0.00000	0.00000	0.09048
9	0.21	0.21	0.206	0.00000	0.00000	0.08479
10	0.19	0.20	0.196	0.00008	0.00008	0.07687
<b>Grand sum</b>	4.2932	<b>Grand mean</b>	0.21466			
	<b>Sum squares</b>	<b>Mean Sq Error</b>	<b>Std Dev</b>	<b>Rel Std Dev (%)</b>		
<b>Within Run</b>	0.00158451	0.000158451	0.012587732	5.86		
<b>Between Run</b>	0.002614338	0.000290482	0.008124992	3.79		
<b>Total</b>	0.004198848		0.014982206	<b>6.98</b>		
<b>Quality material 2</b>						
Run	Result 1	Result 2	Mean	SS 1	SS 2	2*mean^2
1	6.6	6.7	6.65	0.005783602	0.005783603	88.37718301
2	6.1	6.3	6.20	0.004025903	0.004025902	76.94573405
3	6.0	6.1	6.03	0.00467856	0.00467856	72.64463648
4	6.1	6.3	6.18	0.013328703	0.013328703	76.26248501
5	5.7	5.7	5.73	5.476E-05	5.476E-05	65.60393058
6	6.0	6.2	6.14	0.00870489	0.00870489	75.374642
7	6.0	6.1	6.05	0.00509796	0.00509796	73.29698888
8	6.0	6.0	5.98	0.00077284	0.00077284	71.62129928
9	5.9	6.0	5.98	0.001402502	0.001402502	71.59856113
10	6.0	6.2	6.09	0.004935063	0.004935062	74.26270321
<b>Grand sum</b>	122.0661	<b>Grand mean</b>	6.103305			
	<b>Sum squares</b>	<b>Mean Sq Error</b>	<b>Std Dev</b>	<b>Rel Std Dev</b>		
<b>Within Run</b>	0.097569565	0.009756957	0.098777308	1.62		
<b>Between Run</b>	0.981525144	0.109058349	0.222824362	3.65		
<b>Total</b>	1.079094709		0.243736852	<b>3.99</b>		

v. Cesium

<b>Precision</b>						
Total relative standard deviation should be $\leq 15\%$ (CV $\leq 15\%$ )						
Method name:	Urine Multi-Element ICP-DRC-MS					
Method #:	3018					
Matrix:	Urine					
Units:	$\mu\text{g/L}$					
Analyte:	cesium					
<b>Quality material 1</b>						
Run	Result 1	Result 2	Mean	SS 1	SS 2	2*mean <sup>2</sup>
1	4.0	4.0	4.03	7.396E-05	7.396E-05	32.53663112
2	4.0	4.0	3.98	0.000217563	0.000217562	31.66090313
3	4.0	4.0	3.97	1.98025E-05	1.98025E-05	31.47655825
4	3.9	4.0	3.95	0.000133403	0.000133402	31.25953381
5	3.9	3.9	3.91	0.000223502	0.000223503	30.53789401
6	4.0	4.0	3.97	0.000260822	0.000260823	31.51624225
7	4.0	3.8	3.94	0.01038361	0.01038361	31.0393205
8	4.0	4.1	4.01	0.00290521	0.00290521	32.17303328
9	4.0	4.0	4.02	0.00012769	0.00012769	32.369058
10	4.0	4.0	4.03	4.84E-06	4.84E-06	32.52695168
<b>Grand sum</b>	79.6321	<b>Grand mean</b>	3.981605			
	<b>Sum squares</b>	<b>Mean Sq Error</b>	<b>Std Dev</b>	<b>Rel Std Dev (%)</b>		
<b>Within Run</b>	0.028700805	0.002870081	0.053573132	1.35		
<b>Between Run</b>	0.032558484	0.003617609	0.019332989	0.49		
<b>Total</b>	0.061259289		0.056954762	<b>1.43</b>		
<b>Quality material 2</b>						
Run	Result 1	Result 2	Mean	SS 1	SS 2	2*mean <sup>2</sup>
1	14	14	13.67	0.000867302	0.000867303	373.9100618
2	14	14	13.57	0.002814303	0.002814302	368.3467962
3	14	14	13.61	0.00683929	0.00683929	370.4750881
4	13	13	13.47	0.000545222	0.000545223	362.8360034
5	13	13	13.37	0.00084681	0.00084681	357.3961537
6	13	13	13.37	0.00015129	0.00015129	357.3640717
7	13	13	13.44	0.000484	0.000484	361.2403205
8	14	14	13.74	0.009830722	0.009830722	377.3306676
9	14	14	13.68	0.00164836	0.00164836	374.230082
10	14	14	13.86	0.010090203	0.010090203	383.9359051
<b>Grand sum</b>	271.5357	<b>Grand mean</b>	13.576785			
	<b>Sum squares</b>	<b>Mean Sq Error</b>	<b>Std Dev</b>	<b>Rel Std Dev</b>		
<b>Within Run</b>	0.068235005	0.0068235	0.082604482	0.61		
<b>Between Run</b>	0.483331441	0.053703493	0.153101262	1.13		
<b>Total</b>	0.551566446		0.173964068	<b>1.28</b>		



vi. Cobalt

<b>Precision</b>						
Total relative standard deviation should be ≤ 15% (CV ≤ 15%)						
Method name:	Urine Multi-Element ICP-DRC-MS					
Method #:	3018					
Matrix:	Urine					
Units:	µg/L					
Analyte:	cobalt					
<b>Quality material 1</b>						
Run	Result 1	Result 2	Mean	SS 1	SS 2	2*mean <sup>2</sup>
1	0.44	0.45	0.44	9.61E-06	9.61E-06	0.39587202
2	0.41	0.42	0.41	9.61E-06	9.61E-06	0.34262642
3	0.46	0.46	0.46	3.025E-07	3.025E-07	0.429757205
4	0.42	0.42	0.42	1.8225E-06	1.8225E-06	0.349698845
5	0.44	0.46	0.45	8.649E-05	8.649E-05	0.40392072
6	0.43	0.44	0.43	0.000016	0.000016	0.373248
7	0.44	0.42	0.43	0.000140423	0.000140422	0.369026405
8	0.41	0.39	0.40	5.11225E-05	5.11225E-05	0.321201125
9	0.56	0.57	0.56	3.364E-05	3.364E-05	0.63258752
10	0.48	0.48	0.48	1.849E-05	1.849E-05	0.46099202
<b>Grand sum</b>	8.9894	<b>Grand mean</b>	0.44947			
	<b>Sum squares</b>	<b>Mean Sq Error</b>	<b>Std Dev</b>	<b>Rel Std Dev (%)</b>		
<b>Within Run</b>	0.00073502	0.000073502	0.008573331	1.91		
<b>Between Run</b>	0.038464662	0.004273851	0.045827663	10.20		
<b>Total</b>	0.039199682		0.046622705	<b>10.37</b>		
<b>Quality material 2</b>						
Run	Result 1	Result 2	Mean	SS 1	SS 2	2*mean <sup>2</sup>
1	4.5	4.5	4.54	7.569E-05	7.569E-05	41.21048898
2	4.3	4.3	4.30	0.00033856	0.00033856	37.01268722
3	4.3	4.3	4.28	0.00030625	0.00030625	36.58032578
4	4.3	4.3	4.31	0.00137641	0.00137641	37.17633992
5	4.3	4.2	4.26	0.000455822	0.000455822	36.30116425
6	4.4	4.4	4.39	5.625E-07	5.625E-07	38.51172085
7	4.4	4.5	4.44	0.00267289	0.00267289	39.4183205
8	4.3	4.2	4.26	0.005409603	0.005409603	36.29605201
9	4.5	4.7	4.60	0.00527076	0.00527076	42.283208
10	4.3	4.4	4.35	0.001676903	0.001676902	37.81107761
<b>Grand sum</b>	87.4468	<b>Grand mean</b>	4.37234			
	<b>Sum squares</b>	<b>Mean Sq Error</b>	<b>Std Dev</b>	<b>Rel Std Dev (%)</b>		
<b>Within Run</b>	0.0351669	0.00351669	0.059301686	1.36		
<b>Between Run</b>	0.254243588	0.028249288	0.111203861	2.54		
<b>Total</b>	0.289410488		0.12602773	<b>2.88</b>		

vii. Lead

<b>Precision</b>						
Total relative standard deviation should be $\leq 15\%$ (CV $\leq 15\%$ )						
Method name:	Urine Multi-Element ICP-DRC-MS					
Method #:	3018					
Matrix:	Urine					
Units:	$\mu\text{g/L}$					
Analyte:	lead					
<b>Quality material 1</b>						
Run	Result 1	Result 2	Mean	SS 1	SS 2	2*mean <sup>2</sup>
1	0.48	0.48	0.48	5.625E-07	5.625E-07	0.457254845
2	0.42	0.42	0.42	5.5225E-06	5.5225E-06	0.353388245
3	0.43	0.44	0.43	1.3225E-06	1.3225E-06	0.377667405
4	0.44	0.44	0.44	7.84E-06	7.84E-06	0.39002112
5	0.44	0.44	0.44	0.000016	0.000016	0.38825672
6	0.44	0.43	0.43	2.89E-06	2.89E-06	0.3758445
7	0.45	0.42	0.44	0.000324	0.000324	0.38141378
8	0.43	0.42	0.43	1.80625E-05	1.80625E-05	0.364914245
9	0.44	0.46	0.45	0.00010201	0.00010201	0.39747528
10	0.43	0.42	0.43	1.296E-05	1.296E-05	0.36431648
<b>Grand sum</b>	8.7704	<b>Grand mean</b>	0.43852			
	<b>Sum squares</b>	<b>Mean Sq Error</b>	<b>Std Dev</b>	<b>Rel Std Dev (%)</b>		
<b>Within Run</b>	0.00098234	0.000098234	0.009911307	2.26		
<b>Between Run</b>	0.004556812	0.000506312	0.01428423	3.26		
<b>Total</b>	0.005539152		0.017386007	<b>3.96</b>		
<b>Quality material 2</b>						
Run	Result 1	Result 2	Mean	SS 1	SS 2	2*mean <sup>2</sup>
1	6.8	6.7	6.71	0.001958062	0.001958062	89.97977101
2	6.5	6.4	6.45	0.00045369	0.00045369	83.3211405
3	6.6	6.7	6.69	0.001853303	0.001853303	89.43595025
4	6.7	6.6	6.63	0.000753502	0.000753502	87.81702865
5	6.5	6.5	6.53	0.000128823	0.000128822	85.36932445
6	6.7	6.7	6.69	0.000625	0.000625	89.38914632
7	6.5	6.5	6.48	8.649E-05	8.649E-05	84.06895112
8	6.6	6.7	6.66	0.00093636	0.00093636	88.64993858
9	6.7	7.1	6.90	0.04485924	0.04485924	95.13997682
10	6.4	6.2	6.33	0.01336336	0.01336336	80.18084978
<b>Grand sum</b>	132.1282	<b>Grand mean</b>	6.60641			
	<b>Sum squares</b>	<b>Mean Sq Error</b>	<b>Std Dev</b>	<b>Rel Std Dev</b>		
<b>Within Run</b>	0.13003566	0.013003566	0.114033179	1.73		
<b>Between Run</b>	0.459015698	0.051001744	0.137837183	2.09		
<b>Total</b>	0.589051358		0.178892859	<b>2.71</b>		

### viii. Manganese

<b>Precision</b>						
Total relative standard deviation should be $\leq 15\%$ (CV $\leq 15\%$ )						
Method name:	Urine Multi-Element ICP-DRC-MS					
Method #:	3018					
Matrix:	Urine					
Units:	$\mu\text{g/L}$					
Analyte:	manganese					
<b>Quality material 1</b>						
Run	Result 1	Result 2	Mean	SS 1	SS 2	2*mean <sup>2</sup>
1	4.8	5.4	5.12	0.104555223	0.104555223	52.49538113
2	5.6	5.8	5.72	0.017969403	0.017969402	65.33502361
3	4.9	4.9	4.91	1.6E-07	1.6E-07	48.3046205
4	5.3	5.9	5.61	0.07750656	0.07750656	63.05195808
5	4.8	4.8	4.79	0.000280563	0.000280563	45.98117305
6	5.2	5.4	5.31	0.00579121	0.00579121	56.3603445
7	5.3	5.3	5.29	0.001306823	0.001306823	55.99888621
8	5.1	4.9	5.00	0.00915849	0.00915849	49.940018
9	4.9	5.2	5.05	0.02653641	0.02653641	51.04944968
10	4.8	5.2	4.98	0.030958403	0.030958403	49.52811865
<b>Grand sum</b>	103.5769	<b>Grand mean</b>	5.178845			
	<b>Sum squares</b>	<b>Mean Sq Error</b>	<b>Std Dev</b>	<b>Rel Std Dev (%)</b>		
<b>Within Run</b>	0.548126485	0.054812649	0.234121013	4.52		
<b>Between Run</b>	1.636262705	0.181806967	0.251986427	4.87		
<b>Total</b>	2.18438919		0.343961928	<b>6.64</b>		
<b>Quality material 2</b>						
Run	Result 1	Result 2	Mean	SS 1	SS 2	2*mean <sup>2</sup>
1	7.2	7.5	7.34	0.017689	0.017689	107.7600082
2	7.2	7.7	7.46	0.04875264	0.04875264	111.306184
3	6.7	6.9	6.80	0.00678976	0.00678976	92.59155362
4	7.5	8.1	7.82	0.09211225	0.09211225	122.2328666
5	6.6	6.6	6.56	1.6E-07	1.6E-07	86.1853205
6	7.5	7.8	7.64	0.01803649	0.01803649	116.769762
7	6.9	7.4	7.14	0.049217422	0.049217422	101.9206476
8	7.5	7.0	7.25	0.04268356	0.04268356	105.1511016
9	7.0	7.3	7.17	0.02387025	0.02387025	102.932552
10	6.9	7.3	7.07	0.043911203	0.043911203	100.0843668
<b>Grand sum</b>	144.5306	<b>Grand mean</b>	7.22653			
	<b>Sum squares</b>	<b>Mean Sq Error</b>	<b>Std Dev</b>	<b>Rel Std Dev (%)</b>		
<b>Within Run</b>	0.68612547	0.068612547	0.261939968	3.62		
<b>Between Run</b>	2.479646152	0.275516239	0.32163931	4.45		
<b>Total</b>	3.165771622		0.414806453	<b>5.74</b>		

## ix. Molybdenum

<b>Precision</b>						
Total relative standard deviation should be $\leq 15\%$ (CV $\leq 15\%$ )						
Method name:	Urine Multi-Element ICP-DRC-MS					
Method #:	3018					
Matrix:	Urine					
Units:	$\mu\text{g/L}$					
Analyte:	molybdenum					
<b>Quality material 1</b>						
Run	Result 1	Result 2	Mean	SS 1	SS 2	2*mean <sup>2</sup>
1	44	44	44.08	1E-08	1E-08	3885.757799
2	42	42	42.17	0.04959529	0.04959529	3556.567196
3	43	43	42.95	0.004096	0.004096	3689.164484
4	43	43	42.54	3.78225E-05	3.78225E-05	3619.941328
5	43	43	42.63	0.00015129	0.00015129	3635.367073
6	43	43	43.28	0.000402002	0.000402003	3745.806113
7	44	41	42.67	2.81266441	2.81266441	3642.055205
8	42	42	42.09	0.087645603	0.087645602	3542.454375
9	43	43	43.09	0.08048569	0.08048569	3712.824026
10	42	43	42.56	0.00344569	0.00344569	3622.332682
<b>Grand sum</b>	856.1097	<b>Grand mean</b>	42.805485			
	<b>Sum squares</b>	<b>Mean Sq Error</b>	<b>Std Dev</b>	<b>Rel Std Dev (%)</b>		
<b>Within Run</b>	6.077047615	0.607704761	0.779554207	1.82		
<b>Between Run</b>	6.079359531	0.675484392	0.184091867	0.43		
<b>Total</b>	12.15640715		0.800995991	<b>1.87</b>		
<b>Quality material 2</b>						
Run	Result 1	Result 2	Mean	SS 1	SS 2	2*mean <sup>2</sup>
1	118	118	117.78	0.020635322	0.020635323	27743.38523
2	113	111	112.33	0.97140736	0.97140736	25234.17069
3	115	115	114.72	0.000150063	0.000150062	26320.0949
4	114	114	114.46	9.90025E-05	9.90025E-05	26202.29766
5	114	113	113.43	0.05461569	0.05461569	25732.86592
6	117	116	116.33	0.046504922	0.046504923	27064.29084
7	113	114	113.96	0.22705225	0.22705225	25974.0367
8	114	112	113.33	1.046631303	1.046631303	25686.76582
9	114	118	115.93	2.948089	2.948089	26878.64874
10	114	114	113.60	0.00447561	0.00447561	25811.46498
<b>Grand sum</b>	2291.7205	<b>Grand mean</b>	114.586025			
	<b>Sum squares</b>	<b>Mean Sq Error</b>	<b>Std Dev</b>	<b>Rel Std Dev (%)</b>		
<b>Within Run</b>	10.63932105	1.063932105	1.031470845	0.90		
<b>Between Run</b>	48.87898037	5.430997819	1.477678198	1.29		
<b>Total</b>	59.51830142		1.802072407	<b>1.57</b>		

**x. Platinum**

<b>Precision</b>						
Total relative standard deviation should be ≤ 15% (CV ≤ 15%)						
Method name:	Urine Multi-Element ICP-DRC-MS					
Method #:	3018					
Matrix:	Urine					
Units:	µg/L					
Analyte:	platinum					
<b>Quality material 1</b>						
Run	Result 1	Result 2	Mean	SS 1	SS 2	2*mean <sup>2</sup>
1	0.16	0.16	0.16	5.625E-07	5.625E-07	0.050466645
2	0.16	0.15	0.16	1.64025E-05	1.64025E-05	0.050466645
3	0.17	0.16	0.16	1.8225E-06	1.8225E-06	0.054417005
4	0.16	0.16	0.16	5.625E-07	5.625E-07	0.049329405
5	0.16	0.16	0.16	8.1E-07	8.1E-07	0.05372642
6	0.15	0.16	0.16	1.96E-06	1.96E-06	0.04811202
7	0.16	0.14	0.15	0.000111303	0.000111303	0.047155205
8	0.16	0.17	0.16	3.66025E-05	3.66025E-05	0.053693645
9	0.16	0.17	0.17	1.12225E-05	1.12225E-05	0.054747405
10	0.15	0.16	0.16	1E-08	1E-08	0.04805
<b>Grand sum</b>	3.1931	<b>Grand mean</b>	0.159655			
	<b>Sum squares</b>	<b>Mean Sq Error</b>	<b>Std Dev</b>	<b>Rel Std Dev (%)</b>		
<b>Within Run</b>	0.000362515	3.62515E-05	0.006020922	3.77		
<b>Between Run</b>	0.000370014	4.11127E-05	0.001559042	0.98		
<b>Total</b>	0.000732529		0.006219494	<b>3.90</b>		
<b>Quality material 2</b>						
Run	Result 1	Result 2	Mean	SS 1	SS 2	2*mean <sup>2</sup>
1	1.3	1.3	1.34	1.521E-05	1.521E-05	3.596562
2	1.3	1.3	1.28	0.000522123	0.000522122	3.289356005
3	1.3	1.3	1.33	2.116E-05	2.116E-05	3.55697792
4	1.3	1.3	1.31	0.00012321	0.00012321	3.45161538
5	1.3	1.3	1.31	0.000122103	0.000122103	3.416759405
6	1.3	1.3	1.28	0.000748022	0.000748022	3.300651245
7	1.3	1.3	1.27	1.12225E-05	1.12225E-05	3.224530125
8	1.3	1.3	1.33	1.156E-05	1.156E-05	3.55057952
9	1.3	1.3	1.32	6.25E-06	6.25E-06	3.50489288
10	1.3	1.3	1.28	0.00032761	0.00032761	3.29679842
<b>Grand sum</b>	26.1446	<b>Grand mean</b>	1.30723			
	<b>Sum squares</b>	<b>Mean Sq Error</b>	<b>Std Dev</b>	<b>Rel Std Dev</b>		
<b>Within Run</b>	0.00381694	0.000381694	0.019536991	1.49		
<b>Between Run</b>	0.011717442	0.001301938	0.021450455	1.64		
<b>Total</b>	0.015534382		0.029014066	<b>2.22</b>		

**xi. Strontium**

<b>Precision</b>						
Total relative standard deviation should be $\leq 15\%$ (CV $\leq 15\%$ )						
Method name:	Urine Multi-Element ICP-DRC-MS					
Method #:	3018					
Matrix:	Urine					
Units:	$\mu\text{g/L}$					
Analyte:	strontium					
<b>Quality material 1</b>						
Run	Result 1	Result 2	Mean	SS 1	SS 2	2*mean <sup>2</sup>
1	86	86	86.39	0.005191203	0.005191203	14925.09927
2	83	84	83.21	0.13461561	0.13461561	13848.34075
3	84	84	84.33	0.011161923	0.011161922	14221.46185
4	80	82	81.33	0.770620622	0.770620623	13227.7227
5	85	84	84.25	0.145656722	0.145656723	14196.91696
6	85	85	84.79	4.42225E-05	4.42225E-05	14377.31463
7	73	68	70.32	7.79805625	7.79805625	9888.539081
8	81	78	79.42	2.62083721	2.62083721	12615.77171
9	84	87	85.32	1.93599396	1.93599396	14558.49288
10	83	85	83.94	0.313656002	0.313656003	14093.40853
<b>Grand sum</b>	1646.5752	<b>Grand mean</b>	82.32876			
	<b>Sum squares</b>	<b>Mean Sq Error</b>	<b>Std Dev</b>	<b>Rel Std Dev (%)</b>		
<b>Within Run</b>	27.47166745	2.747166745	1.657457917	2.01		
<b>Between Run</b>	392.5738902	43.61932114	4.520627965	5.49		
<b>Total</b>	420.0455577		4.814898124	<b>5.85</b>		
<b>Quality material 2</b>						
Run	Result 1	Result 2	Mean	SS 1	SS 2	2*mean <sup>2</sup>
1	482	486	483.75	4.338264123	4.338264123	468033.4463
2	464	466	464.92	0.490910422	0.490910422	432298.8882
3	443	438	440.49	6.698520423	6.698520422	388065.435
4	459	470	464.81	30.90414872	30.90414872	432094.3482
5	438	430	433.99	14.06475009	14.06475009	376690.4739
6	475	472	473.32	1.40541025	1.40541025	448062.1302
7	610	647	628.80	335.4923406	335.4923406	790768.442
8	443	428	435.28	59.53511281	59.53511281	378936.4862
9	458	490	474.26	253.8875758	253.8875758	449848.415
10	458	495	476.50	356.2259886	356.2259886	454096.5901
<b>Grand sum</b>	9552.2229	<b>Grand mean</b>	477.611145			
	<b>Sum squares</b>	<b>Mean Sq Error</b>	<b>Std Dev</b>	<b>Rel Std Dev (%)</b>		
<b>Within Run</b>	2126.086044	212.6086044	14.58110436	3.05		
<b>Between Run</b>	56646.53854	6294.059838	55.14277484	11.55		
<b>Total</b>	58772.62459		57.03800681	<b>11.94</b>		

xii. Tin

<b>Precision</b>						
Total relative standard deviation should be $\leq 15\%$ (CV $\leq 15\%$ )						
Method name:	Urine Multi-Element ICP-DRC-MS					
Method #:	3018					
Matrix:	Urine					
Units:	$\mu\text{g/L}$					
Analyte:	tin					
<b>Quality material 1</b>						
Run	Result 1	Result 2	Mean	SS 1	SS 2	2*mean <sup>2</sup>
1	1.6	1.7	1.65	0.000165123	0.000165123	5.474409605
2	1.5	1.5	1.49	5.29E-06	5.29E-06	4.46347442
3	1.7	1.6	1.66	0.00023104	0.00023104	5.53047282
4	1.7	1.7	1.72	1.936E-05	1.936E-05	5.94711072
5	1.6	1.6	1.60	0.000786803	0.000786802	5.089007045
6	1.5	1.6	1.55	0.00036481	0.00036481	4.81554578
7	1.6	1.4	1.49	0.003825423	0.003825423	4.436326845
8	1.9	1.8	1.82	0.000784	0.000784	6.6430125
9	1.5	1.5	1.54	8.1E-05	8.1E-05	4.730888
10	1.7	1.7	1.74	0.000160023	0.000160023	6.020797005
<b>Grand sum</b>	<b>32.5348</b>	<b>Grand mean</b>	<b>1.62674</b>			
	<b>Sum squares</b>	<b>Mean Sq Error</b>	<b>Std Dev</b>	<b>Rel Std Dev (%)</b>		
<b>Within Run</b>	0.01284574	0.001284574	0.035840954	2.20		
<b>Between Run</b>	0.225384188	0.025042688	0.108991086	6.70		
<b>Total</b>	0.238229928		0.114732867	<b>7.05</b>		
<b>Quality material 2</b>						
Run	Result 1	Result 2	Mean	SS 1	SS 2	2*mean <sup>2</sup>
1	21	21	21.02	0.00218089	0.00218089	883.8405592
2	19	18	18.56	0.019307103	0.019307103	689.2924592
3	19	19	19.19	0.02679769	0.02679769	736.4738205
4	19	20	19.71	0.058491422	0.058491422	776.8144696
5	19	19	19.02	0.013030222	0.013030223	723.3876661
6	21	20	20.49	0.028951022	0.028951022	839.5777531
7	19	19	18.87	1.521E-05	1.521E-05	712.1915405
8	19	21	19.93	1.020605063	1.020605063	794.6768844
9	19	19	18.67	0.01238769	0.01238769	697.4141434
10	22	22	21.68	0.00804609	0.00804609	940.2009025
<b>Grand sum</b>	<b>394.3009</b>	<b>Grand mean</b>	<b>19.715045</b>			
	<b>Sum squares</b>	<b>Mean Sq Error</b>	<b>Std Dev</b>	<b>Rel Std Dev (%)</b>		
<b>Within Run</b>	2.379624805	0.237962481	0.487813981	2.47		
<b>Between Run</b>	20.21021158	2.245579065	1.001902337	5.08		
<b>Total</b>	22.58983639		1.114347689	<b>5.65</b>		

xiii. Thallium

<b>Precision</b>						
Total relative standard deviation should be $\leq 15\%$ (CV $\leq 15\%$ )						
Method name:	Urine Multi-Element ICP-DRC-MS					
Method #:	3018					
Matrix:	Urine					
Units:	$\mu\text{g/L}$					
Analyte:	thallium					
<b>Quality material 1</b>						
Run	Result 1	Result 2	Mean	SS 1	SS 2	2*mean^2
1	0.18	0.18	0.18	2.56E-06	2.56E-06	0.06690482
2	0.16	0.17	0.17	2.401E-05	2.401E-05	0.05584482
3	0.17	0.17	0.17	5.625E-07	5.625E-07	0.057494405
4	0.16	0.17	0.17	2.55025E-05	2.55025E-05	0.056213045
5	0.17	0.17	0.17	9.025E-07	9.025E-07	0.055211645
6	0.17	0.18	0.17	7.5625E-06	7.5625E-06	0.060169805
7	0.18	0.16	0.17	4.29025E-05	4.29025E-05	0.057223445
8	0.17	0.17	0.17	6.25E-08	6.25E-08	0.056548845
9	0.18	0.19	0.18	1.56025E-05	1.56025E-05	0.065848205
10	0.16	0.16	0.16	2.89E-06	2.89E-06	0.05294258
<b>Grand sum</b>	3.4165	<b>Grand mean</b>	0.170825			
	<b>Sum squares</b>	<b>Mean Sq Error</b>	<b>Std Dev</b>	<b>Rel Std Dev (%)</b>		
<b>Within Run</b>	0.000245115	2.45115E-05	0.004950909	2.90		
<b>Between Run</b>	0.000778002	8.64447E-05	0.005564765	3.26		
<b>Total</b>	0.001023117		0.007448363	<b>4.36</b>		
<b>Quality material 2</b>						
Run	Result 1	Result 2	Mean	SS 1	SS 2	2*mean^2
1	2.5	2.5	2.52	1.849E-05	1.849E-05	12.72500352
2	2.4	2.4	2.39	0.000140422	0.000140423	11.39983501
3	2.4	2.4	2.40	0.000121	0.000121	11.54689568
4	2.4	2.4	2.43	7.83225E-05	7.83225E-05	11.83557205
5	2.4	2.4	2.40	8.1E-05	8.1E-05	11.56420232
6	2.5	2.5	2.46	3.136E-05	3.136E-05	12.06583688
7	2.5	2.5	2.54	6.76E-06	6.76E-06	12.88085768
8	2.4	2.5	2.45	0.000150062	0.000150062	12.04275965
9	2.5	2.5	2.50	0.000340403	0.000340403	12.51950761
10	2.4	2.4	2.40	0.001112223	0.001112223	11.47827785
<b>Grand sum</b>	48.9907	<b>Grand mean</b>	2.449535			
	<b>Sum squares</b>	<b>Mean Sq Error</b>	<b>Std Dev</b>	<b>Rel Std Dev</b>		
<b>Within Run</b>	0.004160085	0.000416009	0.020396286	0.83		
<b>Between Run</b>	0.0543139	0.006034878	0.0530041	2.16		
<b>Total</b>	0.058473985		0.056792985	<b>2.32</b>		



#### xiv. Tungsten

<b>Precision</b>						
Total relative standard deviation should be $\leq 15\%$ (CV $\leq 15\%$ )						
Method name:	Urine Multi-Element ICP-DRC-MS					
Method #:	3018					
Matrix:	Urine					
Units:	$\mu\text{g/L}$					
Analyte:	tungsten					
<b>Quality material 1</b>						
Run	Result 1	Result 2	Mean	SS 1	SS 2	2*mean <sup>2</sup>
1	0.24	0.24	0.24	4.41E-06	4.41E-06	0.11290752
2	0.22	0.22	0.22	0.000001	0.000001	0.094178
3	0.23	0.23	0.23	4.2025E-06	4.2025E-06	0.104653125
4	0.22	0.22	0.22	6.4E-07	6.4E-07	0.09513522
5	0.23	0.22	0.22	8.41E-06	8.41E-06	0.09927968
6	0.23	0.24	0.23	7.48225E-05	7.48225E-05	0.109278125
7	0.25	0.23	0.24	0.000119903	0.000119903	0.112101125
8	0.22	0.22	0.22	4E-06	4E-06	0.09618498
9	0.23	0.23	0.23	6.76E-06	6.76E-06	0.10598408
10	0.22	0.22	0.22	1.44E-06	1.44E-06	0.09618498
<b>Grand sum</b>	4.5271	<b>Grand mean</b>	0.226355			
	<b>Sum squares</b>	<b>Mean Sq Error</b>	<b>Std Dev</b>	<b>Rel Std Dev (%)</b>		
<b>Within Run</b>	0.000451175	4.51175E-05	0.006716956	2.97		
<b>Between Run</b>	0.001155115	0.000128346	0.006450913	2.85		
<b>Total</b>	0.00160629		0.00931299	<b>4.11</b>		
<b>Quality material 2</b>						
Run	Result 1	Result 2	Mean	SS 1	SS 2	2*mean <sup>2</sup>
1	4.1	4.1	4.05	2.5E-07	2.5E-07	32.85686048
2	3.9	3.9	3.89	6.32025E-05	6.32025E-05	30.30544805
3	3.9	3.9	3.87	3.249E-05	3.249E-05	29.9615405
4	4.0	4.0	4.01	0.000133403	0.000133402	32.23482925
5	3.9	3.9	3.89	0.00037636	0.00037636	30.22064768
6	3.9	3.9	3.93	0.000144	0.000144	30.8819405
7	4.0	3.9	3.96	0.00079524	0.00079524	31.4345205
8	3.9	4.0	3.94	0.00032041	0.00032041	30.99678848
9	4.0	4.0	4.00	0.002425562	0.002425562	31.99440025
10	4.0	4.0	3.97	0.000138063	0.000138062	31.54800745
<b>Grand sum</b>	79.0406	<b>Grand mean</b>	3.95203			
	<b>Sum squares</b>	<b>Mean Sq Error</b>	<b>Std Dev</b>	<b>Rel Std Dev</b>		
<b>Within Run</b>	0.00885796	0.000885796	0.029762325	0.75		
<b>Between Run</b>	0.064160702	0.007128967	0.05587115	1.41		
<b>Total</b>	0.073018662		0.063303882	<b>1.60</b>		

**xv. Uranium**

<b>Precision</b>						
Total relative standard deviation should be $\leq 15\%$ (CV $\leq 15\%$ )						
Method name:	Urine Multi-Element ICP-DRC-MS					
Method #:	3018					
Matrix:	Urine					
Units:	$\mu\text{g/L}$					
Analyte:	uranium					
<b>Quality material 1</b>						
Run	Result 1	Result 2	Mean	SS 1	SS 2	2*mean <sup>2</sup>
1	0.020	0.019	0.02	3.025E-07	3.025E-07	0.000756605
2	0.016	0.016	0.02	4E-08	4E-08	0.00049298
3	0.017	0.018	0.02	6.25E-08	6.25E-08	0.000595125
4	0.018	0.018	0.02	2.5E-09	2.5E-09	0.000623045
5	0.018	0.018	0.02	6.25E-08	6.25E-08	0.000630125
6	0.017	0.017	0.02	1.225E-07	1.225E-07	0.000574605
7	0.018	0.016	0.02	1.3225E-06	1.3225E-06	0.000561125
8	0.018	0.019	0.02	9E-08	9E-08	0.00066248
9	0.018	0.018	0.02	2.25E-08	2.25E-08	0.000623045
10	0.019	0.018	0.02	3.025E-07	3.025E-07	0.000688205
<b>Grand sum</b>	0.3518	<b>Grand mean</b>	0.01759			
	<b>Sum squares</b>	<b>Mean Sq Error</b>	<b>Std Dev</b>	<b>Rel Std Dev (%)</b>		
<b>Within Run</b>	0.00000466	0.000000466	0.000682642	3.88		
<b>Between Run</b>	1.9178E-05	2.13089E-06	0.000912384	5.19		
<b>Total</b>	2.3838E-05		0.001139493	<b>6.48</b>		
<b>Quality material 2</b>						
Run	Result 1	Result 2	Mean	SS 1	SS 2	2*mean <sup>2</sup>
1	0.44	0.43	0.44	1.3225E-06	1.3225E-06	0.379059245
2	0.38	0.38	0.38	4E-06	4E-06	0.2926125
3	0.39	0.41	0.40	8.464E-05	8.464E-05	0.31872128
4	0.41	0.41	0.41	2.25E-08	2.25E-08	0.330891125
5	0.41	0.42	0.41	2.916E-05	2.916E-05	0.33833538
6	0.42	0.42	0.42	1.225E-07	1.225E-07	0.356590125
7	0.38	0.37	0.38	1.33225E-05	1.33225E-05	0.284786045
8	0.43	0.44	0.43	3.30625E-05	3.30625E-05	0.374718245
9	0.40	0.46	0.43	0.000970323	0.000970323	0.371608205
10	0.41	0.41	0.41	9E-08	9E-08	0.34229538
<b>Grand sum</b>	8.2246	<b>Grand mean</b>	0.41123			
	<b>Sum squares</b>	<b>Mean Sq Error</b>	<b>Std Dev</b>	<b>Rel Std Dev (%)</b>		
<b>Within Run</b>	0.00227213	0.000227213	0.015073586	3.67		
<b>Between Run</b>	0.007415272	0.000823919	0.017272899	4.20		
<b>Total</b>	0.009687402		0.022925227	<b>5.57</b>		

## C. Stability

### i. Antimony

<b>Stability</b>									
<b>The initial measurement can be from the same day for all stability experiments.</b>									
<b>Freeze and thaw stability</b> = Assess for a minimum of 3 freeze-thaw cycles; conditions should mimic intended sample handling conditions Describe condition: Three times frozen at -70°C and then thawed (3 freeze-thaw cycles) to room temperature.									
<b>Bench-top stability</b> = Assess short-term stability for length of time needed to handle study samples (typically at room temperature) Describe condition: Original samples stored capped at room temperature for 1 day prior to preparation and analysis.									
<b>Processed sample stability</b> = Assess short-term stability of processed samples, including resident time in autosampler Describe condition: Processed samples (ready for instrument analysis) stored capped at room temperature for 1 day prior to analysis.									
<b>Long-term stability</b> = Assess long-term stability that equals or exceeds time between date of first sample collection and date of last sample analysis Describe condition: Samples stored at -70°C for 2 years.									
All stability sample results should be within ±15% of nominal concentration									
Method name: Urine Multi-Element ICP-DRC-MS									
Method #: 3018									
Matrix: Urine									
Units: µg/L									
Analyte: <b>antimony</b>									
<b>Quality material 1</b>									
	Initial measurement	Three freeze-thaw cycles	Initial measurement	Bench-top stability	Initial measurement	Processed sample stability	Initial measurement	Long-term stability	
Replicate 1	0.16	0.17	0.15	0.17	0.17	0.17	0.18	0.16	
Replicate 2	0.17	0.15	0.17	0.17	0.15	0.16	0.16	0.18	
Replicate 3	0.16	0.17	0.17	0.17	0.16	0.16	0.15	0.17	
Mean	0.1629	0.165733333	0.1627	0.2	0.160366667	0.1621	0.1643	0.2	
% difference from initial measurement	--	1.7	--	4.1	--	1.1	--	2.4	
<b>Quality material 2</b>									
	Initial measurement	Three freeze-thaw cycles	Initial measurement	Bench-top stability	Initial measurement	Processed sample stability	Initial measurement	Long-term stability	
Replicate 1	4.4	4.5	4.4	4.4	4.3	4.4	0.6	0.6	
Replicate 2	4.3	4.5	4.5	4.4	4.3	4.4	0.6	0.6	
Replicate 3	4.4	4.6	4.4	4.4	4.3	4.3	0.6	0.6	
Mean	4.348166667	4.534233333	4.4361	4.4	4.322066667	4.351366667	0.593316667	0.6	
% difference from initial measurement	--	4.3	--	-0.6	--	0.7	--	0.8	

## ii. Barium

### Stability

The initial measurement can be from the same day for all stability experiments.

**Freeze and thaw stability** = Assess for a minimum of 3 freeze-thaw cycles; conditions should mimic intended sample handling conditions

Describe condition: Three times frozen at -70°C and then thawed (3 freeze-thaw cycles) to room temperature.

**Bench-top stability** = Assess short-term stability for length of time needed to handle study samples (typically at room temperature)

Describe condition: Original samples stored capped at room temperature for 1 day prior to preparation and analysis.

**Processed sample stability** = Assess short-term stability of processed samples, including resident time in autosampler

Describe condition: Processed samples (ready for instrument analysis) stored capped at room temperature for 1 day prior to analysis.

**Long-term stability** = Assess long-term stability that equals or exceeds time between date of first sample collection and date of last sample analysis

Describe condition: Samples stored at -70°C for 2 years.

All stability sample results should be within  $\pm 15\%$  of nominal concentration

Method name: Urine Multi-Element ICP-DRC-MS

Method #: 3018

Matrix: Urine

Units:  $\mu\text{g/L}$

Analyte: barium

#### Quality material 1

	Initial measurement	Three freeze-thaw cycles	Initial measurement	Bench-top stability	Initial measurement	Processed sample	Initial measurement	Long-term
Replicate 1	1.5	1.6	1.5	1.5	1.5	1.6	1.3	1.2
Replicate 2	1.5	1.5	1.5	1.5	1.5	1.5	1.2	1.3
Replicate 3	1.6	1.5	1.5	1.5	1.5	1.5	1.2	1.3
Mean	1.544333333	1.544333333	1.519466667	1.5	1.519766667	1.5291	1.21	1.3
% difference from initial measurement	--	0.0	--	-2.7	--	0.6	--	3.6

#### Quality material 2

	Initial measurement	Three freeze-thaw cycles	Initial measurement	Bench-top stability	Initial measurement	Processed sample	Initial measurement	Long-term
Replicate 1	14	14	13	13	13	13	6.6	6.5
Replicate 2	14	14	13	13	14	13	6.4	6.5
Replicate 3	14	14	13	13	13	13	6.4	6.3
Mean	13.881033333	13.8	13.4244	13.2	13.3833	13.237533333	6.47	6.5
% difference from initial measurement	--	-0.2	--	-2.0	--	-1.1	--	-0.3

### iii. Beryllium

#### Stability

The initial measurement can be from the same day for all stability experiments.

**Freeze and thaw stability** = Assess for a minimum of 3 freeze-thaw cycles; conditions should mimic intended sample handling conditions

Describe condition: Three times frozen at -70°C and then thawed (3 freeze-thaw cycles) to room temperature.

**Bench-top stability** = Assess short-term stability for length of time needed to handle study samples (typically at room temperature)

Describe condition: Original samples stored capped at room temperature for 1 day prior to preparation and analysis.

**Processed sample stability** = Assess short-term stability of processed samples, including resident time in autosampler

Describe condition: Processed samples (ready for instrument analysis) stored capped at room temperature for 1 day prior to analysis.

**Long-term stability** = Assess long-term stability that equals or exceeds time between date of first sample collection and date of last sample analysis

Describe condition: Samples stored at -70°C for 2 years.

All stability sample results should be within ±15% of nominal concentration

Method name: Urine Multi-Element ICP-DRC-MS

Method #: 3018

Matrix: Urine

Units: µg/L

Analyte: beryllium

Quality material 1									
	Initial measurement	Three freeze-thaw cycles	Initial measurement	Bench-top stability	Initial measurement	Processed sample stability	Initial measurement	Long-term stability	
Replicate 1	0.41	0.42	0.43	0.42	0.45	0.44	0.36	0.38	
Replicate 2	0.42	0.41	0.43	0.41	0.42	0.43	0.39	0.40	
Replicate 3	0.42	0.43	0.43	0.40	0.43	0.43	0.37	0.40	
Mean	0.4159	0.417033333	0.426333333	0.4	0.431966667	0.431833333	0.372666667	0.4	
% difference from initial measurement	--	0.3	--	-3.4	--	0.0	--	6.0	
Quality material 2									
	Initial measurement	Three freeze-thaw cycles	Initial measurement	Bench-top stability	Initial measurement	Processed sample stability	Initial measurement	Long-term stability	
Replicate 1	7.2	7.4	7.2	7.1	7.3	7.3	5.2	5.1	
Replicate 2	7.4	7.3	7.4	7.1	7.4	7.2	5.0	5.1	
Replicate 3	7.2	7.4	7.3	7.0	7.3	7.2	5.0	5.1	
Mean	7.2679	7.367366667	7.282166667	7.1	7.347966667	7.223966667	5.066666667	5.1	
% difference from initial measurement	--	1.4	--	-2.7	--	-1.7	--	0.5	

#### iv. Cadmium

##### Stability

The initial measurement can be from the same day for all stability experiments.

**Freeze and thaw stability** = Assess for a minimum of 3 freeze-thaw cycles; conditions should mimic intended sample handling conditions

Describe condition: Three times frozen at -70°C and then thawed (3 freeze-thaw cycles) to room temperature.

**Bench-top stability** = Assess short-term stability for length of time needed to handle study samples (typically at room temperature)

Describe condition: Original samples stored capped at room temperature for 1 day prior to preparation and analysis.

**Processed sample stability** = Assess short-term stability of processed samples, including resident time in autosampler

Describe condition: Processed samples (ready for instrument analysis) stored capped at room temperature for 1 day prior to analysis.

**Long-term stability** = Assess long-term stability that equals or exceeds time between date of first sample collection and date of last sample analysis

Describe condition: Samples stored at -70°C for 2 years.

All stability sample results should be within ±15% of nominal concentration

Method name: Urine Multi-Element ICP-DRC-MS

Method #: 3018

Matrix: Urine

Units: µg/L

Analyte: cadmium

Quality material 1		Initial measurement	Three freeze-thaw cycles	Initial measurement	Bench-top stability	Initial measurement	Processed sample stability	Initial measurement	Long-term stability
Replicate 1		0.21	0.23	0.21	0.18	0.22	0.19	0.16	0.15
Replicate 2		0.23	0.21	0.22	0.19	0.20	0.20	0.17	0.16
Replicate 3		0.23	0.23	0.21	0.19	0.21	0.19	0.13	0.16
Mean		0.224766667	0.224033333	0.208866667	0.2	0.209233333	0.194033333	0.150783333	0.2
% difference from initial measurement		--	-0.3	--	-10.2	--	-7.3	--	3.7

Quality material 2		Initial measurement	Three freeze-thaw cycles	Initial measurement	Bench-top stability	Initial measurement	Processed sample stability	Initial measurement	Long-term stability
Replicate 1		6.0	6.1	6.1	5.9	6.2	5.8	1.4	1.5
Replicate 2		5.9	6.0	6.1	6.0	6.2	5.9	1.3	1.5
Replicate 3		6.0	6.2	6.0	6.0	6.1	5.8	1.6	1.6
Mean		5.9609	6.107466667	6.0673	6.0	6.17	5.8263	1.4479	1.6
% difference from initial measurement		--	2.5	--	-1.2	--	-5.6	--	7.3

## v. Cesium

### Stability

The initial measurement can be from the same day for all stability experiments.

**Freeze and thaw stability** = Assess for a minimum of 3 freeze-thaw cycles; conditions should mimic intended sample handling conditions

Describe condition: Three times frozen at -70°C and then thawed (3 freeze-thaw cycles) to room temperature.

**Bench-top stability** = Assess short-term stability for length of time needed to handle study samples (typically at room temperature)

Describe condition: Original samples stored capped at room temperature for 1 day prior to preparation and analysis.

**Processed sample stability** = Assess short-term stability of processed samples, including resident time in autosampler

Describe condition: Processed samples (ready for instrument analysis) stored capped at room temperature for 1 day prior to analysis.

**Long-term stability** = Assess long-term stability that equals or exceeds time between date of first sample collection and date of last sample analysis

Describe condition: Samples stored at -70°C for 2 years.

All stability sample results should be within ±15% of nominal concentration

Method name: Urine Multi-Element ICP-DRC-MS

Method #: 3018

Matrix: Urine

Units: µg/L

Analyte: cesium

#### Quality material 1

	Initial measurement	Three freeze-thaw cycles	Initial measurement	Bench-top stability	Initial measurement	Processed sample stability	Initial measurement	Long-term stability
Replicate 1	4.0	4.0	4.0	3.9	4.0	4.0	4.6	4.5
Replicate 2	4.0	4.0	4.0	3.9	3.9	3.9	4.5	4.5
Replicate 3	4.0	4.0	4.0	3.9	4.0	3.9	4.4	4.5
Mean	4.0291	3.997766667	3.995833333	3.9	3.9705	3.955933333	4.479483333	4.5
% difference from initial measurement	--	-0.8	--	-2.1	--	-0.4	--	-0.4

#### Quality material 2

	Initial measurement	Three freeze-thaw cycles	Initial measurement	Bench-top stability	Initial measurement	Processed sample stability	Initial measurement	Long-term stability
Replicate 1	14	14	14	13	13	14	13	12
Replicate 2	14	14	14	13	14	13	12	12
Replicate 3	14	14	14	13	13	13	12	12
Mean	13.74476667	13.63736667	13.5593	13.4	13.46753333	13.48116667	12.49213333	12.2
% difference from initial measurement	--	-0.8	--	-1.3	--	0.1	--	-2.2

## vi. Cobalt

### Stability

The initial measurement can be from the same day for all stability experiments.

**Freeze and thaw stability** = Assess for a minimum of 3 freeze-thaw cycles; conditions should mimic intended sample handling conditions

Describe condition: Three times frozen at -70°C and then thawed (3 freeze-thaw cycles) to room temperature.

**Bench-top stability** = Assess short-term stability for length of time needed to handle study samples (typically at room temperature)

Describe condition: Original samples stored capped at room temperature for 1 day prior to preparation and analysis.

**Processed sample stability** = Assess short-term stability of processed samples, including resident time in autosampler

Describe condition: Processed samples (ready for instrument analysis) stored capped at room temperature for 1 day prior to analysis.

**Long-term stability** = Assess long-term stability that equals or exceeds time between date of first sample collection and date of last sample analysis

Describe condition: Samples stored at -70°C for 2 years.

All stability sample results should be within ±15% of nominal concentration

Method name: Urine Multi-Element ICP-DRC-MS

Method #: 3018

Matrix: Urine

Units: µg/L

Analyte: cobalt

Quality material 1		Quality material 1		Quality material 1		Quality material 1		
	Initial measurement	Three freeze-thaw cycles	Initial measurement	Bench-top stability	Initial measurement	Processed sample stability	Initial measurement	Long-term stability
Replicate 1	0.43	0.44	0.43	0.44	0.46	0.44	0.55	0.49
Replicate 2	0.45	0.43	0.43	0.43	0.43	0.44	0.50	0.47
Replicate 3	0.43	0.44	0.44	0.45	0.43	0.45	0.47	0.48
Mean	0.436266667	0.434233333	0.433466667	0.4	0.443566667	0.442933333	0.507	0.5
% difference from initial measurement	--	-0.5	--	1.7	--	-0.1	--	-5.2
Quality material 2		Quality material 2		Quality material 2		Quality material 2		
	Initial measurement	Three freeze-thaw cycles	Initial measurement	Bench-top stability	Initial measurement	Processed sample stability	Initial measurement	Long-term stability
Replicate 1	4.2	4.2	4.2	4.4	4.3	4.3	2.4	2.5
Replicate 2	4.3	4.2	4.2	4.5	4.3	4.3	2.4	2.4
Replicate 3	4.2	4.3	4.3	4.4	4.3	4.3	2.4	2.5
Mean	4.253533333	4.247933333	4.244766667	4.4	4.288566667	4.336266667	2.428116667	2.5
% difference from initial measurement	--	-0.1	--	4.0	--	1.1	--	1.3



## vii. Lead

### Stability

The initial measurement can be from the same day for all stability experiments.

**Freeze and thaw stability** = Assess for a minimum of 3 freeze-thaw cycles; conditions should mimic intended sample handling conditions

Describe condition: Three times frozen at -70°C and then thawed (3 freeze-thaw cycles) to room temperature.

**Bench-top stability** = Assess short-term stability for length of time needed to handle study samples (typically at room temperature)

Describe condition: Original samples stored capped at room temperature for 1 day prior to preparation and analysis.

**Processed sample stability** = Assess short-term stability of processed samples, including resident time in autosampler

Describe condition: Processed samples (ready for instrument analysis) stored capped at room temperature for 1 day prior to analysis.

**Long-term stability** = Assess long-term stability that equals or exceeds time between date of first sample collection and date of last sample analysis

Describe condition: Samples stored at -70°C for 2 years.

All stability sample results should be within ±15% of nominal concentration

Method name: Urine Multi-Element ICP-DRC-MS

Method #: 3018

Matrix: Urine

Units: µg/L

Analyte: lead

Quality material 1		Quality material 1		Quality material 1		Quality material 1		
	Initial measurement	Three freeze-thaw cycles	Initial measurement	Bench-top stability	Initial measurement	Processed sample stability	Initial measurement	Long-term stability
Replicate 1	0.43	0.44	0.45	0.44	0.45	0.45	0.41	0.39
Replicate 2	0.44	0.43	0.43	0.43	0.44	0.45	0.37	0.40
Replicate 3	0.43	0.44	0.44	0.42	0.43	0.43	0.40	0.40
Mean	0.429566667	0.436633333	0.4409	0.4	0.442866667	0.443333333	0.394283333	0.4
% difference from initial measurement	--	1.6	--	0.5	--	0.1	--	0.1
Quality material 2		Quality material 2		Quality material 2		Quality material 2		
	Initial measurement	Three freeze-thaw cycles	Initial measurement	Bench-top stability	Initial measurement	Processed sample stability	Initial measurement	Long-term stability
Replicate 1	6.4	6.6	6.6	6.5	6.6	6.6	2.8	2.8
Replicate 2	6.4	6.6	6.7	6.6	6.7	6.7	2.9	2.8
Replicate 3	6.5	6.6	6.6	6.5	6.6	6.6	2.9	2.8
Mean	6.450066667	6.588733333	6.605466667	6.6	6.6233	6.670466667	2.850166667	2.8
% difference from initial measurement	--	2.1	--	-0.8	--	0.7	--	-1.4

### viii. Manganese

#### Stability

The initial measurement can be from the same day for all stability experiments.

**Freeze and thaw stability** = Assess for a minimum of 3 freeze-thaw cycles; conditions should mimic intended sample handling conditions

Describe condition: Three times frozen at -70°C and then thawed (3 freeze-thaw cycles) to room temperature.

**Bench-top stability** = Assess short-term stability for length of time needed to handle study samples (typically at room temperature)

Describe condition: Original samples stored capped at room temperature for 1 day prior to preparation and analysis.

**Processed sample stability** = Assess short-term stability of processed samples, including resident time in autosampler

Describe condition: Processed samples (ready for instrument analysis) stored capped at room temperature for 1 day prior to analysis.

**Long-term stability** = Assess long-term stability that equals or exceeds time between date of first sample collection and date of last sample analysis

Describe condition: Samples stored at -70°C for 2 years.

All stability sample results should be within ±15% of nominal concentration

Method name: Urine Multi-Element ICP-DRC-MS

Method #: 3018

Matrix: Urine

Units: µg/L

Analyte: manganese

Quality material 1		Initial measurement		Bench-top stability		Processed sample stability		Long-term stability	
	Initial measurement	Three freeze-thaw cycles	Initial measurement	Bench-top stability	Initial measurement	Processed sample stability	Initial measurement	Long-term stability	% difference from initial measurement
Replicate 1	5.9	6.1	5.0	4.5	5.4	6.3	0.67	0.77	
Replicate 2	5.3	5.3	4.9	4.7	5.4	4.8	0.78	0.83	
Replicate 3	5.0	5.2	5.1	4.9	5.0	4.8	0.72	0.74	
Mean	5.4087	5.5198	5.010266667	4.7	5.268366667	5.291233333	0.722716667	0.8	
% difference from initial measurement	--	2.1	--	-6.3	--	0.4	--	8.1	

Quality material 2		Initial measurement		Bench-top stability		Processed sample stability		Long-term stability	
	Initial measurement	Three freeze-thaw cycles	Initial measurement	Bench-top stability	Initial measurement	Processed sample stability	Initial measurement	Long-term stability	% difference from initial measurement
Replicate 1	7.2	7.2	7.1	6.6	7.2	6.9	2.8	3.0	
Replicate 2	7.1	7.0	7.1	6.6	7.1	6.9	2.5	3.1	
Replicate 3	7.3	7.1	7.1	6.6	7.2	6.8	3.5	2.8	
Mean	7.175233333	7.092233333	7.067633333	6.6	7.1585	6.84	2.92895	2.9	
% difference from initial measurement	--	-1.2	--	-6.4	--	-4.4	--	0.6	

## ix. Molybdenum

### Stability

The initial measurement can be from the same day for all stability experiments.

**Freeze and thaw stability** = Assess for a minimum of 3 freeze-thaw cycles; conditions should mimic intended sample handling conditions

Describe condition: Three times frozen at -70°C and then thawed (3 freeze-thaw cycles) to room temperature.

**Bench-top stability** = Assess short-term stability for length of time needed to handle study samples (typically at room temperature)

Describe condition: Original samples stored capped at room temperature for 1 day prior to preparation and analysis.

**Processed sample stability** = Assess short-term stability of processed samples, including resident time in autosampler

Describe condition: Processed samples (ready for instrument analysis) stored capped at room temperature for 1 day prior to analysis.

**Long-term stability** = Assess long-term stability that equals or exceeds time between date of first sample collection and date of last sample analysis

Describe condition: Samples stored at -70°C for 2 years.

All stability sample results should be within ±15% of nominal concentration

Method name: Urine Multi-Element ICP-DRC-MS

Method #: 3018

Matrix: Urine

Units: µg/L

Analyte: molybdenum

Quality material 1								
	Initial measurement	Three freeze-thaw cycles	Initial measurement	Bench-top stability	Initial measurement	Processed sample stability	Initial measurement	Long-term stability
Replicate 1	43	44	44	44	45	44	37	39
Replicate 2	44	45	44	44	44	44	38	39
Replicate 3	43	45	44	44	44	44	37	40
Mean	43.67926667	44.68183333	44.13593333	43.9	44.07906667	44.15946667	37.22275	39.4
% difference from initial measurement	--	2.3	--	-0.5	--	0.2	--	6.0

Quality material 2								
	Initial measurement	Three freeze-thaw cycles	Initial measurement	Bench-top stability	Initial measurement	Processed sample stability	Initial measurement	Long-term stability
Replicate 1	115	118	115	117	116	118	124	131
Replicate 2	115	118	117	118	118	119	129	132
Replicate 3	116	119	117	118	116	118	127	133
Mean	115.6496333	118.705	116.3135333	117.8	116.8394333	118.0265667	126.6666667	132.0
% difference from initial measurement	--	2.6	--	1.2	--	1.0	--	4.2

## x. Platinum

### Stability

The initial measurement can be from the same day for all stability experiments.

**Freeze and thaw stability** = Assess for a minimum of 3 freeze-thaw cycles; conditions should mimic intended sample handling conditions

Describe condition: Three times frozen at -70°C and then thawed (3 freeze-thaw cycles) to room temperature.

**Bench-top stability** = Assess short-term stability for length of time needed to handle study samples (typically at room temperature)

Describe condition: Original samples stored capped at room temperature for 1 day prior to preparation and analysis.

**Processed sample stability** = Assess short-term stability of processed samples, including resident time in autosampler

Describe condition: Processed samples (ready for instrument analysis) stored capped at room temperature for 1 day prior to analysis.

**Long-term stability** = Assess long-term stability that equals or exceeds time between date of first sample collection and date of last sample analysis

Describe condition: Samples stored at -70°C for 2 years.

All stability sample results should be within ±15% of nominal concentration

Method name: Urine Multi-Element ICP-DRC-MS

Method #: 3018

Matrix: Urine

Units: µg/L

Analyte: platinum

Quality material 1		Quality material 1		Quality material 1		Quality material 1		
	Initial measurement	Three freeze-thaw cycles	Initial measurement	Bench-top stability	Initial measurement	Processed sample stability	Initial measurement	Long-term stability
Replicate 1	0.16	0.16	0.17	0.17	0.17	0.18	0.086	0.10
Replicate 2	0.16	0.16	0.16	0.17	0.17	0.17	0.090	0.089
Replicate 3	0.16	0.16	0.16	0.16	0.17	0.18	0.12	0.091
Mean	0.1593	0.159233333	0.1644	0.2	0.1665	0.1758	0.098666667	0.1
% difference from initial measurement	--	0.0	--	2.7	--	5.6	--	-5.4
Quality material 2		Quality material 2		Quality material 2		Quality material 2		
	Initial measurement	Three freeze-thaw cycles	Initial measurement	Bench-top stability	Initial measurement	Processed sample stability	Initial measurement	Long-term stability
Replicate 1	1.3	1.3	1.3	1.4	1.4	1.4	0.84	0.77
Replicate 2	1.3	1.3	1.3	1.4	1.3	1.4	0.84	0.87
Replicate 3	1.3	1.4	1.4	1.4	1.3	1.4	0.96	1.10
Mean	1.322066667	1.345133333	1.344966667	1.4	1.3438	1.398866667	0.88	0.9
% difference from initial measurement	--	1.7	--	3.8	--	4.1	--	3.8

## xi. Strontium

### Stability

The initial measurement can be from the same day for all stability experiments.

**Freeze and thaw stability** = Assess for a minimum of 3 freeze-thaw cycles; conditions should mimic intended sample handling conditions

Describe condition: Three times frozen at -70°C and then thawed (3 freeze-thaw cycles) to room temperature.

**Bench-top stability** = Assess short-term stability for length of time needed to handle study samples (typically at room temperature)

Describe condition: Original samples stored capped at room temperature for 1 day prior to preparation and analysis.

**Processed sample stability** = Assess short-term stability of processed samples, including resident time in autosampler

Describe condition: Processed samples (ready for instrument analysis) stored capped at room temperature for 1 day prior to analysis.

**Long-term stability** = Assess long-term stability that equals or exceeds time between date of first sample collection and date of last sample analysis

Describe condition: Samples stored at -70°C for 2 years.

All stability sample results should be within ±15% of nominal concentration

Method name: Urine Multi-Element ICP-DRC-MS

Method #: 3018

Matrix: Urine

Units: µg/L

Analyte: strontium

Quality material 1									
	Initial measurement	Three freeze-thaw cycles	Initial measurement	Bench-top stability	Initial measurement	Processed sample stability	Initial measurement	Long-term stability	
Replicate 1	82	85	84	84	85	84	74	79	
Replicate 2	83	85	84	84	84	84	80	81	
Replicate 3	82	85	85	83	84	84	79	84	
Mean	82.46663333	84.88183333	84.31546667	83.6	84.36816667	84.00343333	77.72816667	81.3	
% difference from initial measurement	--	2.9	--	-0.8	--	-0.4	--	4.6	
Quality material 2									
	Initial measurement	Three freeze-thaw cycles	Initial measurement	Bench-top stability	Initial measurement	Processed sample stability	Initial measurement	Long-term stability	
Replicate 1	439	447	443	446	445	446	151	160	
Replicate 2	438	446	448	451	450	448	165	168	
Replicate 3	438	450	445	448	445	448	165	171	
Mean	438.09833333	447.39916667	445.56013333	448.3	446.59143333	447.2905	160.33333333	166.3	
% difference from initial measurement	--	2.1	--	0.6	--	0.2	--	3.7	

## xii. Tin

### Stability

The initial measurement can be from the same day for all stability experiments.

**Freeze and thaw stability** = Assess for a minimum of 3 freeze-thaw cycles; conditions should mimic intended sample handling conditions

Describe condition: Three times frozen at -70°C and then thawed (3 freeze-thaw cycles) to room temperature.

**Bench-top stability** = Assess short-term stability for length of time needed to handle study samples (typically at room temperature)

Describe condition: Original samples stored capped at room temperature for 1 day prior to preparation and analysis.

**Processed sample stability** = Assess short-term stability of processed samples, including resident time in autosampler

Describe condition: Processed samples (ready for instrument analysis) stored capped at room temperature for 1 day prior to analysis.

**Long-term stability** = Assess long-term stability that equals or exceeds time between date of first sample collection and date of last sample anal

Describe condition: Samples stored at -70°C for 2 years.

All stability sample results should be within ±15% of nominal concentration

Method name: Urine Multi-Element ICP-DRC-MS

Method #: 3018

Matrix: Urine

Units: µg/L

Analyte: tin

Quality material 1								
	Initial measurement	Three freeze-thaw cycles	Initial measurement	Bench-top stability	Initial measurement	Processed sample	Initial measurement	Long-term stability
Replicate 1	1.7	1.8	1.7	1.7	1.8	1.7	2.4	2.3
Replicate 2	1.7	1.8	1.6	1.6	1.7	1.6	2.6	2.6
Replicate 3	1.7	1.8	1.6	1.6	1.6	1.6	2.3	2.8
Mean	1.670833333	1.774	1.621366667	1.6	1.690733333	1.6527	2.44375	2.6
% difference from initial measurement	--	6.2	--	0.1	--	-2.2	--	4.6

Quality material 2								
	Initial measurement	Three freeze-thaw cycles	Initial measurement	Bench-top stability	Initial measurement	Processed sample	Initial measurement	Long-term stability
Replicate 1	21	22	20	20	20	20	15	15
Replicate 2	21	22	20	20	20	20	15	15
Replicate 3	21	22	20	20	20	20	15	15
Mean	20.6902	22.07023333	19.73306667	19.9	19.76133333	19.933333	15.01686667	15.0
% difference from initial measurement	--	6.7	--	1.1	--	0.9	--	-0.1

### xiii. Thallium

#### Stability

The initial measurement can be from the same day for all stability experiments.

**Freeze and thaw stability** = Assess for a minimum of 3 freeze-thaw cycles; conditions should mimic intended sample handling conditions

Describe condition: Three times frozen at -70°C and then thawed (3 freeze-thaw cycles) to room temperature.

**Bench-top stability** = Assess short-term stability for length of time needed to handle study samples (typically at room temperature)

Describe condition: Original samples stored capped at room temperature for 1 day prior to preparation and analysis.

**Processed sample stability** = Assess short-term stability of processed samples, including resident time in autosampler

Describe condition: Processed samples (ready for instrument analysis) stored capped at room temperature for 1 day prior to analysis.

**Long-term stability** = Assess long-term stability that equals or exceeds time between date of first sample collection and date of last sample analysis

Describe condition: Samples stored at -70°C for 2 years.

All stability sample results should be within ±15% of nominal concentration

Method name: Urine Multi-Element ICP-DRC-MS

Method #: 3018

Matrix: Urine

Units: µg/L

Analyte: thallium

Quality material 1		Initial measurement		Three freeze-thaw cycles		Bench-top stability		Processed sample stability		Initial measurement		Long-term stability	
Replicate 1		0.16	0.18	0.16	0.18	0.21	0.18	0.18	0.18	0.17	0.17	0.17	0.17
Replicate 2		0.17	0.18	0.16	0.18	0.16	0.18	0.18	0.18	0.17	0.17	0.17	0.17
Replicate 3		0.17	0.18	0.17	0.17	0.17	0.17	0.17	0.17	0.17	0.17	0.17	0.17
Mean		0.166733333	0.180733333	0.160733333	0.2	0.177666667	0.1742	0.1742	0.1742	0.172116667	0.1742	0.1742	0.2
% difference from initial measurement		--	8.4	--	9.1	--	-2.0	-2.0	-2.0	--	-1.5	-1.5	-1.5

Quality material 2		Initial measurement		Three freeze-thaw cycles		Bench-top stability		Processed sample stability		Initial measurement		Long-term stability	
Replicate 1		2.4	2.5	2.5	2.5	2.5	2.5	2.5	2.5	0.45	0.47	0.47	0.47
Replicate 2		2.4	2.5	2.6	2.6	2.5	2.5	2.5	2.5	0.46	0.46	0.46	0.46
Replicate 3		2.4	2.5	2.5	2.5	2.5	2.5	2.5	2.5	0.46	0.47	0.47	0.47
Mean		2.397	2.507466667	2.513033333	2.5	2.510966667	2.503066667	2.503066667	2.503066667	0.459566667	0.4666667	0.4666667	0.5
% difference from initial measurement		--	4.6	--	0.6	--	-0.3	-0.3	-0.3	--	0.9	0.9	0.9

### xiv. Tungsten

#### Stability

The initial measurement can be from the same day for all stability experiments.

**Freeze and thaw stability** = Assess for a minimum of 3 freeze-thaw cycles; conditions should mimic intended sample handling conditions

Describe condition: Three times frozen at -70°C and then thawed (3 freeze-thaw cycles) to room temperature.

**Bench-top stability** = Assess short-term stability for length of time needed to handle study samples (typically at room temperature)

Describe condition: Original samples stored capped at room temperature for 1 day prior to preparation and analysis.

**Processed sample stability** = Assess short-term stability of processed samples, including resident time in autosampler

Describe condition: Processed samples (ready for instrument analysis) stored capped at room temperature for 1 day prior to analysis.

**Long-term stability** = Assess long-term stability that equals or exceeds time between date of first sample collection and date of last sample analysis

Describe condition: Samples stored at -70°C for 2 years.

All stability sample results should be within ±15% of nominal concentration

Method name: Urine Multi-Element ICP-DRC-MS

Method #: 3018

Matrix: Urine

Units: µg/L

Analyte: tungsten

Quality material 1								
	Initial measurement	Three freeze-thaw cycles	Initial measurement	Bench-top stability	Initial measurement	Processed sample stability	Initial measurement	Long-term stability
Replicate 1	0.22	0.23	0.24	0.23	0.24	0.22	0.12	0.15
Replicate 2	0.21	0.23	0.21	0.22	0.23	0.24	0.14	0.16
Replicate 3	0.22	0.23	0.23	0.24	0.23	0.22	0.17	0.17
Mean	0.218133333	0.2314	0.226233333	0.2	0.231266667	0.227166667	0.14275	0.2
% difference from initial measurement	--	6.1	--	0.8	--	-1.8	--	9.6

Quality material 2								
	Initial measurement	Three freeze-thaw cycles	Initial measurement	Bench-top stability	Initial measurement	Processed sample stability	Initial measurement	Long-term stability
Replicate 1	3.9	4.1	4.0	3.9	3.9	4.0	0.87	0.92
Replicate 2	3.9	4.1	4.0	3.9	4.0	4.0	0.89	0.93
Replicate 3	3.9	4.0	3.9	3.9	3.9	4.0	0.91	0.95
Mean	3.909266667	4.058066667	3.9719	3.9	3.957833333	4.0424	0.892316667	0.9
% difference from initial measurement	--	3.8	--	-1.6	--	2.1	--	4.8



## xv. Uranium

### Stability

The initial measurement can be from the same day for all stability experiments.

**Freeze and thaw stability** = Assess for a minimum of 3 freeze-thaw cycles; conditions should mimic intended sample handling conditions

Describe condition: Three times frozen at -70°C and then thawed (3 freeze-thaw cycles) to room temperature.

**Bench-top stability** = Assess short-term stability for length of time needed to handle study samples (typically at room temperature)

Describe condition: Original samples stored capped at room temperature for 1 day prior to preparation and analysis.

**Processed sample stability** = Assess short-term stability of processed samples, including resident time in autosampler

Describe condition: Processed samples (ready for instrument analysis) stored capped at room temperature for 1 day prior to analysis.

**Long-term stability** = Assess long-term stability that equals or exceeds time between date of first sample collection and date of last sample analysis

Describe condition: Samples stored at -70°C for 2 years.

All stability sample results should be within ±15% of nominal concentration

Method name: Urine Multi-Element ICP-DRC-MS

Method #: 3018

Matrix: Urine

Units: µg/L

Analyte: uranium

Quality material 1								
	Initial measurement	Three freeze-thaw cycles	Initial measurement	Bench-top stability	Initial measurement	Processed sample stability	Initial measurement	Long-term stability
Replicate 1	0.017	0.018	0.016	0.016	0.019	0.017	0.017	0.019
Replicate 2	0.018	0.020	0.017	0.016	0.016	0.016	0.019	0.018
Replicate 3	0.017	0.020	0.016	0.016	0.017	0.018	0.018	0.019
Mean	0.017133333	0.019066667	0.0167	0.0	0.017066667	0.017033333	0.018166667	0.0
% difference from initial measurement	--	11.3	--	-3.8	--	-0.2	--	2.0

Quality material 2								
	Initial measurement	Three freeze-thaw cycles	Initial measurement	Bench-top stability	Initial measurement	Processed sample stability	Initial measurement	Long-term stability
Replicate 1	0.41	0.45	0.39	0.39	0.40	0.39	0.12	0.12
Replicate 2	0.41	0.45	0.40	0.39	0.39	0.38	0.12	0.12
Replicate 3	0.41	0.44	0.38	0.38	0.39	0.38	0.12	0.12
Mean	0.410033333	0.4464	0.389466667	0.4	0.392833333	0.3831	0.118233333	0.1
% difference from initial measurement	--	8.9	--	-0.2	--	-2.5	--	0.4

## D. Analytical Sensitivity and Specificity

### LOD, specificity and fit for intended use

Method name: Urine Multi-Element ICP-DRC-MS  
 Method #: 3018  
 Matrix: Urine  
 Units: µg/L

Analytes	Limit of Detection (LOD)	Interferences successfully checked in at least 50 human samples	Accuracy, precision, LOD, specificity and stability meet performance specifications for intended use
barium (UBA)	0.06	Yes	Yes
beryllium (UBE)	0.016	Yes	Yes
cadmium (UCD)	0.036	Yes	Yes
cobalt (UCO)	0.023	Yes	Yes
cesium (UCS)	0.086	Yes	Yes
manganese (UMN)	0.13	Yes	Yes
molybdenum (UMO)	0.80	Yes	Yes
lead (UPB)	0.03	Yes	Yes
platinum (UPT)	0.010	Yes	Yes
antimony (USB)	0.022	Yes	Yes
tin (USN)	0.09	Yes	Yes
strontium (USR)	2.34	Yes	Yes
thallium (UTL)	0.018	Yes	Yes
tungsten (UTU)	0.018	Yes	Yes
uranium (UUR)	0.0020	Yes	Yes

## 16. Appendix B. Ruggedness testing results

### A. Ruggedness parameter Test #1: RF Power.

- i. Test Details: Three different RF power settings were tested in separately prepared, consecutive runs on the instrument without turning off the plasma. At least 15 minutes stabilization time was allowed between each run after the RF power was changed. "Junk urine" samples (20) were analyzed between the beginning and ending QC of each run. All other method parameters were kept per method. Run order was as follows: run #1 (1450W), run #2 (1150W), run #3 (1600W).
- ii. Results: See Ruggedness Table 1, Ruggedness Table 2, Ruggedness Table 3, and Ruggedness Table 4.
- iii. Conclusion: Results are not compromised by changes in RF power within the range of 1150W to 1600W.

**Ruggedness Table 1. Parameter test 1 (Ba, Be, Cd, Co). Impact of RF power changes on observed results.**

ID	RF power tested*	Ba (µg/L)	Be (µg/L)	Cd (µg/L)	Co (µg/L)
LU-04310_UMP3_e	<i>characterized mean</i>	0.76	0.69	0.32	0.42
	<i>(±2SD range)</i>	(0.65 - 0.86)	(0.59 - 0.79)	(0.28 - 0.36)	(0.38 - 0.47)
	1150W	0.67	0.62	0.28	0.39
	1450W	0.70	0.66	0.31	0.32
	1600W	0.75	0.75	0.33	0.42
HU-04311_UMP3_e	<i>characterized mean</i>	5.01	5.28	1.62	1.88
	<i>(±2SD range)</i>	(4.54 - 5.24)	(4.48 - 6.07)	(1.47 - 1.78)	(1.66 - 2.09)
	1150W	4.93	5.75	1.6	1.96
	1450W	5.55	5.28	1.75	1.67
	1600W	4.85	5.82	1.58	1.90

\* Test performed 3/24/2010 by Denise Tevis using ELAN DRC-2N.

**Ruggedness Table 2. Parameter test 1 (Cs, Mo, Pb, Tl). Impact of RF power changes on observed results.**

ID	RF power tested*	Cs (µg/L)	Mo (µg/L)	Pb (µg/L)	Pt (µg/L)
LU-04310_UMP_e	<i>characterized mean (±2SD Range)</i>	2.38 (2.25 - 2.51)	19.3 (18.6 - 20.0)	0.42 (0.37 - 0.48)	0.10 (0.07 - 0.13)
	1150W	2.13 †	17.2 **	0.42	0.09
	1450W	2.32	18.9	0.41	0.09
	1600W	2.42	18.8	0.43	0.10
HU-04311_UMP_e	<i>characterized mean (±2SD Range)</i>	9.82 (9.03 - 10.6)	136 (131 - 142)	2.95 (2.82 - 3.08)	0.85 (0.71 - 1.00)
	1150W	9.62	136	3.03	0.93
	1450W	10.56	133	2.89	1.02
	1600W	9.55	135	3.05	1.08 #

\* Test performed 3/23/2011 by Denise Tevis using ELAN DRC-2N. † Low in one pool only and was within expected precision of method from default setting result. \*\* Low result in one pool only (3SD range = 18.2 – 20.4). # Result at 1600W within expected precision of result at default method setting.

**Ruggedness Table 3. Parameter test 1 (Sb, Tl, W, U). Impact of RF power changes on observed results.**

ID	RF power tested*	Sb (µg/L)	Tl (µg/L)	W (µg/L)	U (µg/L)
LU-04310_UMP_e	<i>characterized mean (±2SD Range)</i>	0.19 (0.17 - 0.21)	0.18 (0.17 - 0.19)	0.22 (0.19 - 0.24)	0.014 (0.011 - 0.016)
	1150W	0.21	0.16	0.19	0.017 <sup>α</sup>
	1450W	0.16	0.19	0.22	0.014
	1600W	0.20	0.18	0.22	0.017 <sup>α</sup>
HU-04311_UMP_e	<i>characterized mean (±2SD Range)</i>	0.66 (0.60 - 0.71)	0.58 (0.55 - 0.61)	0.94 (0.90 - 0.99)	0.128 (0.115 - 0.141)
	1150W	0.69	0.59	0.90	0.153 <sup>α</sup>
	1450W	0.61	0.57	0.93	0.126
	1600W	0.66	0.60	0.91	0.150 <sup>α</sup>

\* Test performed 3/23/2011 by Denise Tevis using ELAN DRC-2N. <sup>α</sup> Results within expected precision of the method of result at default setting (3 SD).

**Ruggedness Table 4. Parameter test 1 (Mn, Sn, and Sr). Impact of RF power changes on observed results.**

ID	RF power tested*	Mn (µg/L)	Sn (µg/L)	Sr (µg/L)
NYDOH UE09-05‡	<i>characterized mean (±2SD Range)</i>	1.37 (1.55 -1.19)	2.2 (2.0-2.8)	
	1150W	1.22	2.9	
	1450W	0.98	2.8	
	1600W	1.30	3.0	
NYDOH UE09-06‡	<i>characterized mean (±2SD Range)</i>	31.1 (26.3 -35.9)	61 (55.0 - 67.0)	
	1150W	29.4	67.4	
	1450W	23.8	68.5	
	1600W	30.0	66.7	
Seronorm Trace Elements Urine§	<i>characterized mean (±2SD Range)</i>	12.3 (10.9 - 13.7)	54.6 (51.9 - 57.3)	110 (104 -116)
	1150W	10.4	62.3	113
	1450W	8.46	62.3	111
	1600W	10.5	61.4	111

\* Tests performed 3/23/2011 by Denise Tevis using ELAN DRC-2N. §Purchased from Sero AS, Billingstad, Norway. ‡ Purchased from Wadsworth Center, New York State Department of Health

**B. Ruggedness parameter test #2: DRC mode cell gas flow rate.**

- i. Test details: Three different cell gas flow rates were tested in separately prepared, consecutive runs on the instrument without turning off the plasma. Samples were prepared with diluent containing the internal standards. At least 15 minutes stabilization time was allowed between each run after the cell gas flow rate was changed. “Junk urine” samples (20) were analyzed between the beginning and ending QC of each run.
1. Run #1: method default = 2.3 mL/min O<sub>2</sub>
  2. Run #2: decreased cell gas flow rate by 20% to 1.84 mL/min O<sub>2</sub>
  3. Run #3: increased cell gas flow rate by 20% to 2.76 mL/min O<sub>2</sub>
- ii. Results: See Ruggedness Table 5.
- iii. Conclusions: The accuracy and intra-measurement precision of Cd and Mn results are not affected by changes in the cell gas flow rate, within the tested range (1.84-2.76 mL/min).

**Ruggedness Table 5. Parameter test 2 (Cd, Mn, Ir). Impact of cell gas flow rate changes on observed results.**

ID	cell gas flow rate	Cd (µg/L)		Mn (µg/L)		Ir
LU10709	NexION mean (+/- 2SD)	0.150 (0.114 – 0.186) N=80	%RSD	0.79 (0.56 – 1.02) N=80	%RSD	%RSD
	1.84 mL/min O <sub>2</sub>	0.151	11.7	0.781	13.3	0.6
	2.3 mL/min O <sub>2</sub> (default)	0.146	4.3	0.731	0.3	0.9
	2.76 mL/min O <sub>2</sub>	0.129	14.6	0.673	4.1	0.6
HU10710	NexION mean (+/- 2SD)	1.52 (1.40 – 1.64) N=80	%RSD	2.95 (2.43 – 3.47) N=80	%RSD	%RSD
	1.84 mL/min O <sub>2</sub>	1.47	0.6	2.89	3.5	0.9
	1.3 mL/min O <sub>2</sub> (default)	1.51	2.5	2.76	4.0	0.7
	2.76 mL/min O <sub>2</sub>	1.39	5.7	2.48	5.5	1.0
SRM 2668 Level 2	NexION mean (+/- 2SD)	16.0 (14.6 – 17.5) N=48	%RSD	51.3 (44.0 – 58.7) N=48	%RSD	%RSD
	target (95% C.I.)	16.4 (15.3 – 17.5)	-	47.6 (44.2 – 51.0)	-	-
	1.84 mL/min O <sub>2</sub>	16.1	1.1	49.3	1.8	0.7
	2.3 mL/min O <sub>2</sub> (default)	16.2	0.7	48.5	1.3	0.4
	2.76 mL/min O <sub>2</sub>	14.7	1.4	43.3	1.4	1.1

\* Results collected 12/27/2012 on NexION C.

### C. Ruggedness parameter test #3: DRC mode RPq.

- i. Test details: Three RPq settings were tested for cadmium and manganese in separately prepared, consecutive runs on the instrument without turning off the plasma. Samples were prepared with diluent containing the internal standards. At least 15 minutes stabilization time was allowed between each run after DRC RPq was changed. “Junk urine” samples (20) were analyzed between the beginning and ending QC of each run.
1. Run #1: instrument default DRC RPq: 0.75
  2. Run #2: decreased; DRC RPq: 0.65
  3. Run #3: increased; DRC RPq: 0.85
  4. Run #4: increased; DRC RPq: 0.80
- ii. Results: See Ruggedness Table 6
- iii. Conclusions: Neither accuracy nor within-measurement precision of Cd and Mn results are compromised by changes in RPq within the range of 0.65 – 0.8. However, setting the RPq >0.8 causes problems in with-in measurement precision.

**Ruggedness Table 6. Parameter test 3 (Cd and Mn). Impact of DRC RPq changes on observed results.**

ID	DRC RPq	Cd (µg/L)		Mn (µg/L)		Ir
LU10709	NexION mean (+/- 2SD)	0.150 (0.114 – 0.186) N=80	%RSD	0.79 (0.56 – 1.02) N=80	%RSD	%RSD
	0.65	0.148	8.8	0.744	0.8	0.3
	0.75 (default)	0.155	10.8	0.745	5.5	0.4
	0.80	0.151	5.8	1.02	6.9	0.6
	0.85	0.137	3.8	0.813	27.4 *	0.7
HU10710	NexION mean (+/- 2SD)	1.52 (1.40 – 1.64) N=80	%RSD	2.95 (2.43 – 3.47) N=80	%RSD	%RSD
	0.65	1.51	3.8	2.83	1.6	0.3
	0.75 (default)	1.63	1.6	3.09	3.4	0.4
	0.80	1.50	3.6	3.03	2.1	0.6
	0.85	1.43	1.9	2.31	19.4 *	1.1
SRM 2668 Level 2	NexION mean (+/- 2SD)	16.0 (14.6 – 17.5) N=48	%RSD	51.3 (44.0 – 58.7) N=48	%RSD	%RSD
	target (95% C.I.)	16.4 (15.3 – 17.5)	-	47.6 (44.2 – 51.0)	-	-
	0.65	16.1	0.6	50.4	1.0	0.9
	0.75 (default)	15.9	0.7	50.0	1.6	0.7
	0.80	15.8	0.9	50.0	0.5	0.7
	0.85	15.4	2.0	46.1	0.6	0.8

\* Results collected 1/3/2013 on NexION D.



D. Ruggedness parameter test #4: stability of sample preparations.

i. Test details:

1. Three separate run sets (A, B, and C) were prepared at one sitting from the same starting materials. Set 'A' was analyzed immediately per the assumption of the method. Set's 'B' and 'C' were stored at ambient temperature for 24 and 48 hours, respectively before analysis. "Junk urine samples (20) were analyzed between the beginning and ending QC of each run, making each a normal length run. All other method parameters were kept per method.
2. On day two, a fresh run set ("D") was prepared and analyzed immediately for comparison to results from set "B" (Run 2 of the day. Results not shown).
3. On day three, another fresh run set ("E") was prepared and analyzed immediately for comparison to results from set "C" (Run 2 of the day. Results not shown).

i. Results: See Ruggedness Table 7, Ruggedness Table 8, and Ruggedness Table 9.

ii. Conclusions: Results from all times tested were within acceptable boundaries. Therefore, after preparation, capped working samples can be used up to 48 hours after preparation without compromising observed results.

Ruggedness Table 1. Parameter test 4 (Ba, Be, Cd, Co). Stability of sample preparations.

ID	time from preparation to analysis *	Ba (µg/L)	Be (µg/L)	Cd (µg/L)	Co (µg/L)
LU-04310_UMP3_e	characterized mean (±2SD range)	0.76 (0.65 - 0.86)	0.69 (0.59 - 0.79)	0.32 (0.28 - 0.36)	0.42 (0.38 - 0.47)
	freshly prepared	0.70	0.66	0.32	0.32
	24 hours	0.70	0.69	0.31	0.42
	48 hours	0.86	0.67	0.31	0.43
HU-04311_UMP3_e	characterized mean (±2SD range)	5.01 (4.54 - 5.24)	5.28 (4.48 - 6.07)	1.62 (1.47 - 1.78)	1.88 (1.66 - 2.09)
	freshly prepared	5.55	3.48	1.75	1.67
	24 hours	4.66	5.76	1.57	1.94
	48 hours	5.12	5.49	1.67	1.84

\* Test begun 3/23/11 by Denise Tevis using ELAN DRC-2N.

**Ruggedness Table 2. Parameter test 4 (Cs, Mo, Pb, Pt, Sb, Tl, W, U). Stability of sample preparations.**

ID	time from preparation to analysis	Cs (µg/L)	Mo (µg/L)	Pb (µg/L)	Pt (µg/L)
LU-04310_UMP_e	<i>characterized mean</i>	2.38	19.3	0.42	0.10
	<i>(±2SD range)</i>	(2.25 - 2.51)	(18.6 - 20.0)	(0.37 - 0.48)	(0.07 - 0.13)
	freshly prepared	2.32	18.9	0.41	0.09
	24 hours	2.35	19.2	0.44	0.11
	48 hours	2.30	19.0	0.46	0.10
HU-04311_UMP_e	<i>characterized mean</i>	9.82	136	2.95	0.85
	<i>(±2SD range)</i>	(9.03 - 10.6)	(131 - 142)	(2.82 - 3.08)	(0.71 - 1.00)
	freshly prepared	10.6	133	2.89	1.02
	24 hours	9.36	134	3.08	1.03
	48 hours	10.0	132	3.04	1.12 *
ID	time from preparation to analysis	Sb (µg/L)	Tl (µg/L)	W (µg/L)	U (µg/L)
LU-04310_UMP_e	<i>characterized mean</i>	0.19	0.18	0.22	0.014
	<i>(±2SD range)</i>	(0.17 - 0.21)	(0.17 - 0.19)	(0.19 - 0.24)	(0.011 - 0.016)
	freshly prepared	0.16	0.19	0.22	0.014
	24 hours	0.19	0.18	0.21	0.013
	48 hours	0.19	0.19	0.22	0.014
HU-04311_UMP_e	<i>characterized mean</i>	0.61	0.58	0.94	0.128
	<i>(±2SD range)</i>	(0.60 - 0.71)	(0.55 - 0.61)	(0.90 - 0.99)	(0.115 - 0.141)
	freshly prepared	0.61	0.57	0.93	0.126
	24 hours	0.65	0.60	0.90	0.128
	48 hours	0.69	0.59	0.90	0.126

\* Test begun 3/23/11 by Denise Tevis using ELAN DRC-2N. \*\* Results within expected precision of the method of result at fresh preparation.

**Ruggedness Table 9. Parameter test 4 (Mn, Sn, Sr). Stability of sample preparations.**

Parameter test 4 results (Table 3 of 3). All concentrations in µg/L.				
ID	time from preparation to analysis*	Mn (µg/L)	Sn (µg/L)	Sr (µg/L)
NYDOH UE09-05‡	<i>characterized mean</i> <i>(±2SD range)</i>	1.37 (1.55 -1.19)	2.2 (2.0-2.8)	
	freshly prepared	0.98	2.8	
	24 hours	1.26	2.6	
	48 hours	1.47	2.6	
NYDOH UE09-06‡	<i>characterized mean</i> <i>(±2SD range)</i>	31.1 (26.3 -35.9)	61 (55.0 - 67.0)	
	freshly prepared	23.8	68.5	
	24 hours	30.6	62.8	
	48 hours	31.9	61.4	
Seronom Trace Elements Urine§	<i>characterized mean</i> <i>(±2SD range)</i>	12.3 (10.9 - 13.7)	54.6 (51.9 - 57.3)	110 (104 -116)
	freshly prepared	8.47	62.3	111
	24 hours	10.9	57.5 **	112
	48 hours	10.4	58.3 **	114

\* Test begun 3/23/11 by Denise Tevis using ELAN DRC-2N. §Purchased from Sero AS, Billingstad, Norway.

‡ Purchased from Wadsworth Center, New York State Department of Health. \*\* Results within expected precision of the method of result at default setting

### E. Ruggedness parameter test #5: impact of extra dilutions.

**i. Test details:** Spiked urine was prepared for analysis 5 times at extra dilution levels.

Thallium was re-tested Nov-Dec 2015 using 5 different urine samples. Lead and cadmium were re-evaluated using 2014-2016 and 2017-2019 proficiency testing program results, respectively, across various samples (N=7-64 at the different dilutions).

**ii. Results:** See Ruggedness Table 10.

**iii. Conclusions:** Do not use greater than a 2x extra dilution for Sr, a 4x extra dilution for Cd, or Pb, a 5x extra dilution for Tl, or a 10x extra dilution for Mo or Pt. All others (Ba, Be, Co, Cs, Mn, Sb, Sn, U, and W) can be analyzed at up to a 20x extra dilution without significant effect ( $> \pm 10\%$  error) to the observed concentration.

**Ruggedness Table 10. Parameter test 5. Impact of extra dilutions.**

	<b>Ba</b>	<b>Be</b>	<b>Cd</b>	<b>Co</b>	<b>Cs</b>	<b>Mn</b>
initial conc (µg/L)	297	98	various	149	252	96
	observed concentrations normalized to 'no extra dilution' result					
no extra dilution	1.00 ± 0.02	1.00 ± 0.04	1.00	1.00 ± 0.02	1.00 ± 0.02	1.00 ± 0.03
2x dilution	1.00 ± 0.00	1.00 ± 0.01	1.06 ± 0.07	0.99 ± 0.01	0.99 ± 0.01	1.02 ± 0.01
4x dilution			1.09 ± 0.07			
5x dilution	0.99 ± 0.00	1.01 ± 0.01		0.99 ± 0.00	0.98 ± 0.00	1.03 ± 0.01
10x dilution	0.98 ± 0.00	1.00 ± 0.01	1.09 ± 0.05	0.98 ± 0.00	0.97 ± 0.00	1.03 ± 0.00
20x dilution	0.97 ± 0.00	1.01 ± 0.00	1.11 ± 0.08	0.98 ± 0.00	0.97 ± 0.00	1.05 ± 0.00

	<b>Mo</b>	<b>Pb</b>	<b>Pt</b>	<b>Sb</b>	<b>Sn</b>	<b>Sr</b>
initial conc (µg/L)	1885	various**	137	98	325	1816
	observed concentrations normalized to 'no extra dilution' result					
no extra dilution	1.00 ± 0.02	1.00	1.00 ± 0.01	1.00 ± 0.02	1.00 ± 0.05	1.00 ± 0.03
2x dilution	0.97 ± 0.00	1.03	1.00 ± 0.01	1.00 ± 0.00	1.00 ± 0.02	0.92 ± 0.01
4x dilution		1.05				
5x dilution	0.94 ± 0.00	1.09	0.99 ± 0.00	1.01 ± 0.00	1.00 ± 0.01	0.90 ± 0.00
10x dilution	0.92 ± 0.00	1.08	0.94 ± 0.00	1.00 ± 0.00	0.97 ± 0.01	0.88 ± 0.00
20x dilution	0.90 ± 0.00	1.09	0.89 ± 0.00	0.97 ± 0.00	0.95 ± 0.00	0.87 ± 0.00

	<b>Tl*</b>	<b>U</b>	<b>W</b>
initial conc (µg/L)	25-115	43	99
	observed concentrations normalized to 'no extra dilution' result		
no extra dilution	1.00	1.00 ± 0.06	1.00 ± 0.01
2x dilution	1.01 ± 0.01	1.00 ± 0.03	0.98 ± 0.01
5x dilution	1.04 ± 0.01	1.01 ± 0.01	0.96 ± 0.00
10x dilution	1.07 ± 0.01	1.01 ± 0.01	0.96 ± 0.00
20x dilution	1.09 ± 0.05	1.02 ± 0.00	0.95 ± 0.00

## 17. Appendix C

**Table 1. Instrument and method parameters.**

<b>Instrument:</b> PerkinElmer ELAN DRC II ICP-MS or NexION 300D	
<b>Autosampler:</b> ESI SC4 autosampler with FAST sample introduction system	
<b>Optimization (conditions) window parameters</b>	
RF power	1450W (ELAN), 1600W (NexION)
plasma gas flow (Ar)	15 L/min
auxiliary gas flow (Ar)	1.2 L/min
nebulizer gas flow (Ar)	~0.90 – 1.0 L/min (optimized as needed for sensitivity)
ion lens voltage(s)	AutoLens (optimized as needed for sensitivity)
AFV, QRO, CRO, CPV, discriminator threshold	Optimized per instrument by service engineer, or advanced user.
Parameters of x-y alignment, nebulizer gas flow, AutoLens voltages, mass calibration, dual detector calibration, and detector voltages are optimized regularly. Optimization file name = default.dac.	
<b>Configurations window parameters</b>	
cell gas changes pause times	Pressurize delay (From Standard to DRC) = 30 Exhaust delay (From DRC to Standard mode) = 30 Flow delay (gas changes while in DRC mode) = 30 Channel delay (channel change in DRC mode) = 30
<b>File names &amp; directories</b>	
Method file names	CDC_DLS3018_15 element_urbk.mth CDC_DLS3018_15 element_aqblk.mth
Dataset	Create a new dataset subfolder each day. Name as “2011-0718” for all work done on July 18, 2011
Sample file	Create for each day’s work
Report file name	See Figure 7 in Appendix C. <i>For sample results printouts</i> cdc_quant comprehensive.rop  <i>For calibration curve information</i> CDC_Quant Comprehensive (calib curve info).rop
Tuning	Default.tun
Optimization	Default.dac
Calibration	N/A
Polyatomic	elan.ply (ELAN), polyatomic.ply (NexION)
Report options template (transferring results to the database)	CDC_Database Output.rop <i>Report Format Options: select only “Use Separator”</i> <i>File Write Option: Append</i> <i>Report File name: include date, instrument, and group being analyzed in file name (i.e. 2012-0311b_NexION-A_HM-0364.txt)</i>
<b>Method parameters</b>	
<b>Method parameters: timing page (see Figure 2 in Appendix C)</b>	
sweeps/reading	40
readings/replicate	1
replicates	3

enable QC checking	On (Figures 2a and 2g)
isotopes monitored and internal standard associations (exact mass)	<p><u>Group 1 (use <math>^{103}\text{Rh}</math> as an internal standard)</u>  <math>^9\text{Be}</math> (9.0122), <math>^{59}\text{Co}</math> (58.9332), <math>^{88}\text{Sr}</math> (87.9056), <math>^{98}\text{Mo}</math> (97.9055), <math>^{103}\text{Rh}</math> (102.905), <math>^{118}\text{Sn}</math> (117.902), <math>^{121}\text{Sb}</math> (120.904), <math>^{133}\text{Cs}</math> (132.905), <math>^{138}\text{Ba}</math> (137.905)</p> <p><u>Group 2 (use <math>^{193}\text{Ir}</math> as an internal standard)</u>  <math>^{184}\text{W}</math> (183.951), <math>^{193}\text{Ir}</math> (192.963), <math>^{195}\text{Pt}</math> (194.965), <math>^{205}\text{Tl}</math> (204.975), <math>^{208}\text{Pb}</math> (207.977), <math>^{238}\text{U}</math> (238.05)</p> <p><u>Group 3 NexION only: (use <math>^{193}\text{Ir}</math> as an internal standard)</u>  <math>^{193}\text{Ir}</math> (192.963), <math>^{114}\text{Cd}</math> (113.904), <math>^{55}\text{Mn}</math> (54.9381),</p> <p>3018 can be performed analyzing only a subset of analytes and the appropriate internal standards.</p>
dwel times	<p><b>30 ms</b> for <math>^{59}\text{Co}</math>, <math>^{88}\text{Sr}</math>, <math>^{98}\text{Mo}</math>, <math>^{118}\text{Sn}</math>, <math>^{103}\text{Rh}</math> (vented mode), <math>^{121}\text{Sb}</math>, <math>^{133}\text{Cs}</math>, <math>^{138}\text{Ba}</math>, <math>^{184}\text{W}</math>, <math>^{193}\text{Ir}</math> (vented mode), <math>^{205}\text{Tl}</math>, and <math>^{208}\text{Pb}</math></p> <p><b>100 ms</b> for <math>^9\text{Be}</math>, <math>^{55}\text{Mn}</math>, <math>^{103}\text{Rh}</math> (DRC mode), <math>^{114}\text{Cd}</math>, <math>^{193}\text{Ir}</math> (DRC mode), <math>^{195}\text{Pt}</math>, and <math>^{238}\text{U}</math></p>
scan mode	Peak Hopping for all isotopes (1 MCA channel)
DRC channel A gas flow rate	Not used.
DRC channel B gas flow rate	Oxygen (5-7 psig delivery pressure) Typically 2.3 (1.8 – 2.8) mL/min * * (optimized instrument, and periodically verified)
RPa	0 for all isotopes
RPq	<p><i>Standard Mode</i>: 0.25 for all standard mode isotopes  <i>DRC Mode (Cd and Mn group)</i>: default 0.75 (0.65 – 0.80) *  for <math>^{114}\text{Cd}</math> (113.904) and <math>^{55}\text{Mn}</math> (54.9381), and <math>^{103}\text{Rh}</math> (102.905) or <math>^{193}\text{Ir}</math> (192.963) in DRC mode. Use the same RPQ for each.  (* Optimize per instrument, and periodically verified)</p>
<b>Method parameters: processing page (see Figure 3 in Appendix C)</b>	
detector mode	Dual
process spectral peak	N/A
Autolens	On
isotope ratio mode	Off
enable short settling time	Off
blank subtraction	After internal standard
measurement units	cps
process signal profile	N/A
<b>Method parameters: equations page (see Figures 2c and 3c in the Appendix)</b>	
equations	<p>On <math>^{208}\text{Pb}</math>, use "+ Pb 206 + Pb 207"</p> <p>On <math>^{238}\text{U}</math>, use "+ U 235"</p> <p>On <math>^{114}\text{Cd}</math>, use "- 0.027250 * Sn 118"</p>
<b>Method parameters: calibration page (see Figures 2d and 3d in the Appendix)</b>	
calibration type	external standard

curve type	weighted linear
sample units	"µg/L" or "ppb"
calibration standard concentrations (µg/L)	Be: 0.1, 0.3, 1, 3, 10 Co: 0.075, 0.225, 0.75, 2.25, 7.5 Sr: 6, 18, 60, 180, 600 Mo: 3, 9, 30, 90, 300 Sn: 0.3, 0.9, 3, 9, 30 Sb: 0.08, 0.24, 0.8, 2.4, 8 Cs: 0.2, 0.6, 2, 6, 20 Ba: 0.2, 0.6, 2, 6, 20 W: 0.06, 0.18, 0.6, 1.8, 6 Pt: 0.025, 0.075, 0.25, 0.75, 2.5 Tl: 0.04, 0.12, 0.4, 1.2, 4 Pb: 0.1, 0.3, 1, 3, 10 U: 0.005, 0.015, 0.05, 0.15, 0.5 Cd: 0.08, 0.24, 0.8, 2.4, 8 Mn: 0.1, 0.3, 1, 3, 10
<b>Method parameters: sampling page (see Figures 2e and 3e in the Appendix)</b>	
"peristaltic pump under computer control"	On
autosampler tray port sampling device	<b><i>If using ESI autosampler</i></b> Autosampler Type: AS-93plus Tray Name: esi.try Sampling Device: None  <b><i>If using other autosampler</i></b> Refer to autosampler user guide.
sample flush	<b><i>FAST defaults for all 15 elements</i></b> 10s at 6 rpm (standard ICP-MS peristaltic pump) 10s at 3 rpm (ESI DXi peristaltic pump)  <b><i>FAST defaults for a 3 element subset</i></b> 3.5s at 6 rpm (standard ICP-MS peristaltic pump) 3.5s at 3 rpm (ESI DXi peristaltic pump)  <b><i>FAST defaults for a single element subset</i></b> 2s at 6 rpm (standard ICP-MS peristaltic pump) 2s at 3 rpm (ESI DXi peristaltic pump)  Can be optimized as needed to adequately fill the FAST loop. As a matter of lab practice, set this time to equal the loop fill time in the ESI FAST program. As long as the combined time of sample flush + read delay is equal to the time required for signal to reach stability, analytical measurement will be good.

<p>read delay</p>	<p><b>Default for all 15 elements or down to a 3 element subset</b>  30s at 6 rpm (standard ICP-MS peristaltic pump)  30s at 3 rpm (ESI DXi peristaltic pump)</p> <p><b>Default for a single element subset</b>  20s at 6 rpm (standard ICP-MS peristaltic pump)  20s at 3rpm (ESI DXi peristaltic pump)</p> <p>Can be optimized as needed to reach signal stability before beginning analysis. As a matter of lab practice, set this time equal to the total time required for the signal to reach stability minus the loop fill time. As long as the combined time of sample flush + read delay is equal to the time required for signal to reach stability, analytical measurement will be good.</p>																																																
<p>wash</p>	<p><b>Default for all 15 elements or down to a 3 element subset</b>  50s at 6 rpm (standard ICP-MS peristaltic pump)  50s at 3 rpm (ESI DXi peristaltic pump)</p> <p><b>Default for a single element subset</b>  20s at 6 rpm (standard ICP-MS peristaltic pump)  20s at 3rpm (ESI DXi peristaltic pump)</p> <p>Can be optimized to allow for changes in FAST loop rinsing (must be greater than total time of steps in FAST program after the initial “on rinse” command).</p>																																																
<p>extended wash (via ICP-MS software QC checking)</p>	<p>For sample concentrations greater than these, setup the ICP-MS software’s ‘QC checking’ feature to “Wash for X and continue.” See Figure 8 in Appendix C.</p> <table border="1" data-bbox="691 1213 1385 1797"> <thead> <tr> <th>Analyte</th> <th>Conc.</th> <th>Extended Rinse Time</th> </tr> </thead> <tbody> <tr><td>Be</td><td>300 µg/L</td><td>200s</td></tr> <tr><td>Co</td><td>225 µg/L</td><td>200s</td></tr> <tr><td>Sr</td><td>18000 µg/L</td><td>200s</td></tr> <tr><td>Mo</td><td>900 µg/L</td><td>200s</td></tr> <tr><td>Sn</td><td>90 µg/L</td><td>200s</td></tr> <tr><td>Sb</td><td>24 µg/L</td><td>200s</td></tr> <tr><td>Cs</td><td>60 µg/L</td><td>200s</td></tr> <tr><td>Ba</td><td>600 µg/L</td><td>200s</td></tr> <tr><td>W</td><td>18 µg/L</td><td>200s</td></tr> <tr><td>Pt</td><td>75 µg/L</td><td>200s</td></tr> <tr><td>Tl</td><td>12 µg/L</td><td>200s</td></tr> <tr><td>Pb</td><td>300 µg/L</td><td>200s</td></tr> <tr><td>U</td><td>1.5 µg/L</td><td>200s</td></tr> <tr><td>Cd</td><td>24 µg/L</td><td>200s</td></tr> <tr><td>Mn</td><td>30 µg/L</td><td>200s</td></tr> </tbody> </table>	Analyte	Conc.	Extended Rinse Time	Be	300 µg/L	200s	Co	225 µg/L	200s	Sr	18000 µg/L	200s	Mo	900 µg/L	200s	Sn	90 µg/L	200s	Sb	24 µg/L	200s	Cs	60 µg/L	200s	Ba	600 µg/L	200s	W	18 µg/L	200s	Pt	75 µg/L	200s	Tl	12 µg/L	200s	Pb	300 µg/L	200s	U	1.5 µg/L	200s	Cd	24 µg/L	200s	Mn	30 µg/L	200s
Analyte	Conc.	Extended Rinse Time																																															
Be	300 µg/L	200s																																															
Co	225 µg/L	200s																																															
Sr	18000 µg/L	200s																																															
Mo	900 µg/L	200s																																															
Sn	90 µg/L	200s																																															
Sb	24 µg/L	200s																																															
Cs	60 µg/L	200s																																															
Ba	600 µg/L	200s																																															
W	18 µg/L	200s																																															
Pt	75 µg/L	200s																																															
Tl	12 µg/L	200s																																															
Pb	300 µg/L	200s																																															
U	1.5 µg/L	200s																																															
Cd	24 µg/L	200s																																															
Mn	30 µg/L	200s																																															



autosampler locations of blanks and standards	<p><i>For calibration curve (points to urine blank)</i>  CDC_DLS3018_15 element 5 calib_urblk.mth  By default (but can be customized), urine blank, 102; calibration stds, 103-107.</p> <p><i>For QC &amp; patient sample (points to aqueous blank)</i>  CDC_DLS3018_15 element 5 calib_aqblk.mth  By default (but can be customized), aqueous blank, 149.</p>
<b>FAST parameters: See Figures 4a through 4h in Appendix C for details</b>	
configuration file	default.sc (saved at C:\Program Files\ESI\ESI-SC\ OR at C:\Users\Public\ESI\ESI SC)
FAST programs	cdc_dls3018_15element_loop3.0ml_scfast_QCen.txt  others will be needed for different loop sizes and subsets of elements
<b><i>Potential emergency response modifications:</i></b>	
<u>cadmium:</u>	Analyze cadmium in standard mode with rhodium as the internal standard. Set dwell time to 50ms, DRC gas flow to 0, and RPq to 0.25.
<u>Non-FAST sample introduction system:</u>	<p>If the FAST sample introduction system is not available on any instruments, the method can still be implemented, but these changes will need to be made in the ICP-MS software (and ESI software if present).</p> <ul style="list-style-type: none"> <li>• <u>Sample flush:</u> Default is ~90s at 10 rpm. Set so that solution reaches nebulizer.</li> <li>• <u>Read delay:</u> Default is 20s at 10rpm. Set for best reproducibility of replicate measured intensities.</li> <li>• <u>Wash:</u> Default is 120s at 24rpm. Set to prevent significant carry-over from one sample to the next.</li> <li>• If using ESI autosampler without FAST, disable FAST in the ESI software before running analysis.</li> </ul>

**Table 2. Suggested maximum analyte concentrations for base urine.**

Analyte	Concentration ( $\mu\text{g/L}$ )
Be	0.5
Co	0.25
Mo	30
Sb	0.2
Cs	3
Ba	2
W	0.2
Pt	0.25
Tl	0.2
Pb	0.75
U	0.03
Cd	0.25
Mn	0.1
Sr	80
Sn	3

**Table 3. Multi-element stock standard concentrations.**

analyte	stock calibration standard conc. (mg/L)	stock calibration standard conc. (mg/L)
	High Purity Standards Item # SM-2107-037 Solution A  (5% HNO <sub>3</sub> )	High Purity Standards Item # SM-2107-037 Solution B  (5% HNO <sub>3</sub> , 1% HF, 0.5% HCl)
Be	200	
Co	150	
Mo		6000
Sb		160
Cs	400	
Ba	400	
W		120
Pt		50
Tl	80	
Pb	200	
U	10	
Cd	160	
Sr	12,000	
Sn		600
Mn	200	

**Table 4. Preparation of multi-element intermediate stock calibration standard.**

volume of flask (mL)	100
volume of spike of stock standard A (mL)*	5
volume of spike of stock standard B (mL)*	5
	<b>concentrations (mg / L)</b>
Be	10
Co	7.5
Mo	300
Sb	8
Cs	20
Ba	20
W	6
Pt	2.5
Tl	4
Pb	10
U	0.5
Cd	8
Sr	600
Sn	30
Mn	10
*If preparing from HPS # SM-2107-037, both stock solutions A and B need to be spiked into the flask	

**Table 5. Preparation of multi-element intermediate working standards.**

standard #	0	1	2	3	4	5
flask vol. (mL)	0	500	200	100	100	100
vol. spike of int. stock std. (mL)	0	0.050	0.060	0.100	0.300	1.00
<b>concentrations (µg/L) ‡</b>						
Be	0	1 (0.1) ‡	3 (0.3) ‡	10 (1.0) ‡	30 (3.0) ‡	100 (10.0) ‡
Co	0	0.75 (0.075) ‡	2.25 (0.225) ‡	7.5 (0.75) ‡	22.5 (2.25) ‡	75 (7.5) ‡
Mo	0	30 (3.0) ‡	90 (9.0) ‡	300 (30) ‡	900 (90) ‡	3000 (300) ‡
Sb	0	0.8 (0.08) ‡	2.4 (0.24) ‡	8 (0.8) ‡	24 (2.4) ‡	80 (8.0) ‡
Cs	0	2 (0.2) ‡	6 (0.6) ‡	20 (2.0) ‡	60 (6.0) ‡	200 (20) ‡
Ba	0	2 (0.2) ‡	6 (0.6) ‡	20 (2.0) ‡	60 (6.0) ‡	200 (20) ‡
W	0	0.6 (0.06) ‡	1.8 (0.18) ‡	6 (0.6) ‡	18 (1.8) ‡	60 (6.0) ‡
Pt	0	0.25 (0.025) ‡	0.75 (0.075) ‡	2.5 (0.25) ‡	7.5 (0.75) ‡	25 (2.5) ‡
Tl	0	0.4 (0.04) ‡	1.2 (0.12) ‡	4 (0.4) ‡	12 (1.2) ‡	40 (4.0) ‡
Pb	0	1 (0.1) ‡	3 (0.3) ‡	10 (1.0) ‡	30 (3.0) ‡	100 (10) ‡
U	0	0.05 (0.005) ‡	0.15 (0.015) ‡	0.5 (0.05) ‡	1.5 (0.15) ‡	5 (0.5) ‡
Cd	0	0.8 (0.08) ‡	2.4 (0.24) ‡	8 (0.8) ‡	24 (2.4) ‡	80 (8.0) ‡
Sr	0	60 (6.0) ‡	180 (18.0) ‡	600 (60) ‡	1800 (180) ‡	6000 (600) ‡
Sn	0	3 (0.3) ‡	9 (0.9) ‡	30 (3.0) ‡	90 (9.0) ‡	300 (30) ‡
Mn	0	1 (0.1) ‡	3 (0.3) ‡	10 (1.0) ‡	30 (3.0) ‡	100 (10) ‡
<b>‡ A further 1:10 dilution occurs when added to base urine. Enter concentrations in parentheses into the ICP-MS software (method window, calibration page).</b>						

**Table 6. Acceptable ways to perform two consecutive analytical runs, bracketing with bench quality control samples.**

<b>setup 1</b>	<b>setup 2 (typical)</b>
<p><i>Run #1</i>  calibrators  low bench QC  high bench QC  patient samples  low bench QC  high bench QC</p> <p><i>Run #2</i>  low bench QC  high bench QC  patient samples  low bench QC  high bench QC</p>	<p><i>Run #1</i>  calibrators  low bench QC  high bench QC  patient samples  low bench QC  high bench QC</p> <p><i>Run #2</i>  calibrators  low bench QC  high bench QC  patient samples  low bench QC  high bench QC</p>

**Table 7. A typical SAMPLE/BATCH window.**

<u>AS location*</u>	<u>sample id</u>	<u>measurements action</u>	<u>method</u>
236	DRCstability1	Run sample	....15elem_urblk.mth
236	DRCstability2	Run sample	....15elem_urblk.mth
236	DRCstability3	Run sample	....15elem_urblk.mth
236	DRCstability4	Run sample	....15elem_urblk.mth
Continue DRC stability samples . . .			
236	DRCstability11	Run sample	....15elem_urblk.mth
236	DRCstability12 <sup>£</sup>	Run sample	....15elem_urblk.mth
101	3018 UrBlkChk Wash1	Run blank, standards, and sample **	....15elem_urblk.mth
113	3018 UrBlkChk Wash2	Run sample	....15elem_urblk.mth
114	3018 UrBlkChk1	Run sample	....15elem_urblk.mth
115	3018 UrBlkChk2	Run sample	....15elem_urblk.mth
150	3018 AQBLK	Run blank and sample ¥	....15elem_aqblk.mth
136	L Bench QC	Run sample	....15elem_aqblk.mth
160	H Bench QC	Run sample	....15elem_aqblk.mth
301	Sample 1	Run sample	....15elem_aqblk.mth
302	Sample 2	Run sample	....15elem_aqblk.mth
303	Sample 3	Run sample	....15elem_aqblk.mth
135	L Bench QC	Run sample	....15elem_aqblk.mth
159	H Bench QC	Run sample	....15elem_aqblk.mth

\* The exact autosampler positions of QCs and patient samples do not have to be those shown above. QC samples do not have to be run in the order of low, then high, then elevated.

\*\* When executing this row, the ICP-MS will first analyze the urine blank (standard 0) at AS position 102, then standards 1-5 at autosampler positions 102-107, then the "UrBlkChk wash1" sample at A/S position 102. The sampling information about AS positions 103-107 are stored in the "urblk" method file and can be customized.

¥ When executing this row, the ICP-MS will first analyze the aqueous blank at AS position 149, then the "AQBlkChk" at AS position 150. The sampling information about AS positions 149 is stored in the "aqblk" method file and can be customized.

£ A larger number of DRC stability samples will need to be analyzed to make this stability period 1-1.5 hrs when measuring only Cd or Mn (50 measurements ~ 1hour).

**Table 8. Preparation of samples, working standards, and QC materials.**

Dilution ID	Water (μL)	Base urine (μL)	AQ intermediate working standard (μL)	Patient or QC urine sample (μL)	Diluent ** (μL)	Total Volume (μL)
working calibrators (S0-S5) and UrBlkChk (S0)	-	900 x 1	100 x 1	-	9,000 (4,500 x 2)*	10,000
AQ blank	1000 x 1	-	-	-	9,000 (4,500 x 2)*	10,000
patient urine or urine-based QC	-	-	-	500 x 1	4,500 x 1	5,000
patient urine <i>2x dilution</i> <sup>H</sup>	500 x 1	-	-	500 x 1	9,000 (4,500 x 2)*	10,000
patient urine <i>5x dilution</i> <sup>H</sup>	800 x 1	-	-	200 x 1	9,000 (4,500 x 2)*	10,000
patient urine <i>10x dilution</i> <sup>H</sup>	900 x 1	-	-	100 x 1	9,000 (4,500 x 2)*	10,000
patient urine <i>20x dilution</i> <sup>H</sup>	4,750 (950 x 5)	-	-	250 x 1	45,000 (7,500 x 6)*	50,000
<b>NOTE:</b> These directions are written with the expectation of a 10,000 μL syringe on the left side and a 1,000 μL syringe on the right side of the benchtop automatic pipette. If a different total volume is prepared, adjust the volumes for each component proportionally.						
** By splitting the dispense step of diluent into two or more portions, liquids pulled up into the right pipette tip are flushed out more completely. For example, when preparing a working calibrator dilution, do the preparation in two steps: in step 1, dispense 4500 μL diluent + 100 μL; in step 2, dispense 4500 μL diluent + 900 μL base urine to prepare a 10 mL total volume dilution.						
<sup>H</sup> Extra dilution is performed on urine samples whose concentration is greater than the concentration of the highest calibrator listed in Table 9 of Appendix C. Any extra dilution within these limits can be prepared as long as the 9:10 ratio of diluent to total dilution volume is maintained. Use of the lowest possible dilution level is preferred to minimize differences between the calibrators and the samples (i.e. 2x dilution is preferred over 5x if 2x is sufficient to dilute analyte into the documented linearity range).						



**Table 9. Reportable range concentrations ( $\mu\text{g/L}$ ).**

Analyte	Limit of Detection (LOD)*	High Calibrator	Maximum Extra Dilution**	Reportable Range Upper Boundary
Be	0.016	10	20	200
Co	0.023	7.5	20	150
Sr	2.34	600	2	1200
Mo	0.80	300	10	3000
Sn	0.09	30	20	600
Sb	0.022	8	20	160
Cs	0.086	20	20	400
Ba	0.06	20	20	400
W	0.018	6	20	120
Pt	0.010	2.5	10	25
Tl	0.018	4	5	20
Pb	0.03	10	4	40
U	0.0020	0.5	20	10
Cd	0.036	8	2	16
Mn	0.13	10	20	200
*Re-evaluated periodically (2+ years) or at significant method changes. LODs shown were calculated 01/08/2013.				
**See ruggedness test 5 in Appendix B for supporting validation data.				

**Table 10. Boundary concentrations for urine concentrations (µg/L).**

analyte	1 <sup>st</sup> upper boundary ("1UB") *	2 <sup>nd</sup> upper boundary ("2UB") **	Range Maximum ("Lim Rep Delta") †	Highest Concentration Validated for Washout
Be	0.2	0.4	0.3 µg/L for values <3 µg/L 10% of value at ≥3 µg/L	300
Co	2.83	5.66	0.3 µg/L for values <3 µg/L 10% of value at ≥3 µg/L	225
Sr	400	800	3 µg/L for values <30 µg/L 10% of value at ≥30 µg/L	18,000
Mo	293.5	587	4.0 µg/L for values <40 µg/L 10% of value at ≥40 µg/L	9,000
Sn	25	50	0.5 µg/L for values <5 µg/L 10% of value at ≥5 µg/L	900
Sb	0.8	1.6	0.2 µg/L for values <2 µg/L 10% of value at ≥2 µg/L	240
Cs	16.5	33	0.5 µg/L for values <5 µg/L 10% of value at ≥5 µg/L	600
Ba	17.1	34.2	0.4 µg/L for values <4 µg/L 10% of value at ≥4 µg/L	600
W	1.38	2.76	0.2 µg/L for values <2 µg/L 10% of value at ≥2 µg/L	180
Pt	0.2	0.4	0.2 µg/L for values <2 µg/L 10% of value at ≥2 µg/L	75
Tl	0.62	1.24	0.2 µg/L for values <2 µg/L 10% of value at ≥2 µg/L	120
Pb	7.8	15.6	0.3 µg/L for values <3 µg/L 10% of value at ≥3 µg/L	300
U	0.277	0.554	0.03 µg/L for values <0.3 µg/L 10% of value at ≥0.3 µg/L	15
Cd	2.54	5.08	0.3 µg/L for values <3 µg/L 10% of value at ≥3 µg/L	240
Mn	4	8	0.4 µg/L for values <4 µg/L 10% of value at ≥4 µg/L	300

\* Typically, the 1UB threshold is based on percentiles of non-weighted, non-creatinine corrected concentration results from NHANES. In the absence of that data, these boundaries can be based on normal ranges reported in the literature. The concentrations assigned to these boundaries is determined by study protocol but default concentrations are listed in this table.

\*\*Typically the 2<sup>nd</sup> upper boundary (2UB) is set to 2x the 1UB. The concentrations is determined by study protocol but default concentrations are listed in this table.

† Range maximum is the range of the three replicate readings for a single sample analysis. This value is also called the "Lim RepDelta" in the database which handles data for the Inorganic and Radiation Analytical Toxicology Branch.

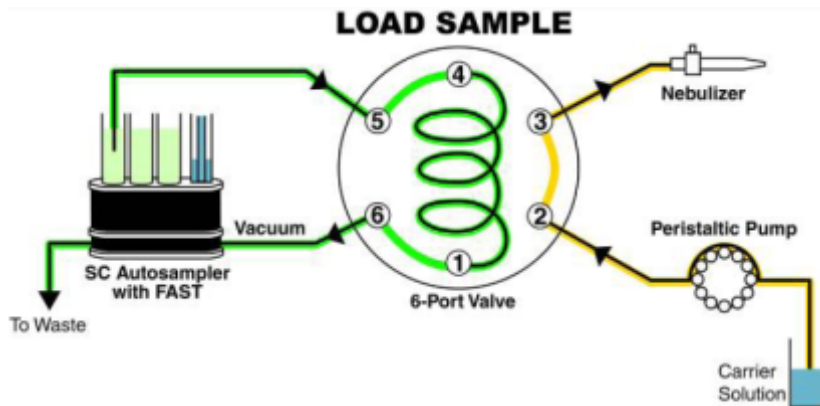
**Table 11. Reference ranges for urine concentrations (from the Fourth National Report on Exposure to Environmental Chemicals [8]). All results in µg/L.**

analyte	survey years	geometric mean	50 <sup>th</sup>	75 <sup>th</sup>	90 <sup>th</sup>	95 <sup>th</sup>	N
Be	09-10*	≤ 0.072	≤ 0.072	≤ 0.072	≤ 0.072	≤ 0.072	2848
Co	13-14	0.391	0.408	0.687	1.04	1.235	2664
Sr	13-14	81.2	88.1	151	231	299	2664
Mo	13-14	33.9	36.0	68.0	105	139	2664
Sn	13-14	0.433	0.380	0.930	2.30	4.14	2664
Sb	13-14	0.043	0.041	0.076	0.130	0.189	2664
Cs	13-14	3.94	4.21	6.63	9.31	11.3	2664
Ba	13-14	1.09	1.10	2.17	4.01	5.67	2664
W	13-14	0.059	0.058	0.129	0.241	0.380	2664
Pt	09-10*	≤ 0.009	≤ 0.009	≤ 0.009	0.009	0.016	2847
Tl	13-14	0.141	0.154	0.248	0.349	0.421	2664
Pb	13-14	0.277	0.290	0.500	0.840	1.17	2664
U	13-14	0.005	0.005	0.010	0.023	0.039	2664
Cd	13-14	0.124	0.126	0.273	0.576	0.842	2664
Mn	13-14	≤ 0.13	≤ 0.13	0.130	0.220	0.300	2664

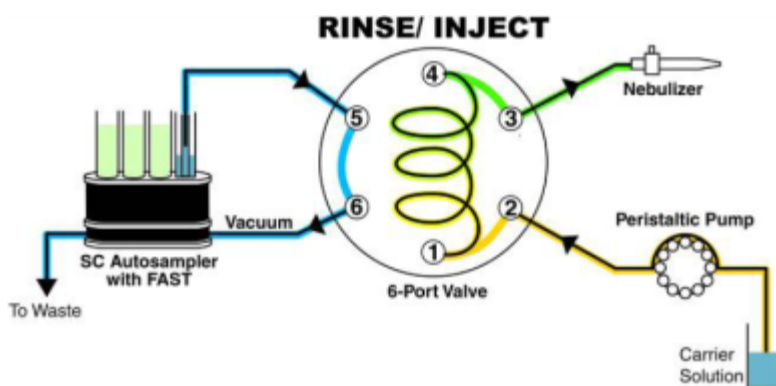
\*Indicates the final cycle year element was measured in the Survey.

**Figure 1. Configuration of tubing and devices for liquid handling using FAST sample introduction.**

Below shows the correct connections to the 6-port FAST valve. The two diagrams show the differences in liquid flow directions when the valve changes from “Load” to “Inject”. This change is internal to the valve. The shift of the valve cannot be seen, but it can be heard, and felt (with hand on the valve). The light indicators on the actuator body also indicate the valve position.



Teflon vacuum pump loads sample into loop while carrier solution is nebulized



Carrier solution pushes sample into nebulizer at the same time sample line is rinsed

The connections to the valve are color-coded (see Section 7.a.i).

Enable the FAST program in the ESI software before running the method, but optimizations can be done in either FAST or non-FAST mode.

Figure 2. NexION ICP-MS method screen shots (timing page) from Syngistix v1.3.

Syngistix™ for ICP-MS - Instrument Control Session

Control Devices Instrument Applications

SmartTune Conditions LogBook Optimize

Method Sample Dataset Analyze

Reporter RealTime Interactive CalibView Results

Scheduler Review Work Flow

Quantitative Analysis Method - C:\Users\Public\Documents\PerkinElmer\Syngistix\ICPMS\Method\3018\_15 element method\cdc\_dfls3018\_15 element 5calibr\_apblik.mth[Modified]

Processing Equation Calibration Sampling Devices... QC... Report Notes

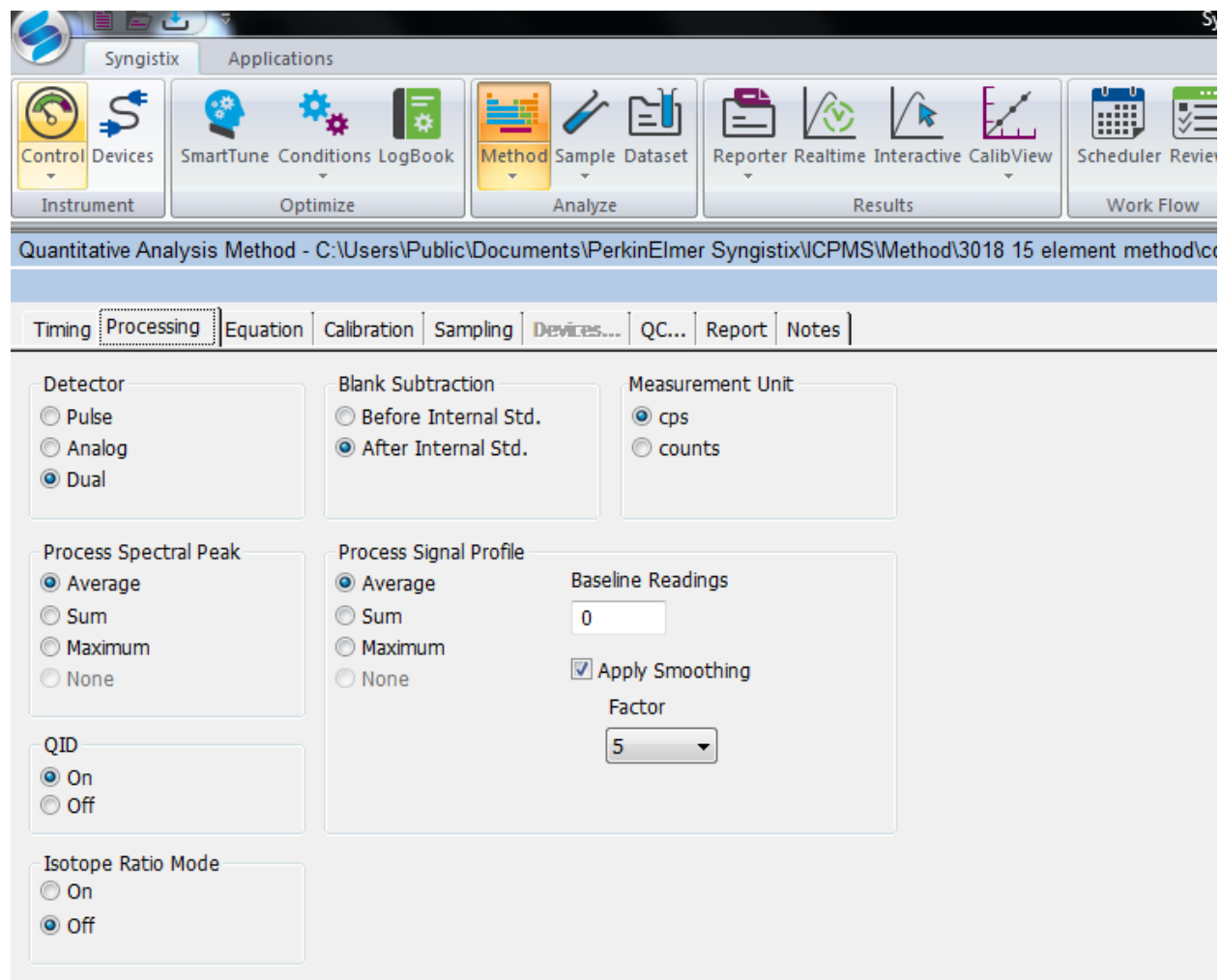
Sweeps / Reading 40 Est. Reading Time 0:00:46.784 MassCal File default.tun Browse...

Readings / Replicate 1 Est. Replicate Time 0:00:46.784 Conditions File c:\users\public\documents\perkinelr Browse...

Replicates 3 Est. Sample Time 0:02:50.352  Enable QC Checking

Int Std	Analyte	Mass (amu)	Scan Mode (*)	MCA Channels	Dwell Time per AMU (ms)	Integration Time (ms)	Corrections	Mode (*)	Cell Gas A	Cell Gas B	RP a	RP q
1	Be	9.0122	Peak Hopping	1	100	4000		Standard	0	0	0	0.25
2	Co	58.9332	Peak Hopping	1	30	1200		Standard	0	0	0	0.25
3	Sr	87.9056	Peak Hopping	1	30	1200		Standard	0	0	0	0.25
4	Mo	97.9055	Peak Hopping	1	30	1200		Standard	0	0	0	0.25
5	Rh	102.905	Peak Hopping	1	30	1200		Standard	0	0	0	0.25
6	Sn	117.902	Peak Hopping	1	30	1200		Standard	0	0	0	0.25
7	Sb	120.904	Peak Hopping	1	30	1200		Standard	0	0	0	0.25
8	Cs	132.905	Peak Hopping	1	30	1200		Standard	0	0	0	0.25
9	Ba	137.905	Peak Hopping	1	30	1200		Standard	0	0	0	0.25
10	W	183.951	Peak Hopping	1	30	1200		Standard	0	0	0	0.25
11	Ir	192.963	Peak Hopping	1	30	1200		Standard	0	0	0	0.25
12	Pt	194.965	Peak Hopping	1	100	4000		Standard	0	0	0	0.25
13	Tl	204.975	Peak Hopping	1	30	1200		Standard	0	0	0	0.25
14	Pb	207.977	Peak Hopping	1	30	1200	Pb, Pb	Standard	0	0	0	0.25
15	U	238.05	Peak Hopping	1	100	4000	U	Standard	0	0	0	0.25
16	Cd	113.904	Peak Hopping	1	100	4000	Sn	DRC	0	2.3	0	0.75
17	Mn	54.9381	Peak Hopping	1	100	4000		DRC	0	2.3	0	0.75
18	Ir-1	192.963	Peak Hopping	1	30	1200		DRC	0	2.3	0	0.75

Figure 3. NexION ICP-MS method screen shots (processing page) from NexION v1.3.



**Figure 4. NexION ICP-MS method screen shots (equation page) from NexION v1.3.**

Quantitative Analysis Method - C:\Users\Public\Documents\PerkinElmer Syngistix\ICPMS\Method\3018\_15 element method\cdc\_dls3018\_15 element 5calibr\_a

Timing | Processing | **Equation** | Calibration | Sampling | Devices... | QC... | Report | Notes

Isotope Information

Isotope	Mass	Abundance	Interferences
Be 9	9.0122	100.000000	

	Int Std	Analyte	Mass (amu)	Corrections	Potential Interferences
1		Be	9.0122		
2		Co	58.9332		CaO
3		Sr	87.9056		Yb++, Lu++
4		Mo	97.9055		Ru, BrO
5	▶	Rh	102.905		SrO
6		Sn	117.902		MoO, U++
7		Sb	120.904		
8		Cs	132.905		
9		Ba	137.905		La, Ce
10		W	183.951		Os, ErO, YbO
11	▶	Ir	192.963		HfO, LuO
12		Pt	194.965		HfO
13		Tl	204.975		
14		Pb	207.977	+ Pb 206 + Pb 207	
15		U	238.05	+ U 235	
16		Cd	113.904	- 0.027250 * Sn 118	Sn, MoO
17		Mn	54.9381		ArN, HClO, ClO
18	▶	Ir-1	192.963		HfO, LuO

Figure 5. NexION ICP-MS method screen shots (calibration page) from NexION v1.3.

Quantitative Analysis Method - C:\Users\Public\Documents\PerkinElmer Syngistix\ICPMS\Method\3018\_15 element method\cdc\_dis3018\_15 element 5calibr\_aqblk.mth

Timing | Processing | Equation | Calibration | Sampling | Devices... | QC... | Report | Notes

External Std.  
 Std. Addition

Int Std	Analyte	Mass (amu)	Curve Type (*)	Sample Units (*)	Standard Units (*)	Std 1	Std 2	Std 3	Std 4	Std 5	Std 6	Std 7
1	Be	9.0122	Weighted Linear	ppb	ppb	0.1	0.3	1	3	10		
2	Co	58.9332	Weighted Linear	ppb	ppb	0.075	0.225	0.75	2.25	7.5		
3	Sr	87.9056	Weighted Linear	ppb	ppb	6	18	60	180	600		
4	Mo	97.9055	Weighted Linear	ppb	ppb	3	9	30	90	300		
5	Rh	102.905	Weighted Linear	ppb	ppb							
6	Sn	117.902	Weighted Linear	ppb	ppb	0.3	0.9	3	9	30		
7	Sb	120.904	Weighted Linear	ppb	ppb	0.08	0.24	0.8	2.4	8		
8	Cs	132.905	Weighted Linear	ppb	ppb	0.2	0.6	2	6	20		
9	Ba	137.905	Weighted Linear	ppb	ppb	0.2	0.6	2	6	20		
10	W	183.951	Weighted Linear	ppb	ppb	0.06	0.18	0.6	1.8	6		
11	Ir	192.963	Weighted Linear	ppb	ppb							
12	Pt	194.965	Weighted Linear	ppb	ppb	0.025	0.075	0.25	0.75	2.5		
13	Tl	204.975	Weighted Linear	ppb	ppb	0.04	0.12	0.4	1.2	4		
14	Pb	207.977	Weighted Linear	ppb	ppb	0.1	0.3	1	3	10		
15	U	238.05	Weighted Linear	ppb	ppb	0.005	0.015	0.05	0.15	0.5		
16	Cd	113.904	Weighted Linear	ppb	ppb	0.08	0.24	0.8	2.4	8		
17	Mn	54.9381	Weighted Linear	ppb	ppb	0.1	0.3	1	3	10		
18	Ir-1	192.963	Weighted Linear	ppb	ppb							



Figure 6. NexION ICP- MS method screen shots (sampling page) from NexION v1.3.

Quantitative Analysis Method - C:\Users\Public\Documents\PerkinElmer Syngistix\ICPMS\Method\3018 15 element met

Timing | Processing | Equation | Calibration | **Sampling** | Devices... | QC... | Report | Notes

**Peristaltic Pump**

	Time (sec)	Speed (+/- rpm)
Sample Flush	12	-3.0
Read Delay	45	-3.0
Analysis		-3.0
Wash	50	-3.0

**Auto Diluter**

Dil. Factor:  Dil. To Vol. (mL):

1st. Dil. Pos:  Probe Purge Pos.:

**Sampling Device**

(None)

ESI

c:\programdata\esi\esi s

Peristaltic Pump Under Computer Control

	Standard	Solution ID	A/S Loc.	Wash Override (sec)
1	Blank		102	
2	Standard 1		103	
3	Standard 2		104	
4	Standard 3		105	
5	Standard 4		106	
6	Standard 5		107	

Figure 7. NexION ICP-MS method screen shots (report page) from NexION v1.3.

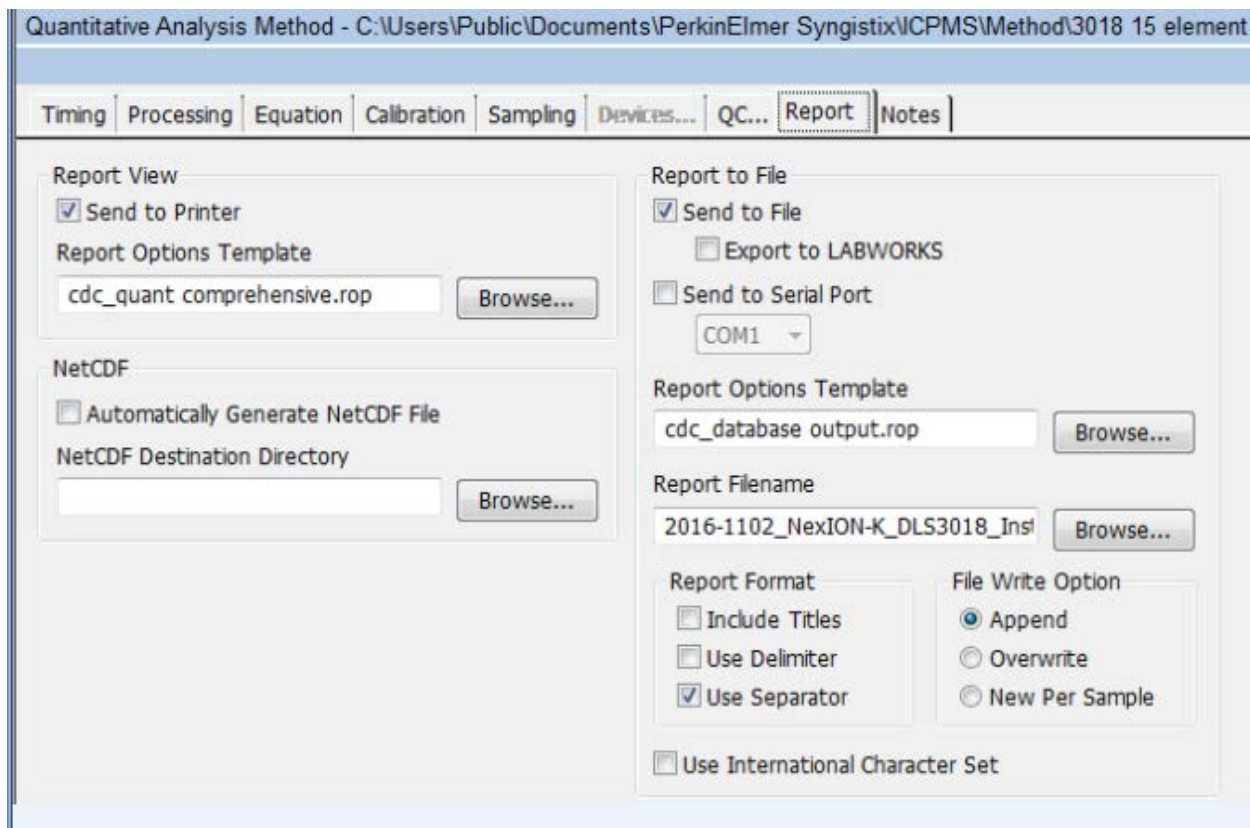


Figure 8. NexION ICP-MS method screen shots (QC / Sample page) from NexION v1.3.

Not all elements shown in bottom table, see Table 1 of Appendix C "Extended Washout".

Quantitative Analysis Method - C:\Users\Public\Documents\PerkinElmer Syngistix\ICPMS\Method\3018 15 elem

Timing	Processing	Equation	Calibration	Sampling	Devices...	QC...	Report	Notes
Analyte	Mass (amu)	QC Action Priority	Sample Lower (Conc.)	Sample Upper (Conc.)	Sample Conc SD	Sample Conc RSD		
1 Be	9.0122	2		300				
2 Co	58.9332	6		225				
3 Sr	87.9056	0		18000				
4 Mo	97.9055	25		900				
5 Sn	117.902	0		90				
6 Sb	120.904	13		24				
7 Cs	132.905	14		60				
8 Ba	137.905	15		600				
9 W	183.951	16		18				
10 Pt	194.965	18		75				
11 Tl	204.975	19		12				
12 Pb	207.977	20		300				
13 U	238.05	3		1.5				
14 Cd	113.904	4		24				
15 Mn	54.9381	1		30				

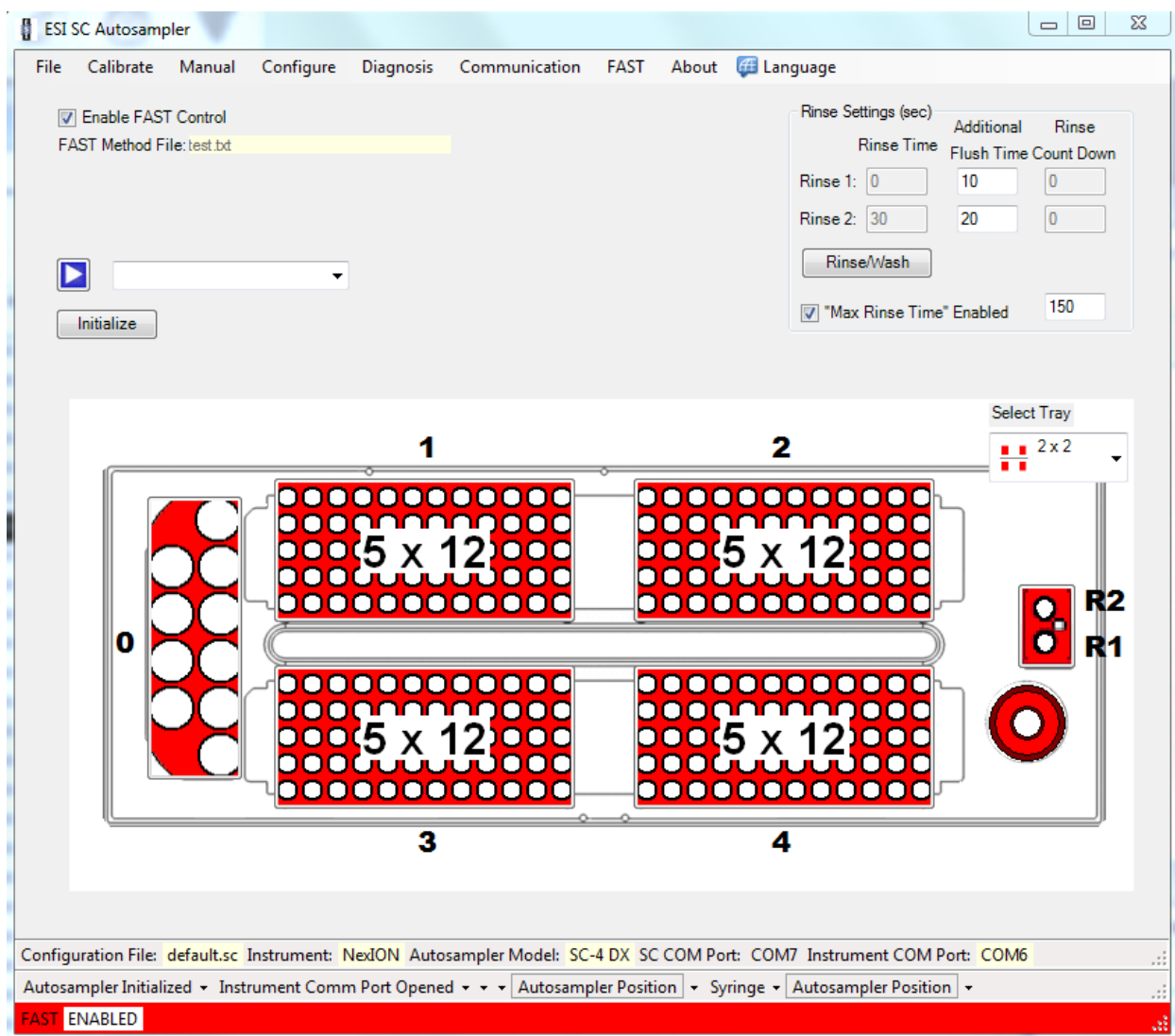
  

	Measurement	Action 1 (*)	Action 1 Data	Action 2 (*)	Action 2 Data
1	Be 9 Lower	Continue		Continue	
2	Be 9 Upper, S, EEE	Wash for X and Continue	200 seconds	Continue	
3	Be 9 Std Dev	Continue		Continue	
4	Be 9 RSD	Continue		Continue	
5	Co 59 Lower	Continue		Continue	
6	Co 59 Upper, S, EEE	Wash for X and Continue	200 seconds	Continue	
7	Co 59 Std Dev	Continue		Continue	
8	Co 59 RSD	Continue		Continue	
9	Sr 88 Lower	Continue		Continue	
10	Sr 88 Upper, S, EEE	Wash for X and Continue	200 seconds	Continue	
11	Sr 88 Std Dev	Continue		Continue	
12	Sr 88 RSD	Continue		Continue	
13	Mo 98 Lower	Continue		Continue	
14	Mo 98 Upper, S, EEE	Wash for X and Continue	200 seconds	Continue	
15	Mo 98 Std Dev	Continue		Continue	
16	Mo 98 RSD	Continue		Continue	

Navigation: < | > \ Calibration \ QC Stds. \ QC Measurement Frequency \ QC Std. Int. Stds. \ Calibration Stds. \ Sample Int Stds \ Sample

**Figure 9. ESI SC4 autosampler screen shots (main page).**

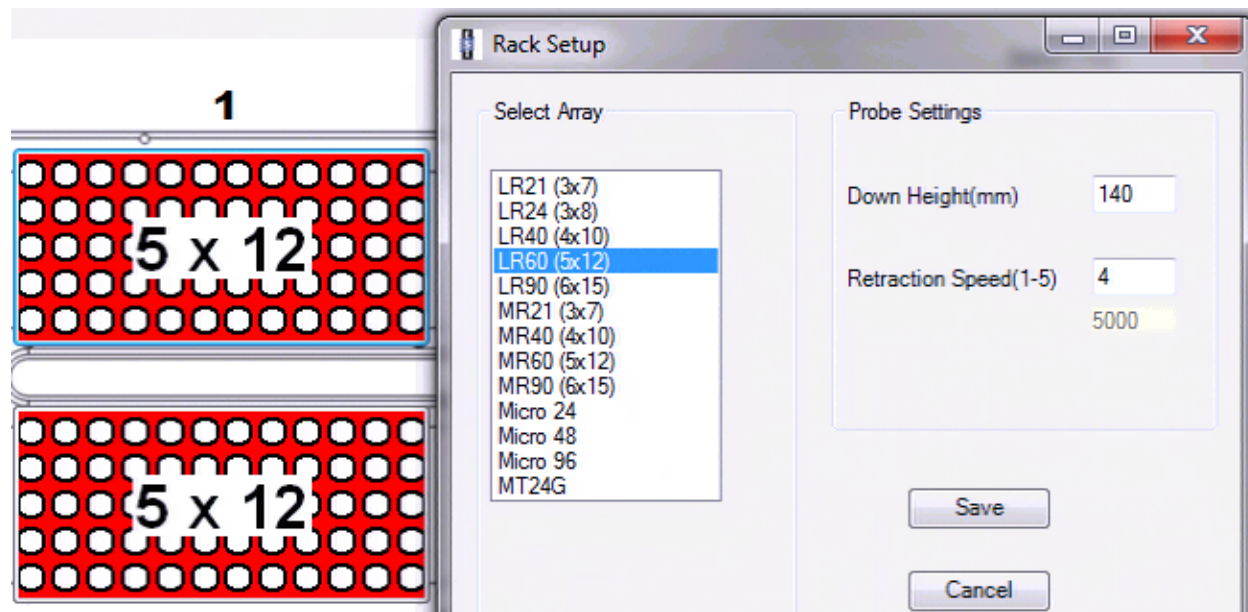
Additional flush times and “Max Rinse Time” are default, but can be optimized for best reduction of elemental carry-over between samples. Tray types can be changed to allow for different volumes of diluted sample digests. ‘FAST control’ must be enabled before start of method, but does not need to be used in instrument optimization (pre-analysis) steps. Rinse and additional flush times for eliminating carry-over from one sample to the next while using the minimum amount of rinse solution.



A rinse time of 0 causes the probe to only dip into the station, but spends no time there. Additional flush times can be optimized to keep the rinse station full while not using too much rinse solution. The inner diameter size of the tubing providing the rinse solution to the rinse station determines how quickly the station will fill. Various sizes are available for purchase or can be made in the laboratory.

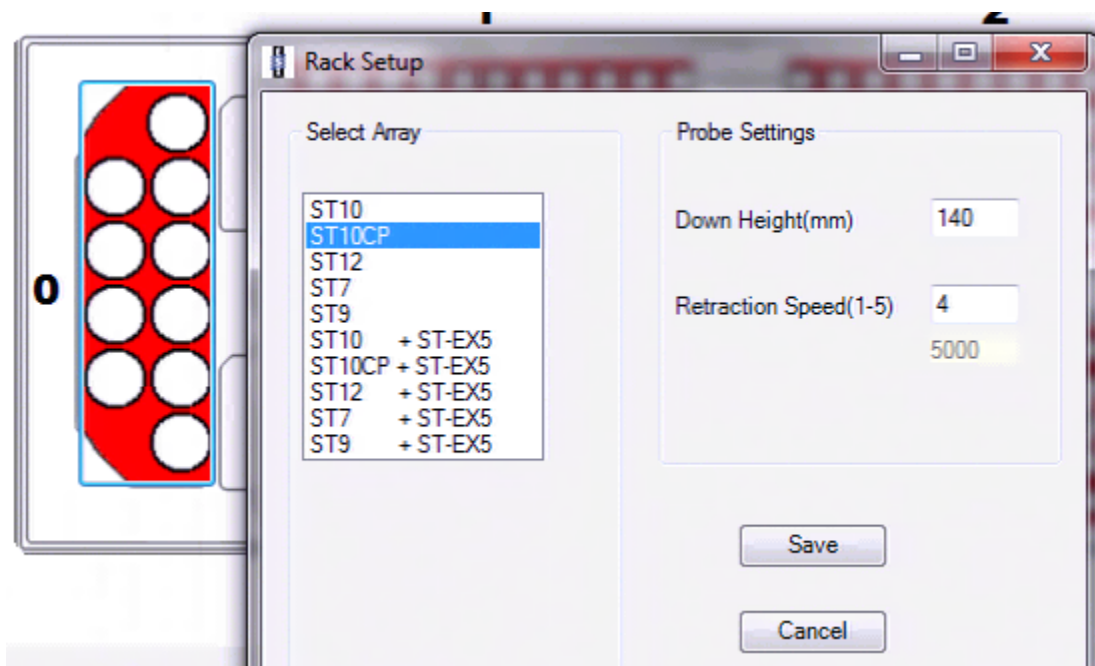
**Figure 10. ESI SC4 autosampler screen shots (5x12 rack setup window).**

Settings are approximate. To be sure the loop is filled, set the probe to go close to the bottom of the cup, but not touch. Optimize retraction speed for least droplet splatter.



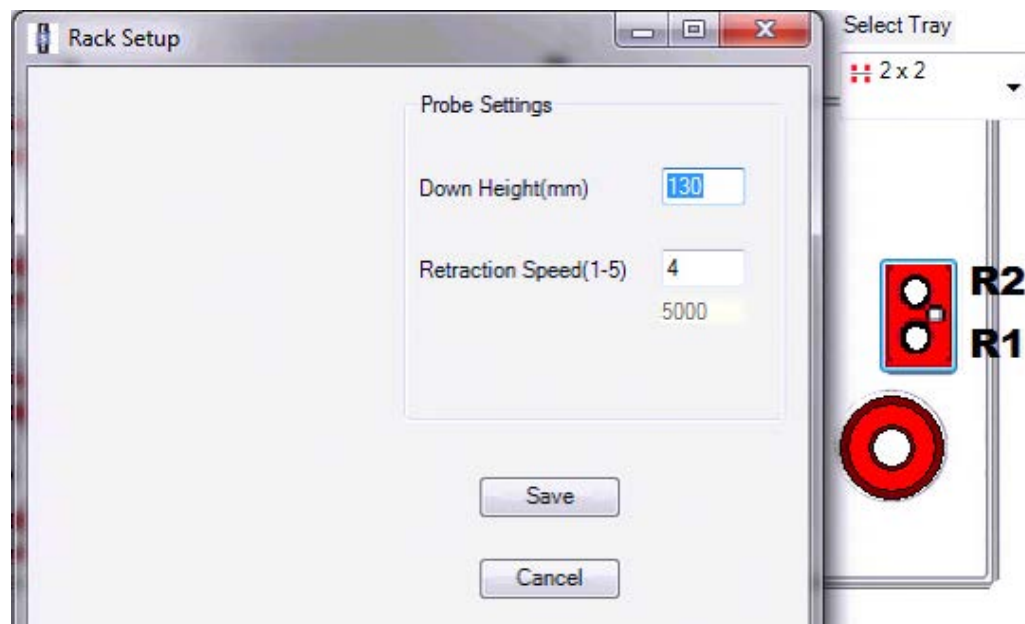
**Figure 11. ESI SC4 autosampler screen shots (50mL tube rack setup window).**

Settings are approximate. To be sure the loop is filled, set the probe to go close to the bottom of the cup, but not touch. Optimize retraction speed for least droplet splatter.



**Figure 12. ESI SC4 autosampler screen shots (rinse station rack setup window).**

Settings are approximate. Optimize down height for best probe cleaning, and retraction speed for least droplet splatter.



**Figure 13. ESI SC4 autosampler screen shots (“Configure” page).**

“High Speed” option is to only be used for ‘High Speed’ models of the SC4 (look for “HS” in serial number). Speeds and accel / decel values can be optimized per analyst preference and to minimize droplet splatter off of probe.

The screenshot shows the 'ConfigureAutosampler' dialog box with the following settings:

- Horizontal:** Start Speed 600, Max Speed 8000, Accel/Decel 8.  High Speed (HS).
- Rotational:** Start Speed 200, Max Speed 750, Accel/Decel 6.  Enable RAF 3.
- Vertical:** Start Speed 750, Max Speed 5000, Accel/Decel 8, Rail Height 16 inches.  High Speed (HS),  Enable Z Homing.
- Configuration File:** Configuration File Name default.sc. Buttons: Open File, Save File, Cancel.  Auto Initialize.
- Autosampler Model:** Autosampler Model SC-4.
- Instrument/Autosampler Emulation:** Instrument Type Perkin Elmer NexION, Autosampler Type AS 93, Dilutor Model None.

**Figure 14. ESI SC4 autosampler screen shots used (“Communication” page).**

Communication ports will differ depending on available ports on instrument control computer.

The screenshot shows the 'ConfigureCommunication' dialog box with the following settings:

- SC Autosampler Communication Port:** COM5
- Instrument Communication Port:** COM4
- Buttons: AutoConfigure, OK, Cancel.



**Figure 15. ESI SC4 autosampler screen shots (“FAST” page).**

Timer A can be optimized to achieve proper filling of loop with diluted sample. Timers B – H control rinsing the loop after analysis and can be optimized for eliminating carry-over from one sample to the next while using the minimum amount of rinse solution. Timer I expires (line 29) only when the extended wash time is triggered (see QC enable Figure 2g) and has the probe return to rinse station 2 for 100 seconds (Timer J). See Figure 3g and 3h for the entire program.

Manually clicking the “Load” button prior to starting analysis will ensure the position of the actuator is always the same at the beginning of the analysis.

Manually clicking the “Vacuum On” button prior to starting the analysis will help initial sample uptake to be consistent.

The screenshot shows the 'FAST Method Control' software interface. On the left is a table with columns: Reload, Event, Action, Parameter, Parameter Units, and Event Parameter. The table lists 32 rows of events and actions, including 'On Probe Down', 'Timer A Expires', 'Rinse Completed', and 'Timer I Expires'. The right side of the interface contains several control panels:

- FAST Control:** Includes a checked 'Enable FAST Control' box, 'Method File Name: CDC\_DLS3018\_15 element\_Loop 3', and 'Rinse Time (s)' fields for Rinse1 (0) and Rinse2 (35). A 'Max Vacuum Time (s)' field is set to 300.
- Events & Actions:** A tabbed interface with sub-tabs for FAST Control, Syringe, Dilution, Peripump, and Flow Controller. It contains lists of actions for 'Host Instrument', 'FAST', 'Syringe', 'Autosampler', 'Valve', 'Peripump', 'Gas Flow', and 'Vorso Rack'.

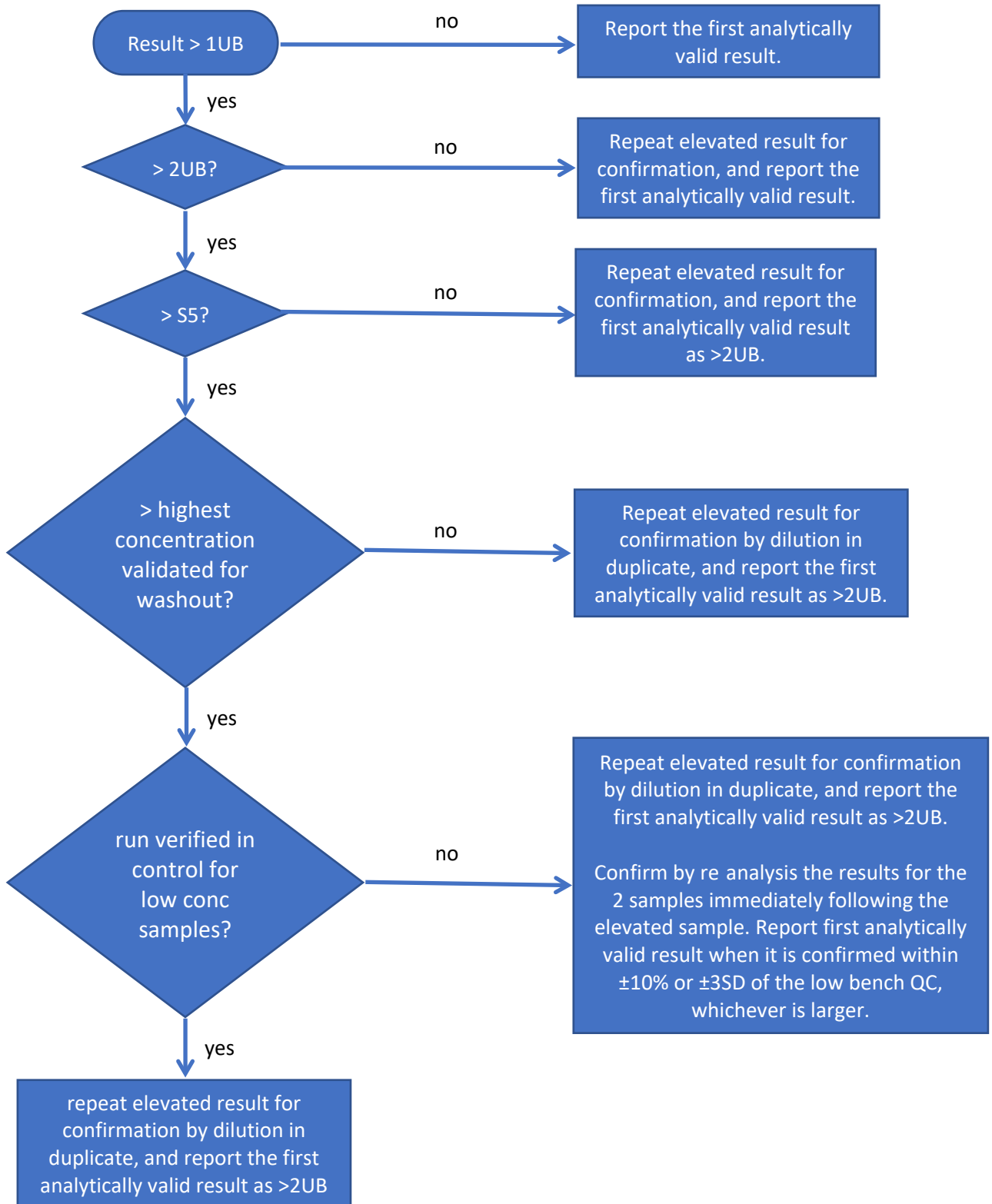


**Figure 16. Entire ESI SC4 autosampler “FAST” program.**

Line	Event	Action	Parameters	Parameter Units
1	On Probe Down	Vacuum1 On		
2	On Probe Down	Load1		
3	Probe In Sample	Timer A	11	seconds
4	Timer A Expires	Inject1		
	Timer A Expires	Move Rinse		
6	Rinse Completed	Probe Up		
7	On Rinse	Vacuum1 On		
8	On Rinse	Probe Down		
9	On Rinse	Load1		
10	On Rinse	Timer B	4	seconds
11	Timer B Expires	Inject1		
12	Timer B Expires	Timer C	4	seconds
13	Timer B Expires	Probe Up		
14	Timer B Expires	A2 On		
	Timer C Expires	Probe Down		
16	Timer C Expires	Load1		
17	Timer C Expires	Timer D	4	seconds
18	Timer D Expires	Probe Up		
19	Timer D Expires	Timer E	4	seconds
20	Timer D Expires	A2 Off		
21	Timer E Expires	Probe Down		
22	Timer E Expires	Timer F	4	seconds
23	Timer F Expires	Probe Up		
24	Timer F Expires	Timer G	4	seconds
	Timer G Expires	Probe Down		
26	Timer G Expires	Timer H	4	seconds
27	Timer H Expires	Move Next		
28	On Rinse	Timer I	75	seconds
29	Timer I Expires	Move Into(rrvv)	R2	rrvv
30	Timer I Expires	Timer J	100	seconds
31	Timer J Expires	Probe Up		

**NOTE:** “Probe in Sample” time will need to be optimized for the appropriate number of elements being analyzed (e.g. 1, 8, 15, etc...) and to ensure no air gaps remain in the loop after filling. Typical times are 8–15 seconds for a 3mL loop (15 elements), 6–10 seconds for a 2mL loop (e.g. 8 element subset), 4–6 seconds for a 1mL loop (e.g. 3 element subset), or 3-5 seconds for a 0.5 mL loop (e.g. 1 element). Customized loop sizes can be created. Fill times need to be optimized per instrument setup.

**Figure 17. Flow chart for handling an elevated result**



## 18. Appendix D: Help Sheets

### Reagent Preparation Help Sheets, page 1 of 2

mg/L = ppm,  $\mu\text{g/L}$  = ppb, and  $\mu\text{g/mL}$  = ppm

#### Rinse Solution – 4 L

##### (5.0% (v/v) HNO<sub>3</sub>, 0.002% Triton X-100, 500 $\mu\text{g/L}$ gold)

1. Partially fill a 4 liter bottle with 18 Mohm·cm water.
2. Add 4 mL of the 2% Triton X-100™ / 5% (v/v) nitric-acid intermediate stock solution.
3. Carefully add 200 mL of concentrated HNO<sub>3</sub>.
4. Add 200  $\mu\text{L}$  of the 10,000  $\mu\text{g/mL}$  gold internal standard solution.
5. Add enough  $\geq$ 18 Mohm·cm water to bring to 4 liter mark.
6. Mix well by gently inverting several times.
7. Label appropriately and store at ambient temperature.

#### Sample Diluent/Carrier Solution – 2 L

##### (2.0% (v/v) HNO<sub>3</sub>, 10 $\mu\text{g/L}$ Ir and Rh, 500 $\mu\text{g/L}$ Au)

1. Partially fill a 2 liter bottle with 18 M $\Omega$ ·cm water.
2. Add 40 mL concentrated HNO<sub>3</sub>.
3. Add 500  $\mu\text{L}$  of the 40  $\mu\text{g/mL}$  Rh and Ir internal standard solution.
4. Add 100  $\mu\text{L}$  of the 10,000  $\mu\text{g/mL}$  gold standard.
5. Add enough 18 Mohm·cm water to bring to 2 liter mark.
6. Mix well by gently inverting several times.
7. Label appropriately and store at ambient temperature.

#### Intermediate Internal Standard Solution – 200 mL

##### (2.0% (v/v) HNO<sub>3</sub>, 40 $\mu\text{g/mL}$ Ir and Rh)

1. Partially fill a 200 mL volumetric flask with 18 Mohm·cm water.
2. Add 4 mL of concentrated HNO<sub>3</sub>.
3. Add 8 mL of 1,000  $\mu\text{g/mL}$  Rh standard.
4. Add 8 mL of 1,000  $\mu\text{g/mL}$  Ir standard.
5. Add enough 18 Mohm·cm water to bring to 200 mL mark.
6. Mix well by gently inverting several times.
7. Label appropriately and store at ambient temperature.

#### 2% Triton X-100 in 5% (v/v) HNO<sub>3</sub> – 2 L

1. Partially fill a 2 liter bottle with 18 Mohm·cm water.
2. Add 100 mL of concentrated HNO<sub>3</sub>.
3. Add 40 mL of Triton X-100.
4. Add enough 18 Mohm·cm water to bring to 2 liter mark.
5. Add a clean Teflon magnetic stirring bar and stir on stirrer until dissolved.
6. Label appropriately and store at ambient temperature.

## **Reagent Preparation Help Sheets, page 2 of 2**

### **0.5% (v/v) HNO<sub>3</sub>**

1. Partially fill a 2 liter bottle with 18 MΩ·cm water.
2. Add 10 mL of concentrated HNO<sub>3</sub>.
3. Add enough 18 Mohm·cm water to bring to 2 liter mark.
4. Mix well by gently inverting several times.
5. Label appropriately and store at ambient temperature.

### **2% (v/v) HNO<sub>3</sub>**

1. Partially fill a 2 liter bottle with 18 MΩ·cm water.
2. Add 40 mL of concentrated HNO<sub>3</sub>.
3. Add enough 18 Mohm·cm water to bring to 2 liter mark.
4. Mix well by gently inverting several times.
5. Label appropriately and store at ambient temperature.

### **5% (v/v) HNO<sub>3</sub>**

1. Partially fill a 2 liter bottle with 18 MΩ·cm water.
2. Add 100 mL of concentrated HNO<sub>3</sub>.
3. Add enough 18 Mohm·cm water to bring to 2 liter mark.
4. Mix well by gently inverting several times.
5. Label appropriately and store at ambient temperature.

### **DRC Stability Solution – 200 mL**

1. Add 180 mL of diluent to 250 mL bottle.
2. Add 18 mL of human urine to bottle.
3. Add 2 mL of Standard 2 to bottle.
4. Mix well by gently inverting several times.
5. Label appropriately and store at ambient temperature.

### **Daily Performance Solution (1 µg/L) in 2% (v/v) HNO<sub>3</sub>**

1. Partially fill a 1 liter volumetric flask with 18 Mohm·cm water.
2. Add 1 mL of High Purity Standard: SM-2107-018.
3. Add 20 mL of concentrated HNO<sub>3</sub>.
4. Add enough 18 Mohm·cm water to bring to 1 liter mark.
5. Mix well by gently inverting several times.
6. Label appropriately and store at ambient temperature.

### **Dual Detector Solution**

1. Partially fill a 50 mL polypropylene tube with 2% (v/v) HNO<sub>3</sub>.
2. Add 100 µL of 100 µg/mL High Purity Standard: SM-2107-053.
3. Add 10 µL of any additional 1,000 µg/mL single element stock standard, if required.
4. Dilute to the 50 mL mark with 2% (v/v) HNO<sub>3</sub>.
5. Label appropriately and store at ambient temperature.

## 19. References

1. Thomas, R., *Practical guide to ICP-MS: a tutorial for beginners*. Third ed. 2013, New York, New York: Marcel Dekker. 336.
2. Tanner, S.D., Baranov, Vladimir I, *Theory, Design, and Operation of a Dynamic Reaction Cell for ICP-MS*. Atomic Spectroscopy, 1999. **20**(2): p. 45-52.
3. Tanner, S.D., V.I. Baranov, and D.R. Bandura, *Reaction cells and collision cells for ICP-MS: a tutorial review*. Spectrochimica Acta Part B-Atomic Spectroscopy, 2002. **57**(9): p. 1361-1452.
4. PerkinElmer SCIEX Instruments, *ELAN DRC II Hardware Guide*. 2001, Canada.
5. Jarrett, J.M., et al., *Eliminating molybdenum oxide interference in urine cadmium biomonitoring using ICP-DRC-MS*. 2008. **23**: p. 962-967.
6. Division of Laboratory Sciences, *Division of Laboratory Sciences Policies and Procedures Manual*. 2017, version 6.0, Centers for Disease Control and Prevention: Atlanta, GA.
7. U.S. Nuclear Regulatory Commission, *Regulatory guide 8.22 (revision 1). Bioassay at uranium mills*. 1988: Atlanta, GA.
8. Centers for Disease Control and Prevention, *Fourth National Report on Human Exposure to Environmental Chemicals, Updated Tables, January 2017, Volume 1*. 2017, CDC: Atlanta, GA.