ANALYSIS OF METALS USING THE THERMO ICAP INDUCTIVELY COUPLED PLASMA MASS SPECTROMETER (ICP-MS)

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1. SCOPE AND APPLICATION

1.1 This standard operating procedure (SOP) outlines the analysis of samples and sample digestates using Thermo iCAP (models Q and RQ) inductively coupled plasma mass spectrometry (ICP-MS).

1.2 The Florida Department of Environmental Protection Central Laboratory (DEP) analyzes for 21 elements using EPA method 200.8 (Revision 5.4) for test IDs W-ICPMS and W-ICPMS-F in aqueous and saline/estuarine matrices.

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	Analyte	Method	NELAP approval	Matrices
1	Al	200.8 Rev. 5.4	Yes	aqueous, saline/estuarine
2	Sb	200.8 Rev. 5.4	Yes	aqueous, saline/estuarine
3	As	200.8 Rev. 5.4	Yes	aqueous, saline/estuarine
4	Ba	200.8 Rev. 5.4	Yes	aqueous, saline/estuarine
5	Be	200.8 Rev. 5.4	Yes	aqueous, saline/estuarine
6	B	200.8 Rev. 5.4	Yes	aqueous, saline/estuarine
7	Cd	200.8 Rev. 5.4	Yes	aqueous, saline/estuarine
8	Cr	200.8 Rev. 5.4	Yes	aqueous, saline/estuarine
9	Co	200.8 Rev. 5.4	Yes	aqueous, saline/estuarine
10	Cu	200.8 Rev. 5.4	Yes	aqueous, saline/estuarine
11	Gd	200.8 Rev. 5.4	No	aqueous, saline/estuarine
12	Pb	200.8 Rev. 5.4	Yes	aqueous, saline/estuarine
13	Mn	200.8 Rev. 5.4	Yes	aqueous, saline/estuarine
14	Mo	200.8 Rev. 5.4	Yes	aqueous, saline/estuarine
15	Ni	200.8 Rev. 5.4	Yes	aqueous, saline/estuarine
16	Se	200.8 Rev. 5.4	Yes	aqueous, saline/estuarine
17	Sr	200.8 Rev. 5.4	No	aqueous, saline/estuarine
18	Ag	200.8 Rev. 5.4	Yes	aqueous, saline/estuarine
19	Tl	200.8 Rev. 5.4	Yes	aqueous, saline/estuarine
20	Zn	200.8 Rev. 5.4	Yes	aqueous, saline/estuarine
21	U	200.8 Rev. 5.4	No	aqueous, saline/estuarine

1.3 Samples are also analyzed using EPA method 6020A for 20 elements for tests W-ICPMS-R, S-ICPMS, T-ICPMS, TCLP-ICPMS, SPLP-ICPMS, and S-ICPMS-TO in aqueous, biological tissue, chemical waste, and solid matrices.

	Analyte	Method	NELAP approval	Matrices
1	Al	6020A	No	aqueous, biological tissue, chemical waste, solids
2	Sb	6020A	Yes*	aqueous, biological tissue, chemical waste, solids
3	As	6020A	Yes*	aqueous, biological tissue, chemical waste, solids
4	Ba	6020A	Yes*	aqueous, biological tissue, chemical waste, solids
5	Be	6020A	Yes*	aqueous, biological tissue, chemical waste, solids
6	B	6020A	No	aqueous
7	Cd	6020A	Yes*	aqueous, biological tissue, chemical waste, solids
8	Cr	6020A	Yes*	aqueous, biological tissue, chemical waste, solids
9	Co	6020A	Yes*	aqueous, biological tissue, chemical waste, solids
10	Cu	6020A	Yes*	aqueous, biological tissue, chemical waste, solids
11	Gd	6020A	No	aqueous

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12	Pb	6020A	Yes*	aqueous, biological tissue, chemical waste, solids
13	Mn	6020A	Yes*	aqueous, biological tissue, chemical waste, solids
14	Mo	6020A	Yes*	aqueous, biological tissue, chemical waste, solids
15	Ni	6020A	Yes*	aqueous, biological tissue, chemical waste, solids
16	Se	6020A	Yes*	aqueous, biological tissue, chemical waste, solids
17	Sr	6020A	No	aqueous
18	Ag	6020A	Yes*	aqueous, biological tissue, chemical waste, solids
19	Tl	6020A	Yes*	aqueous, biological tissue, chemical waste, solids
20	Zn	6020A	Yes*	aqueous, biological tissue, chemical waste, solids
21	U	6020A	No	aqueous

*Not NELAP approved for the biological tissue matrix.

2. SUMMARY OF THE METHOD

2.1 Aqueous samples and sample digestates are arranged on an autosampler. A peristaltic pump passes the liquid sample to the nebulizer. A sample aerosol is generated by the nebulizer. The nebulizer injects the sample aerosol into a Peltier cooled spray chamber which filters out larger aerosol droplets for improved signal stability and low oxide rates. The sample aerosol flows through the injector and into the torch where a radiofrequency plasma causes desolvation, atomization, and ionization. Ions generated in the plasma are transferred from atmospheric pressure to the vacuum region and introduced to the mass spectrometer as an ion beam. A series of lenses focus and accelerate the ion beam to the collision cell. The collision cell contains a quadrupole and is pressurized with a 93 % helium / 7 % hydrogen gas mixture gas for the iCAP Q and helium only for the iCAP RQ. Undesirable molecular ions are removed by kinetic energy discrimination. The ion beam is then introduced into the quadrupole mass analyzer, which filters out ions of specific mass to charge ratio, depending on the RF voltage and DC voltage applied to the quadrupole rods. The ions transmitted through the quadrupole are finally transferred to the dual mode secondary electron multiplier and detected.

3. INTERFERENCES

3.1 The primary interferences in ICP-MS are molecular ions such as oxides, doubly charged ions, and isobaric interferences. The interference check solution, IC6020C, is used to assess the effects of a variety of molecular interferences. The acceptable recovery limit for trace metals in IC6020C is 85 – 115 %. The table below lists common interferences observed while analyzing environmental samples. Bold font indicates the isotope reported.

Target Analyte	Common Interference	Monitor	Correction Based On
$^{9}Be^{+}$			
$^{11}B^{+}$			
$^{27}Al^{+}$			

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$^{52}Cr^{+}$	$^{40}Ar^{12}C^{+}$, $^{35}Cl^{16}O^{1}H^{+}$		
$^{53}Cr^{+}$	$^{37}Cl^{16}O^{+}$		
$^{55}Mn^{+}$			
$^{59}Co^{+}$			
$^{60}Ni^{+}$			
$^{62}Ni^{+}$	$^{46}Ti^{16}O^{+}$	$^{47}Ti^{+}$, $^{48}Ti^{+}$	
$^{63}Cu^{+}$	$^{40}Ar^{23}Na^{+}$, $^{47}Ti^{16}O^{+}$		
$^{65}Cu^{+}$	$^{49}Ti^{16}O^{+}$	$^{47}Ti^{+}$, $^{48}Ti^{+}$	
$^{66}Zn^{+}$	$^{50}Ti^{16}O^{+}$	$^{47}Ti^{+}$, $^{48}Ti^{+}$	
$^{67}Zn^{+}$			
$^{68}Zn^{+}$			
$^{75}As^{+}$	$^{40}Ar^{35}Cl^{+}$	$^{40}Ar^{37}Cl^{+}$	
$^{77}Se^{+}$	$^{40}Ar^{37}Cl^{+}$		
$^{78}Se^{+}$	$^{156}Gd^{2+}$, $^{40}Ar^{38}Ar^{+}$	^{83}Kr, ^{156}Gd	^{83}Kr, ^{156}Gd
$^{82}Se^{+}$	$^{82}Kr^{+}$		
$^{88}Sr^{+}$			
$^{92}Mo^{+}$			
$^{95}Mo^{+}$			
$^{97}Mo^{+}$			
$^{98}Mo^{+}$	$^{98}Ru^{+}$		
$^{106}Cd^{+}$	$^{90}Zr^{16}O^{+}$		
$^{107}Ag^{+}$	$^{91}Zr^{16}O^{+}$	$^{90}Zr^{+}$, $^{91}Zr^{+}$, $^{94}Zr^{+}$	
$^{108}Cd^{+}$	$^{92}Zr^{16}O^{+}$, $^{92}Mo^{16}O^{+}$		
$^{109}Ag^{+}$			
$^{111}Cd^{+}$	$^{95}Mo^{16}O^{+}$	$^{95}Mo^{+}$	
$^{114}Cd^{+}$	$^{98}Mo^{16}O^{+}$		
$^{121}Sb^{+}$			
$^{123}Sb^{+}$			
$^{135}Ba^{+}$			
$^{137}Ba^{+}$			
$^{158}Gd^{+}$			
$^{203}Tl^{+}$			
$^{205}Tl^{+}$			
$^{206}Pb^{+}$			
$^{207}Pb^{+}$			
$^{208}Pb^{+}$	Abundance	$^{206}Pb^{+}$, $^{207}Pb^{+}$	$^{206}Pb^{+}$, $^{207}Pb^{+}$
$^{238}U^{+}$			

3.2 To suppress polyatomic spectral interference, the Thermo iCAP Q/RQ employs a QCell (collision cell), along with a low mass cutoff and kinetic energy discrimination (KED).

3.3 Interference equation for Selenium: This interference equation subtracts the mass 78 krypton contribution and the doubly charged mass 156 contribution.

$^{78}Se = {}^{78}Se - (^{78}Kr/^{83}Kr)*^{83}Kr - (^{78}Se/^{156}Gd)*^{156}Gd$

$^{78}Kr/^{83}Kr$: Use the natural abundance, 0.030404348

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$^{78}Se/^{156}Gd$: Analyze a 25 µg/L Gd solution and use the counts per second (cps). Subtract the ^{78}Se calibration blank cps for this factor.

3.4 Interference equation for Lead: This equation corrects for the isotopic variation of lead isotopes.

$^{208}Pb = 1.0*^{206}Pb + 1.0*^{207}Pb$

4. APPARATUS AND EQUIPMENT

4.1 Inductively coupled plasma mass spectrometer

4.1.1 Thermo Scientific iCAP Q ICP-MS, serial number 03168R, purchased in 2014.

4.1.2 Thermo Scientific iCAP RQ ICP-MS, serial number 00090, purchased in 2015.

4.2 Interface: Nickel/Copper Sample cone (part # 3600812), nickel skimmer cone (part # 1311870), and skimmer cone insert (part # 1318480).

4.3 Nebulizer: Perfluoroalkoxy (PFA), part # 1317090.

4.4 Spray chamber: Peltier-cooled quartz cyclonic spray chamber, part # 1317080.

4.5 Torch: Quartz, part # 1230790.

4.5.1 Injector: Quartz (2.5 mm I.D.), part # 1305600.

4.6 Collision Cell gas:

4.6.1 iCAP Q: A mixture of 7 % hydrogen and 93 % helium, purity > 99.996 %, delivered pressure 7 – 14.5 psi.

4.6.2 iCAP RQ: Helium, purity > 99.996 %, delivered pressure 7 – 14.5 psi.

4.7 Autosampler:

4.7.1 iCAP Q: CETAC model ASX-520.

4.7.2 iCAP RQ: Teledyne CETAC model ASX-560.

4.8 Peristaltic Pump tubing

4.8.1 Sample line: orange-green-orange, part # 1320040.

4.8.2 Internal standard line: black-black-black, part # 1320060.

4.8.3 Drain line: gray-gray-gray, part # 1320110.

4.9 Samples and standards are aspirated from 17 x 100 mm polypropylene round-bottom test tubes (Fisherbrand 14-956-7e) arranged in the autosampler. Rinse with deionized (DI) water and allow to dry prior to use. Use a Kimwipe to prevent dust from entering the tubes as they dry.

4.10 Chiller: NESLAB ThermoFlex2500. During operation, 21 °C, 36 – 80 psi. Fill with distilled water.

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4.11 Argon is supplied to the instrument from the laboratory's Microbulk argon delivery system. The manufacturer's recommended delivery pressure to the instrument is 80 – 90 psi. However, Thermo service technicians have found better instrument performance if the delivery pressure is 90 – 100 psi. The purity must be 99.996 % or better.

4.12 Class A volumetric flasks.

4.13 Pipettes

4.13.1 Class A volumetric glass pipettes.

4.13.2 100 µL Eppendorf.

4.13.3 100 – 1000 µL adjustable volume Eppendorf.

4.13.4 2 – 10 mL Eppendorf.

4.14 HF Scientific Inc. MicroTPW Portable Turbidimeter, S/N 2022030090.

5. STANDARDS AND CHEMICALS

Note: All purchased and prepared standards must be logged into the Laboratory Information Management System (LIMS) standard preparation tracker module. A label must be printed from the standard preparation tracker and the label must be placed on the bottle of standard.

All purchased chemicals (HCl, HNO_3, etc.) shall have the received date documented on the bottle.

Save all certificates of analysis for standards and standard reference materials as pdf files in the network location, \\FLDEP1\Lab\Lab5\Chemistry\InorganicPrep\Certificates of Analysis for Reagents and Standards\.

5.1 Trace Metal Grade Nitric Acid (HNO_3): The expiration date is provided by the manufacturer. Stored in the fume hood cabinet at room temperature.

5.2 Trace Metal Grade Hydrochloric Acid (HCl): The expiration date is provided by the manufacturer. Stored in the fume hood cabinet at room temperature.

5.3 Rinse Solution: 4 % HNO_3 / 3 % HCl. Between samples and while idle, the autosampler probe is parked in the rinse station of the autosampler. The rinse station is filled with the probe rinse solution with an on-board peristaltic pump. Dilute 80 mL of HNO_3 and 60 mL of HCl into a 2000 mL container with DI water. The solution expires according to the manufacturer's expiration date of the purchased concentrated acids.

5.4 Acid Rinse Solution: 2 % HNO_3 / 1 % HCl. Poured directly into autosampler tubes and analyzed after high concentration samples, between the continuous calibration verification standard (CCV) and the continuous calibration blank (CCB), and at the end of the analytical run. Dilute 20 mL of HNO_3 and 10 mL of HCl into a 1000 mL container with DI water. The solution expires according to the manufacturer's expiration date of the purchased concentrated acids.

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5.5 Single Element Stock Standards: Purchased from vendors such as SPEX Certiprep, Inorganic Ventures, Exaxol, High Purity, Ricca, and ERA with concentrations of either 1000 mg/L or 10,000 mg/L. Two identical sets are purchased from two different companies. One set is used to prepare calibration standards and the other set is used to prepare the second source calibration verification standard. The expiration dates are assigned by the manufacturer.

5.6 Tune B Tuning Solution (1 µg/L): This solution is used for tuning the ICP-MS in STD and KED mode as well as for running performance reports. Purchased or prepared from single element stock standards. Dilute appropriate volumes of the single element stock standards to 1 L with 2 % HNO_3 / 0.5 % HCl to produce a solution with 1 µg/L Ba, Bi, Ce, Co, In, Li and U. This solution may be used passed the manufacturer's expiration date as long as the signal produced by the solution is stable.

5.7 iCAP Q/RQ Setup solution: For performing the detector cross-calibration, detector voltage optimization, and mass calibrations. The setup solution can be purchased (e.g. Thermo-5A, Inorganic Ventures) or prepared in the laboratory by diluting appropriate volumes of the single element stock standards to 1 L with 2 % HNO_3 to produce a solution with 3 µg/L of Bi, Ho, Rh, Tb, U, Ce, Cs, In, Ta, and Y; 4 µg/L Ba and Tl; 5 ug/L Sr, 6 µg/L Ag and Mn; 8 µg/L Co, Li, and Sc; 10 µg/L Al, Ga, and Mg; 15 µg/L Cu and Ni; 20 µg/L Zn; 35 µg/L Be. This solution can be used passed the manufacturer's expiration as long as the signal produced by the solution is stable.

5.8 Internal Standard: Dilute appropriate volumes of the single element stock standards to 2 L with 2 % HNO_3 / 0.5 % HCl to produce a solution that contains between 1,000,000 cps and 1,200,000 cps. Each instrument requires different concentrations of single stock element standards to achieve the desired counts.

5.9 Calibration Standards

5.9.1 Calibration Blank (CCB, Rinse): Prepare by diluting 20 mL of HNO_3 and 10 mL of HCl to 1 L. The expiration date of the concentrated acids assigned by the manufacturer shall be used. The solution is also used as a continuous calibration blank every 10 samples. The solution can also be used as a rinse between samples.

5.9.2 ICAP STD 1, CPQLICAP: This standard is prepared at the concentration of the instrument practical quantitation limit (PQL). Dilute appropriate volumes of the single element stock standards to 1 L with 2 % HNO_3 / 1 % HCl to produce a solution at the PQL level. The current instrument PQL concentrations can be found on the Laboratory's computer network, \\FLDEP1\DEAR\Labs\Lab5\Chemistry\Common\MDLs_Control_Limits\Metals_Mercury. Prepare the ICAP STD 1 / CPQLICAP at the instrument PQL concentration. The expiration date for this standard is the same as the single element stock standards from which it was made.

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5.9.3 ICAP STD 2: Dilute appropriate volumes of the single element stock standards to 1 L with 2 % HNO / 1 % HCl to produce a solution with 2 µg/L Tl; 5 µg/L Ag; 10 µg/L Sb, As, Cd, Cr, Co, Cu, Mn, Ni, Se, Zn, Be, B, Pb, Mo, and U; 15 µg/L Ba; 50 µg/L Sr; 75 µg/L Al. The expiration date for this standard is the same as the single element stock standards from which it was made.

5.9.4 ICAP STD 3, ICAPCCV: Dilute appropriate volumes of the single element stock standards to 1 L with 2 % HNO_3 / 1 % HCl to produce a solution with 10 µg/L Tl; 25 µg/L Ag; 50 µg/L As, Cd, Cr, Co, Cu, Mn, Ni, Se, Zn, Sb, Be, B, Pb, Mo, and U; 75 µg/L Ba; 250 µg/L Sr; 375 µg/L Al. The expiration date for this standard is the same as the single element stock standards from which it was made.

5.9.5 ICAP STD 4: Dilute appropriate volumes of the single element stock standards to 1 L with 2 % HNO_3 / 1 % HCl to produce a solution with 20 µg/L Tl; 100 µg/L Ag; 200 µg/L As, Cd, Cr, Co, Cu, Mn, Ni, Se, Zn, Sb, Be, B, Pb, Mo, and U; 300 µg/L Ba; 1000 µg/L Sr; 1500 µg/L Al. The expiration date for this standard is the same as the single element stock standards from which it was made.

5.9.6 ICAP STD 5: Dilute appropriate volumes of the single element stock solutions to 1 L with 2 % HNO_3 / 1 % HCl to produce a solution with 40 ug/L Tl; 200 ug/L Ag; 400 ug/L As, Cd, Cr, Co, Cu, Mn, Ni, Se, Zn, Sb, Be, B, Pb, Mo, and U; 600 ug/L Ba; 2000 ug/L Sr; 3000 ug/L Al. The expiration date for this standard is the same as the single element stock standards from which it was made.

5.10 ICAPMETC: This is the second source standard used to verify the calibration. This standard must be prepared with second source standards (single element stock standards different from the standards used to prepare the calibration standards). Dilute appropriate volumes of the single element stock standards to 1 L with 2 % HNO_3 / 1 % HCl to produce a solution with 10 µg/L of Tl; 100 µg/L Ag, As, Cd, Cr, Co, Cu, Mn, Ni, Se, Zn, Sb, Be, B, Pb, Mo, U, Ba, Sr, and Al. The expiration date for this standard is the same as the single element stock standards from which it was made.

5.11 CPQLICAP#: This is a calibration check standard at the PQL level. It is analyzed after every calibration. A suffix is added to the CPQLICAP abbreviation (e.g. 7, 8, etc.) to indicate the current PQL solution used. Dilute appropriate volumes of the single element stock standards to 1 L with 2 % HNO_3 / 1 % HCl to produce a solution at the PQL level. The current instrument PQL concentrations can be found on the Laboratory's computer network, \\FLDEP1\DEAR\Labs\Lab5\Chemistry\Common\MDLs_Control_Limits\Metals_Mercury. The expiration date for this standard is the same as the single element stock standards from which it was made.

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5.12 DPQLICAP#: This is the PQL check standard for the digested water matrix. It is prepared at 50x the PQL concentration for digested water samples. A suffix is added to the DPQLICP abbreviation (e.g. 7, 8, etc.) to indicate the current PQL solution used. Dilute appropriate volumes of the single element stock standards to 1 L with 2 % HNO_3 / 1 % HCl to produce a solution at 50x the PQL level of digested water samples. The current digested water sample PQL concentrations can be found on the Laboratory's computer network, \\FLDEP1\DEAR\Labs\Lab5\Chemistry\Common\MDLs_Control_Limits\Metals_Mercury. The expiration date for this standard is the same as the single element stock standards from which it was made.

5.13 SDPQLICAP#: This is the PQL check standard for digested sediment, tissue, and waste samples. It is prepared at 250x the PQL concentration for digested sediment, tissue, and waste samples. A suffix is added to the SDPQLICAP abbreviation (e.g. 1, 2, etc.) to indicate the current PQL solution used. Dilute appropriate volumes of the single element stock standards to 1 L with 2 % HNO_3 / 1 % HCl to produce a solution at 250x the PQL level of digested sediment, tissue, and waste samples. The current digested sediment, tissue, and waste PQL concentrations can be found on the Laboratory's computer network, \\FLDEP1\DEAR\Labs\Lab5\Chemistry\Common\MDLs_Control_Limits\Metals_Mercury. The expiration date for this standard is the same as the single element stock standards from which it was made.

5.14 ICAPMSPQL3052#: This is the PQL check standard for totally digested sediment samples digested according to DEP SOP MT-063. It is prepared at 250x the PQL concentration for totally digested sediment samples (Test ID: S-ICPMS-TO). A suffix is added to the ICAPMSPQL3052 abbreviation (e.g. 1, 2, etc.) to indicate the current PQL solution used. Dilute appropriate volumes of the single element stock standards to 1 L with 2 % HNO_3 / 1 % HCl to produce a solution at 250x the PQL for totally digested sediment samples. The current totally digested sediment PQL concentrations can be found on the Laboratory's computer network, \\FLDEP1\DEAR\Labs\Lab5\Chemistry\Common\MDLs_Control_Limits\Metals_Mercury. The expiration date for this standard is the same as the single element stock standards from which it was made.

5.15 ICAPMSPQLSPLP#: This is the PQL check standard for digested sample extracts produced from the SPLP procedure, DEP SOP MT-068. It is prepared at 50x the PQL for digested SPLP extracts. A suffix is added to the ICAPMSPQLSPLP abbreviation (e.g. 1, 2, etc.) to indicate the current PQL solution used. Dilute appropriate volumes of the single element stock standards to 1 L with 2 % HNO_3 / 1 % HCl to produce a solution at 50x the PQL level of digested SPLP extracts. The current digested SPLP extract PQL concentrations can be found on the Laboratory's computer network, \\FLDEP1\DEAR\Labs\Lab5\Chemistry\Common\MDLs_Control_Limits\Metals_Mercury. The expiration date for this standard is the same as the single element stock standards from which it was made.

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5.16 ICAPMSPQLTCLP#: This is the PQL check standard for digested sample extracts produced from the TCLP procedure, DEP SOP MT-068. It is prepared at 50x the PQL for digested TCLP extracts. A suffix is added to the ICAPMSPQLTCLP abbreviation (e.g. 1, 2, etc.) to indicate the current PQL solution used. Dilute appropriate volumes of the single element stock solutions to 1 L with 2 % HNO_3 / 1 % HCl to produce a solution at 50x the PQL level of digested TCLP extracts. The current digested TCLP extract PQL concentrations can be found on the Laboratory's computer network, \\FLDEP1\DEAR\Labs\Lab5\Chemistry\Common\MDLs_Control_Limits\Metals_Mercury. The expiration date for this standard is the same as the single element stock standards from which it was made.

5.17 Interference Check Solution (IC6020C): This interference check solution is prepared to contain known concentrations of interfering elements that will demonstrate the magnitude of interferences and provide an adequate test of interference correction. Chloride in the solution provides a means to evaluate collision cell removal of chloride-related interferences such as $^{40}Ar^{35}Cl^{+}$ on $^{75}As^{+}$. Iron is used to demonstrate adequate resolution of the spectrometer for the determination of manganese. Molybdenum serves to indicate oxide effects on cadmium isotopes. The other components are present to evaluate the ability of the measurement system to remove various molecular-ion isobaric interferences. The IC6020C solution must be analyzed as a calibration check prior to analyzing samples and every 12 hours during the analysis. This standard is typically prepared from two intermediate standards.

5.17.1 ICS Solution A: The interferences intermediate solution, which includes Mo and Ti at 20 mg/L; Al, Mg, P, K, and S at 1000 mg/L; C at 2000 mg/L; Fe and Na at 2500 mg/L; Ca at 3000 mg/L; and Cl at 21215 mg/L, can be purchased from SPEX Certiprep or High Purity (concentrations may vary). The interferences intermediate can also be prepared in the laboratory from single element stock standards and various concentrated acids. The expiration date is assigned by the manufacturer (if prepared, the expiration date of the single element stock standards/acids shall be used).

5.17.2 The trace elements in the IC6020C solution include As, Cd, Cr, Co, Cu, Ni, Se, Zn, and Ag at 20 µg/L; Gd at 50 ug/L; and Mn at 100 µg/L. Prepare the intermediate solution by diluting appropriate volumes of the single element stock standards with 2 % HNO_3 / 1 % HCl to produce a solution with 50 mg/L of As, Cd, Cr, Co, Cu, Ni, Se, Zn, and Ag; 250 mg/L Mn; 150 mg/L Gd. The expiration dates of the single element stock standards shall be used.

5.17.3 The two intermediates described in 5.14.1 and 5.14.2 are combined to prepare the IC6020C standard. Prepare the working standard by diluting appropriate volumes of the intermediate standards with 2 % HNO_3 / 1 % HCl to produce a solution with 2120 mg/L Cl; 300 mg/L Ca; 250 mg/L Na and Fe; 200 mg/L C; 100 mg/L Al, Mg, P, K, and S; 2 mg/L Mo and Ti;

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100 µg/L Mn; 50 ug/L Gd; 20 µg/L As, Cd, Cr, Co, Cu, Ni, Se, Ag, and Zn. Concentrations of interferences may vary by vendor.

5.18 High Conductivity Standard Reference Material (SRM): A high conductivity (> 10,000 umhos/cm) SRM is purchased to assess the instrument's accuracy in a high conductivity sample matrix. The SRM may require spiking to quantitate metals above the digested water sample PQL. Currently, NASS-7, purchased from the National Research Institute of Canada, is used as the high conductivity SRM.

5.18.1 NASS-7 Spike: Prepare a spiking solution by diluting appropriate volumes of the single element stock standards with a 2 % HNO_3 / 1 % HCl to produce a solution with 5 mg/L Cd, Co, Cu, Mn, Mo, Ni, Pb, U, As, and Cr; 10 mg/L Zn. The expirations dates of the single element stock standards shall be used.

5.18.2 NASS-7-SPK3: This is the working standard analyzed on the instrument to assess the instrument's accuracy in a high conductivity matrix. Prepare the NASS-7-SPK3 by spiking 0.1 mL of the NASS-7 Spike to 10 mL of the NASS-7 SRM. Analyze the NASS-7-SPK3 with a dilution of 5 and 10.

6. SAMPLE COLLECTION, PRESERVATION, SHIPMENT, AND STORAGE

6.1 All samples are stored in the DEP Central Laboratory's Receiving Room. The sample pH for water matrices is checked upon arrival at the laboratory by receiving room staff to ensure the pH is less than 2. If a sample is received with pH > 2, it is acidified by the Receiving Room staff to a pH below 2 and preparation / analysis cannot be started for at least 24 hours. The pH is checked again with pH paper by Metals staff (prior to dispensing any sample for digestion or analysis) to verify the pH is below 2 and documented (lot number and expiration date) in the comments section of the prep worksheet in the LIMS Prep Worksheet Manager or Excel. Samples consisting of water matrices are stored at room temperature and samples consisting of solid matrices are stored in one of the receiving room refrigerators as many of these samples also require organic analyses, which require refrigeration.

7. SAMPLE PREPARATION PROCEDURE

Note: Silver is only slightly soluble in the presence of chloride unless there is a sufficient chloride concentration to form the soluble chloride complex. Therefore, low recoveries of silver may occur in samples analyzed by direct analysis. The total recoverable sample digestion procedure, DEP SOP MT-024, is suitable for the determination of silver in aqueous solutions up to 100 µg/L. For samples with concentrations higher than 100 µg/L, the sample should be re-digested using a sample volume that will result in a silver concentration in the digestate of less than 100 µg/L.

Solid or waste samples prepared according to DEP SOP MT-060 that contain silver concentrations greater than 500 µg/L should be treated similarly.

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7.1 Filtered water samples (Test ID: W-ICPMS-F): For undigested, filtered and acidified samples, which are checked out directly from the custody room, prepare matrix spikes and laboratory fortified blanks (LFBs) by pipetting 50 µL of the water spike HW6A and 50 µL of the water spike HW6B into a 15 mL centrifuge tube containing 10 mL of either sample (matrix spike) or the blank solution of 2 % HNO_3 / 1 % HCl (LFB). Mix thoroughly. Each batch of up to 20 samples must contain at least one matrix spike for every 10 samples (two different samples are spiked for batches that contain more than 10 samples) and one set of matrix spike duplicates. One LFB is required for each batch. A PQL check is also prepared for each batch of filtered water samples by pouring the instrument CPQLICP# solution into an analysis tube. See DEP SOP MT-061 for HW6A and HW6B preparation details.

7.2 Unfiltered, digested water samples: See DEP SOP MT-024 for preparation details.

Note: Where EPA Method 200.8 Rev. 5.4 is approved for the determination of metals in drinking water, samples may be analyzed directly without acid digestion if the samples have been properly preserved with acid and have turbidity of < 1 NTU at the time of analysis. This includes aluminum, antimony, arsenic, barium, beryllium, cadmium, chromium, copper, lead, manganese, nickel, selenium, thallium, and zinc.

7.3 Solid Samples (soils, sediments, and wastes): See DEP SOPs MT-060 for details of total recoverable metals preparation and DEP SOP MT-063 for details of total metals determination preparation. All samples and batch QC are diluted by a factor of 5.

7.4 TCLP and SPLP samples: See DEP SOP MT-068 for TCLP extractions and SPLP extractions. TCLP samples are diluted by 10 during preparation. The dilution is noted on the MT-TCLPSPLP-DIG form and must be entered into QC Manager. SPLP samples are not diluted during preparation. The acid matrix of TCLP and SPLP sample digestates does not need dilution prior to analysis, however, the SEDSPK4 concentration, especially when doubled-spiked, will yield trace metal concentrations above the calibration. As such, the QC sample and the spikes are run as D5, with an additional measurement of the neat QC sample. What's more, the samples may contain high concentrations of metals. Depending on the total recoverable results, if available, screening the samples with a dilution of 1000 can prevent instrument contamination.

8. SAMPLE ANALYSIS / QUANTITATION

Note: Read the iCAP Q / RQ Operating Manual and the iCAP Q / RQ Software Manual to assist in the operation of the ICP-MS.

The Instrument Control application is the software used to ignite the plasma, tune the instrument, acquire performance reports, and extinguish the plasma manually. The Qtegra application provides the interface for instrument calibration and the analysis of unknown samples.

8.1 Instrument Startup: The following steps should be followed if the main power switch is off and the vacuum is vented. This will only occur after major

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instrument maintenance, multi-day office closures, or other non-routine occurrences. Typically, the instrument is left in standby mode.

8.1.1 Switch the main power switch to position 'On'.

8.1.2 Start the vacuum system in the Instrument Control window.

8.1.2.1 Click 'On' in the vacuum submenu.

8.1.2.2 Wait for the turbomolecular pump to reach at least a frequency of 800 Hz.

8.1.3 Check the vacuum. The vacuum pressure is correct when the vacuum LED is green.

8.1.4 If the vacuum was vented, the detector should not be calibrated regardless of whether it was changed or not.

8.2 Lighting the Plasma

8.2.1 Make sure the peristaltic pump tubing is replaced daily, not clogged, and the collars are secured on the brackets.

8.2.2 Make sure the oil level in the forepump is at the appropriate level.

8.2.3 Make sure there is an adequate supply of the Rinse solution in the carboy.

8.2.4 Make sure the waste container is not full. The acidic waste can be poured down the sink drain while flushing with tap water.

8.2.5 Check the chiller to make sure the temperature is 21 °C ± 2 °C. Make sure the water level in the chiller is adequate. Refill with distilled water.

8.2.6 Check the argon pressure on the regulator on the wall behind the instrument. The pressure should be between 80 – 90 psi. However, Thermo service technicians have found better instrument performance if the delivery pressure is 90 – 100 psi.

8.2.7 In the Instrument Control Window, click 'On' in the iCAP Q / RQ ribbon. The start-up procedure is triggered and the plasma is lit.

Note: Observe the plasma ignition through the plasma box window to ensure it starts up properly. The ignition process can be stopped by attempting to open the torch box door. The door will not open, but the door interlock will trigger, immediately stopping the ignition process. The most common problem is the generation of 'hot' i.e. orange plasma after running high conductivity or high concentration samples. Abort the ignition process if orange plasma or plasma arcing is observed, the plasma appears dim, or if strange noises are observed such as crackling or hissing. Inspect the torch assembly. If any glassware appears to be melted or cracked, replace the defective glassware and report the replacement in the maintenance logbook. Arcing indicates a dirty torch or cones. Clean the torch and cones according to section 13, Maintenance. Crackling or hissing indicates an argon leak.

Also monitor the peristaltic pump during plasma ignition. Make sure the pump is not stalling which could indicate a weakening motor or the pump tubing is too tight. If the pump stalls, the

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self-aspirating nebulizer (4.3) will deliver solution, while waste from the spray chamber is not pumped out which will flood the injector and torch assembly.

8.2.8 Allow at least 30 minutes for the temperature to equilibrate in the torch box.

8.3 Tuning the ICP-MS

Note: It may not be necessary to tune the instrument every day. If performance report criteria (8.4) are met without tuning, the analyst may proceed to calibration and sample analysis. When selecting an existing Autotune, ensure the Autotune is performed with the peristaltic pump speed at 20 rpm.

8.3.1 Select the STD measurement mode from the drop-down box in the Measurement Mode ribbon tab. The settings last saved for this measurement mode are loaded into Instrument Control.

8.3.2 Place the sample probe and the internal standard probe into the Tune B solution.

8.3.3 In the Instrument Control window, select the iCAP Q / RQ tab. In the Wizard group, click ‘Autotune’ and then click ‘Source Autotune’. Click next to initiate the autotune sequence.

8.3.4 When an autotune is successful and instrument parameters are acceptable to the analyst, click ‘Yes’ to save the results. Select ‘Open Report’ to view the report.

8.3.5 Select the KED measurement mode from the drop-down box in the Measurement Mode ribbon tab. The settings last saved for this measurement mode are loaded into Instrument Control.

8.3.6 Make sure the sample and internal standard probes are in the Tune B solution.

8.3.7 In the Instrument Control window, select the iCAP Q / RQ tab. In the Wizard group, click ‘Autotune’ and then click ‘!CaliTune KEDS Line1’. Click next to initiate the autotune sequence.

8.3.8 When finished, click ‘Yes’ to save the results. Select ‘Open Report’ to view the report.

Note: Thermo service technicians have suggested running two tunes when the detector is nearing the end of its life and cps are low and not sufficient for a passing performance report. Run tune ‘CaliTune STDS’ followed by ‘CaliTune STD-100V’. Technicians have also suggested that the pirani pressure be monitored daily. This can be used to determine if there is a loose fitting in the cones or if a cone orifice is degraded beyond use.

8.4 Performance Report

Note: A passing performance report is required before calibrating the instrument and analyzing samples. To determine if the performance report passes, verify the sensitivity checks, the stability checks, and the mass calibration passes for all masses.

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8.4.1 Select the STD measurement mode from the drop-down box in the Measurement Mode ribbon tab. The settings last saved for this measurement mode are loaded into Instrument Control.

8.4.2 Place the sample probe and the internal standard probe into the Tune B solution.

8.4.3 In the Instrument Control window, select the iCAP Q / RQ tab. In the wizard group, click 'Performance Report', run an existing performance report, STD.

8.4.4 When the acquisition is completed, click 'Next', then select the check box 'Open Report' to view the results. Click 'Finish' to complete the performance report wizard. See section 9 for saving performance reports to the Laboratory computer network.

8.4.5 Select the KED measurement mode from the drop-down box in the Measurement Mode ribbon tab. The settings last saved for this measurement mode are loaded into Instrument Control.

8.4.6 Make sure the sample and internal standard probes are in the Tune B solution.

8.4.7 In the Instrument Control window, select the iCAP Q / RQ tab. In the wizard group, click 'Performance Report' and then click 'KED'. Click run an existing performance report, KED with double Ba 0.05 and click 'Next' to initiate the performance report sequence.

8.4.8 When the acquisition is completed, click 'Next', then select the check box 'Open Report' to view the results. Click 'Finish' to complete the performance report wizard. See section 9 for saving performance reports to the Laboratory computer network.

Note: If the background 4.5 fails in the STD or KED performance report, access the factory settings and ensure the CCT RF Amplitude Offset is set to a negative voltage in both STD and KED modes. The voltage should be between -230 to -250.

8.5 Detector Setup

Note: The detector setup should only be performed when the instrument sensitivity is starting to decline. The procedure is performed on average once a month and typically, the automated procedure will increase the voltage applied to the pulse section of the detector so that it just lies on the plateau of the detector gain curve. The voltage of the analog section of the detector is normally also increased to ensure the cross calibration is accurate and maintained at a defined level.

Note: If the detector appears to be nearing the end of its life, see section 8.3 to extend the life of the detector while a new one is shipping.

8.5.1 To perform a detector setup and cross calibration with the Detector Setup Wizard, open the Instrument Control window.

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8.5.2 Select the STD measurement mode from the drop-down box in the Measurement Mode ribbon tab. The settings last saved for this measurement mode are loaded into Instrument Control.

8.5.3 In the Instrument Control window, select the iCAP Q / RQ tab. In the wizard group, click 'Detector Setup'. Select 'Detector HV Setup and Cross Calibration'. Make sure the sample and internal standard probes are in the iCAP Q / RQ Setup solution and click next to initiate the sequence.

8.5.4 Click 'Finish' to save the detector voltages as well as the cross calibration factors and to exit the Detector Setup wizard.

8.6 Mass Calibration

Note: It is necessary to perform a mass calibration whenever the peak width determination specified in the performance report fails or the mass peaks are not aligned correctly. The mass calibration should be performed monthly. The software uses a mass calibration equation so that when a mass is selected for measurement, the control electronics can set the quadrupole to transmit that mass.

8.6.1 To perform a mass calibration with the wizard, open the Instrument Control window.

8.6.2 Select the STD measurement mode from the drop-down box in the Measurement Mode ribbon tab. The settings last saved for this measurement mode are loaded into Instrument Control.

8.6.3 In the Instrument Control window, select the iCAP Q / RQ tab. In the wizard group, click 'Mass Calibration'. The checkbox for 'Execute a Coarse Mass Calibration first' only needs to be checked if the mass calibration is expected to be significantly different, for example, due to new hardware, or if a mass calibration without this option has failed.

8.6.3.1 If 'Load Tune Settings' is checked, also select a tune setting from the drop-down list, otherwise, the tune settings currently loaded will be used.

8.6.4 Make sure the sample and internal standard probes are in the iCAP Q / RQ Setup solution and click next to initiate the sequence.

8.6.5 When the wizard finishes the mass calibration, the result window opens automatically. Click 'Next' and then 'Finish' to save the new mass calibration.

8.7 Calibration and Sample Analysis

8.7.1 Qtegra is the software used to analyze samples. Within Qtegra, the 'Homepage' is the default start page.

8.7.2 The Homepage gives access to all existing templates, allows for the generation of new LabBooks, and provides an overview of current system conditions (dashboard).

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8.7.3 LabBooks created from a template inherit the method parameters from that template.

8.7.4 To create a new LabBook from a template, click on ‘LabBook’ on the left. In the LabBook menu, set the radio button to ‘create a new LabBook’. Select the appropriate template from the drop-down menu, enter the correct file name (e.g. 20190219-03168R and 20190219A-03168R) and enter the number samples. Then click ‘Create LabBook’.

8.7.4.1 iCap Q analysis template name:

TEST STD & KED DEP Template no Gd STDs.

8.7.4.2 iCap RQ analysis template name:

8.7.4.2.1 As only: STD & KED Template- High Matrix DOH

8.7.4.2.2 Full list: STD & KED Template- High Matrix – 5 point cal

8.7.5 Type samples to be analyzed into the Sample List. The Sample List should start with a few rinses, followed by the calibration blank, calibration standards, calibration check standards, and then the samples to be analyzed. A CCV and CCB are analyzed every 10 samples. An example Sample List is shown below:

Rinse
Rinse
CCB
Rinse
Calibration Blank
CPQLiCAP1
ICAP STD 2
ICAP STD 3
ICAP STD 4
ICAP STD 5
CCB
Rinse
CPQLiCAP1
ICAPMETC
IC6020C
Rinse
ICAPCCV
CCB

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10 Samples from 1st batch

ICAPCCV

Rinse

CCB

8.7.6 Once the Sample List has been typed in and the samples poured, initiate the experiment by clicking 'Run' in the menu of the LabBook. Make sure the internal standard probe is in the bottle of internal standard.

8.7.7 Low conductivity samples (up to 10,000 umhos/cm): Sample dilution not necessary for sample matrix.

8.7.8 High conductivity samples (> 10,000 umhos/cm): Analyze samples with a dilution of 4 and 10. When high conductivity samples are analyzed, a NASS-7 SRM must also be analyzed. The NASS-7 SRM must be analyzed with both a dilution of 5 and 10.

8.8 Turning off the instrument

8.8.1 Standby Mode: After the experiments are finished and the system was flushed with Rinse, the instrument is set to standby mode. This applies when a LabBook finishes during normal working hours and a second LabBook will be run or retuning required.

8.8.2 Auto Plasma shutdown after completion of a scheduled LabBook (Qtegra): After the scheduled LabBooks are finished and the system was flushed with rinse, the instrument is set to '*close down on empty queue or LabBook error'*. This applies when a LabBook completes and the instrument needs to shut down automatically on schedule. See Fig. 1.

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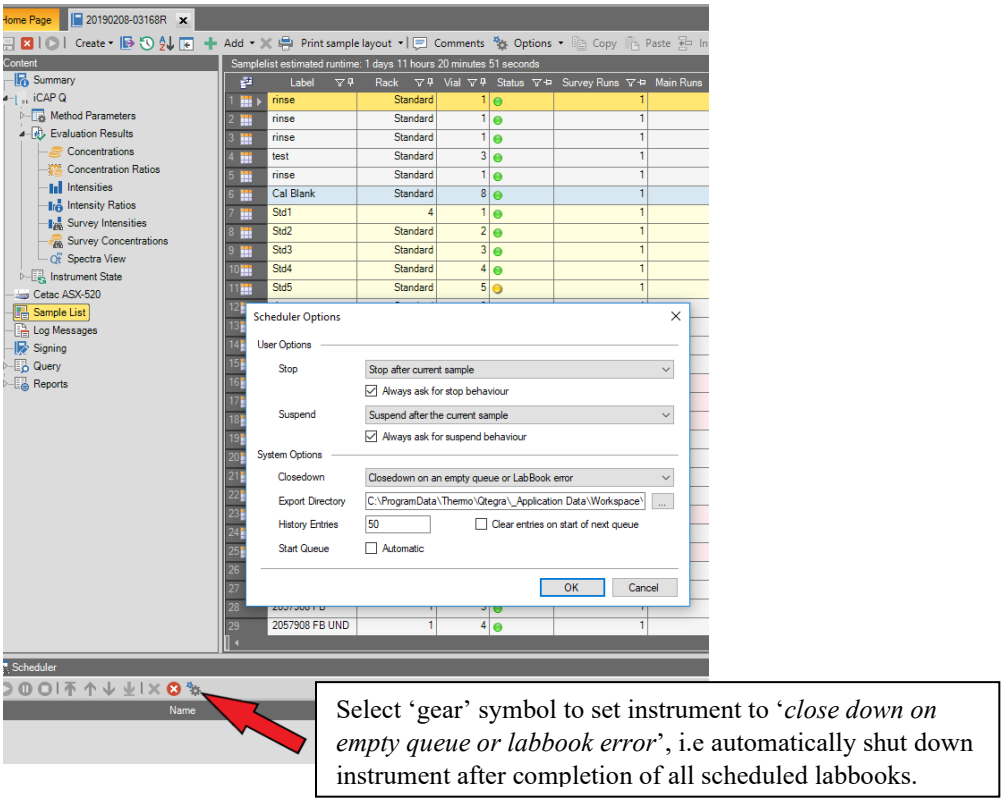


Figure 1. The scheduler in the bottom panel of Qtegra is used to set the Instrument to automatically shut off after the completion of all scheduled LabBooks.

8.8.3 Turn off instrument manually (Instrument Control): To turn off the instrument manually, set the instrument to STD mode in the Instrument Control Window and rinse for 10 minutes in 2% HNO_3 / 1% HCl. When the background levels are acceptable click 'Off' to turn the plasma off.

8.8.4 Complete System Shutdown: To completely shut down the system, in the Instrument Control Window, click 'Off' to turn the plasma off. Switch off the vacuum in the Vacuum submenu. Switch the main power switch to position Off. **This only applies when a larger down time is planned (office closure during hurricane season, planned A/C shutdown for repairs/maintenance).**

9. DATA ARCHIVAL

9.1 A total of eight files are stored on the network for each analysis. All files are saved to the folder \\FLDEP1\DEAR\Labs\Lab3\Chem_Data\Inorganic\Metals\ICP-MS-iCAP or \\FLDEP1\DEAR\Labs\Lab3\Chem_Data\Inorganic\Metals\ICP-MS-iCAP-RQ.

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9.1.1 Performance Report for STD mode. Exported from Qtegra and saved on the computer network as a .pdf file.

9.1.2 Performance Report for KED mode. Exported from Qtegra and saved on the computer network as a .pdf file.

9.1.3 Calibration Report. Exported from Qtegra and saved on the computer network as a .pdf file.

9.1.4 Intensity Report. Exported from Qtegra and saved as a pdf.

9.1.5 Cover Page for laboratory report. Created in Microsoft Excel and saved as a .xls file. Printed and included with each laboratory report.

9.1.6 Solutions Log. Created in Microsoft Excel and saved as a .xls file. Printed and included with each laboratory report.

9.1.7 Mass Corrected concentration data. Comma separated values file exported from Qtegra and converted into the LIMS QC Manager module.

9.1.7.1 To export the concentration data of a currently opened LabBook, in the toolbar of the LabBook, click 'Export'. Select the exporter as 'CSV Export'. Check that the column separator is set to Custom with ',' selected and that the Decimal Symbol is set to Point. The checkboxes for 'Export sample lines are rows', 'Group exported blocks', and 'Export complete LabBook' are to be checked.

9.1.7.2 Select from the Schemes drop down the option of 'Concentration Export' that has concentration average and Start Time selected. In the Excel Export Options, select a path and enter the file name. For the ICP-MS iCap Q, the path should be \\FLDEP1\DEAR\Labs\Lab3\Chem_Data\Inorganic\Metals\ICP-MS-iCAP and the file with the format 'yyyymmdd-03168R Intensities'.

For the ICP-MS iCAP RQ, the path should be \\FLDEP1\DEAR\Labs\Lab3\Chem_Data\Inorganic\Metals\ICP-MS-iCAP-RQ and the file with the format 'yyyymmdd-00090 Intensities'.

9.1.8 Mass uncorrected intensity as counts per second. Exported from Qtegra as a .csv file.

9.1.8.1 To export the counts per second intensity data of a currently opened LabBook, in the toolbar of the LabBook, click 'Export'. Select the exporter as 'CSV Export'. Check that the column separator is set to Custom with ',' selected and that the Decimal Symbol is set to Point. The checkboxes for 'Export sample lines as rows', 'Group exported blocks', and 'Export complete LabBook' are to be checked.

9.1.8.2 Select from the Schemes drop down the option of 'Intensity' that has intensity average, Intensity per Run, and Start Time selected. In the Excel Export Options, select a path and enter the file name.

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For the ICP-MS iCap Q, the path should be \\FLDEP1\DEAR\Labs\Lab3\Chem_Data\Inorganic\ICP-MS-iCAP and the file with the format 'yyyymmdd-03168R Intensities'.

For the ICP-MS iCap-RQ, the path should be \\FLDEP1\DEAR\Labs\Lab3\Chem_Data\Inorganic\Metals\ICP-MS-iCap-RQ and the file with the format 'yyyymmdd-00090 Intensities'.

10. QUALITY CONTROL

Note: The calibration range for each analyte is demonstrated to be within the linear operating range of the instrument at the beginning of each analytical run. The instrument is calibrated with 5 standards of varying concentration and a blank and the calibration is forced through the blank.

The correlation coefficient for the first order linear plot of the calibration curve must be 0.998 or greater. Additionally, the % relative error for calibration standard 1 must be within 30% of the expected recovery and the % relative error for calibration standard 4 must be within 10% of the expected recovery.

$$\% \text{ Relative Error} = [(x'_i - x_i) / x_i)] \times 100$$

x_i = True value for the calibration standard

x'_i = Measured concentration of the calibration standard

The validity of the curve is then further tested with a solution (ICAPMETC) prepared from standards purchased from a vendor that is different from the one used to prepare the calibration standards. The ICAPMETC standard verifies the calibration curve, serving as the initial calibration verification and instrument performance. The analytes present in the second source standard, ICAPMETC, are the same as those the instrument was calibrated for and at a concentration of 100 µg/L (Tl 10 µg/L). If the determined concentration for any analyte in the ICAPMETC standard is less than 90 % or greater than 110 % of the stated value then the data for that analyte are considered invalid and cannot be used, unless appropriately qualified. Data are not reported for samples with concentrations of analytes outside of the established calibration range without qualification. The calibration range may be extended by dilution.

10.1 The procedure for data review is summarized in DEP SOP MT-058, which also lists the acceptance criteria for standard reference materials, matrix spikes, internal standards, and precision data. Contingencies for out-of-control QC data are listed in DEP SOP LB-027 as well as DEP SOP MT-058.

10.2 A minimum of three replicate integrations are required for data acquisition, routinely 6 replicate main runs are performed. The average of the integrations must be used for instrument calibration and data reporting.

10.3 Interference Check Solution (IC6020C): The acceptable recovery limits are 85 – 115 %. If the analyte recoveries in the initial IC6020C analyzed after calibration and prior to samples are outside of the acceptable recovery limits, no data may be

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reported without appropriate qualifications, except for when the IC6020C recovery is > 115 %, non-detect sample results may be reported. During an analytical run, IC6020C is analyzed every 12 hours. If any recoveries are outside of acceptable limits, samples analyzed after the IC6020C shall be re-analyzed or qualified accordingly.

10.4 Internal Standard Limits: The internal standard limits for EPA method 200.8 are 60 – 125 %. Data reported by EPA Method 200.8 includes test IDs W-ICPMS and W-ICPMS-F. The internal standard limit for EPA Method 6020A is 70 – 130 %. Data reported by EPA Method 6020A includes test IDs T-ICPMS, S-ICPMS, S-ICPMS-TO, SPLP-ICPMS, TCLP-ICPMS, WASTEICPMS, W-ICPMS-R, and W-ICPMSR-F. These limits do not apply to samples of known concentration that are not reported, e.g. CCVs, CCBs and the IC6020 solution.

10.5 Calibration Blank: Familiarity with appropriate analyte background levels (e.g. Be, Al, As, Cr, Ni, Cu, Zn, Tl, Pb) is essential in both STD and KED mode to determine if front-end glassware, nebulizer, or cones require cleaning. The most common failure is with aluminum. Peristaltic pump tubing may be rinsed with CCB in advance to remedy high background counts. Monitor the aluminum cps in each beginning rinse and ensure a stable cps between analyses. If 3 rinses are analyzed and the aluminum cps are not constant, insert another rinse. This is done to prevent negative concentrations in the CCBs, which can affect the data.

11. SAFETY / HAZARDOUS WASTE MANAGEMENT

11.1 Review the Health and Safety Plan located on the Laboratory's intranet.

11.2 Use caution with strong irritants such as acids and bases. Avoid breathing the fumes of these irritants by using them in a hood when possible and keeping the face away from open containers of these. Avoid contact of these irritants with skin and clothing by appropriate use of gloves, apron, facemask, safety glasses, hood shield, etc.

11.3 Store any waste that cannot be disposed of down the sink drain (see Laboratory Health and Safety Plan) in the appropriate plastic waste container properly labeled with hazardous waste labels. These wastes include used vacuum pump oil and aqueous solutions known to contain metals at concentrations that exceed the sewer discharge limits. Empty full containers of waste into the waste barrels located in the waste storage room on the loading dock. The limits for discharging metals to the City of Tallahassee sewer system are: As < 132 µg/L, Cd < 79 µg/L, Cr < 2898 µg/L, Cu < 852 µg/L, Pb < 540 µg/L, Mo < 105 µg/L, Ni < 1428 µg/L, Se < 353 µg/L, Ag 3190 µg/L, Zn < 2730 µg/L.

12. TROUBLESHOOTING

Note: See chapter 7 of the iCAP Q / RQ Operating Manual for additional details.

12.1 If a problem occurs, first examine the recent history of use of the instrument. Consider the simple causes first (for example, sample introduction failure is more

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likely than quadrupole failure). The most useful tool for probing analytical problems is the performance report.

12.2 Problem: Plasma does not ignite / plasma goes out unexpectedly. Make sure all components of sample introduction are installed correctly. Ensure the nebulizer gas line is not leaking and there is a tight fit. Ensure that there is enough cool gas flow. This can be done by comparing the setting used in the last successful analysis. Insufficient cool gas flow can cause the plasma to go out unexpectedly. Take apart the sample introduction and make sure it is not flooded, it can be dried with air for the wall. The elbow and torch should be dry, large amounts of liquid will stop the plasma from igniting or cause it to flicker.

12.3 Problem: Sensitivity problems / no signal. Make sure all components of sample introduction are installed correctly. Make sure all cables between instrument and computer are securely connected. Make sure the peristaltic pump is delivering sample. Ensure the cones are installed properly and are secured tightly. If a very odd signal is seen, such as Tune B cps in excess of 17,000,000 cps, check the skimmer cone insert. It is possible the insert fell out of its housing. If the counts are still low and standard autotuning is not helping, a cali tunes STDS can help the sensitivity, this is recommended to be followed by a Cali tunes – 100 V autotune.

12.4 Problem: Signal drifts with time. The usual cause of signal drift is gradual deposition of undissociated sample matrix on the sample and skimmer cones. Make sure cones are conditioned (see iCAP Q Operating Manual, p. 8-19 and iCAP-RQ Operating Manual, p. 8-17 for cone conditioning procedure). Make sure the sample flow rate is consistent and continuous and is being drained appropriately from the spray chamber. Make sure the chiller and room temperature are stable.

12.5 Problem: Poor signal stability. Examine sample introduction system and make sure all components are installed correctly. Check the nebulizer pressure in Qtegra. Ensure the flow rate from the peristaltic pump is stable. This includes ensuring both the internal standard line and the sample line have a stable flow.

12.6 Problem: High background 4.5. Adjusting the extraction lenses and nebulizer gas can help. After troubleshooting, select iCAP Q factory from experiment configuration, select the mass calibration tab, adjust the RF Amplitude Offset to -230 to -250. If this still does not resolve the problem and all troubleshooting pathways have been used, run a full detector set up. This should only be performed as a last resort solution as this adjusts the voltage in the detector. Increasing the voltage increases noise.

12.7 Problem: Stalling peristaltic pump, ‘grinding’ sound from the peristaltic pump during rabitting. Ensure the pump tubing is not too tight. This is the most common cause of a stalling pump. When the pump tubing is changed, the tightness of the clamp on the tubing should be reevaluated. After the 30-minute warm up, reevaluate the tightness of the clamp once again, as the tubes can stretch and

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require less tension. Contact service technicians to arrange for a replacement peristaltic pump.

12.8 Problem: Pirani pressure is too high; plasma turns off automatically. This indicates that the vacuum created behind the sample cones is not at the desired pressure. The pressure should be below 2.0 X 10^0 when the plasma is on. This is typically a gasket placement issue where the fitting is not creating a perfect seal. Remove the cones and gasket, place a new gasket in ensuring a tight seal. Always watch the vacuum pressures to ensure the instrument is running efficiently.

12.9 Problem: Cross Calibration failure. If running a cross calibration fails, the reason most likely is the intensities of the counts was too low. Before running a cross calibration, make sure the counts in either the set-up solution or Tune B solution are at the appropriate intensities.

13. MAINTENANCE

Note: The maintenance procedures do not require removing the instrument housing. Always wear clean, talc-free, and lint-free gloves when handling components of the iCAP ICP-MS. Always place components on clean, lint-free surfaces.

13.1 Peristaltic pump tubing: Check daily for clogging or damage. Tubing should be replaced after rotation to ensure acceptable flow and signal stabilization.

13.2 Nebulizer: Clean when necessary. Replace when necessary. See iCAP Q Operating Manual, page 8-13, for the nebulizer cleaning procedure. See iCAP RQ Operating Manual, page 8-12, for the nebulizer cleaning procedure.

13.3 Spray Chamber: Clean when necessary. Replace when necessary. See iCAP Q Operating Manual, page 8-15, for the spray chamber cleaning procedure. See iCAP RQ Operating Manual, page 8-14, for the spray chamber cleaning procedure.

13.4 Injector: Clean when necessary. Replace when necessary. See iCAP Q Operating Manual, page 8-16, for the injector cleaning procedure. See iCAP RQ Operating Manual, page 8-15, for the injector cleaning procedure.

13.5 Torch: Clean when necessary. Replace when necessary. See iCAP Q/RQ Operating Manual, page 8-16, for the torch cleaning procedure.

13.6 Cones: Clean the cones when build-up of matrix has visibly reduced the orifice size, sensitivity of the instrument is below performance specifications, a detector set-up is not increasing the sensitivity, and cleaning the extraction lens did not increase sensitivity. Cones can be cleaned quickly with canned air. Replace when necessary. See iCAP Q Operating Manual, page 8-22 & 8-23, for the cone cleaning procedure. See iCAP Q/RQ Operating Manual, page 8-20, for the cone cleaning procedure.

13.7 Extraction Lens: Clean the extraction lens when the sensitivity of the instrument is below specification and a detector set-up is not increasing the sensitivity.

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13.8 Detector: The detector has reached the end of its life when it cannot be calibrated anymore. See page 8-29 of the iCAP Q Operating Manual for replacement procedure. See page 8-27 of the iCAP RQ Operating Manual for replacement procedure.

13.9 Gas supply: Leak check the gas lines annually. See page 8-9 of the iCAP Q/RQ Operating Manual for the leak check procedure.

13.10 Air filter: The instrument air filter is located on the left side, bottom. Check and clean monthly.

13.11 Vacuum pump: See the Leybold Vacuum Operating Instructions for further detail.

- Check oil level daily. While the pump is in operation, the oil level should be close to the MAX mark.
- Check gas ballast valve monthly.
- Replace exhaust filter annually.
- Check anti-suck back valve annually.
- Clean fan cover annually.
- Replace PFPE (perfluoropolyether) oil annually.

13.12 Chiller: See the Thermo Scientific ThermoFlex SID Recirculating Chiller Installation, Operation, and Basic Service Manual.

- Clean fluid bag filter weekly by rinsing in DI water.
- Condenser filter: Check weekly and clean at least once per month.
- Clean reservoir as needed.
- 1.5 gallons of distilled water should be used, in lieu of distilled water, DI water that is pH adjusted can be used.

Note: Any part replacement should be logged in the maintenance log book along with the reason of the replacement.

14. REFERENCES

14.1 Thermo Fisher Scientific iCAP

14.1.1 Q Operating Manual, Revision C – 1288090.

14.1.2 RQ Operating Manual, Revision B – BRE0003606.

14.2 Thermo Fisher Scientific iCAP

14.2.1 Q Software Manual, Revision B – 1288010.

14.2.2 RQ Software Manual, Revision B – BRE0005922.

14.3 EPA Methods 200.8 Revision 5.4 and 6020A.

14.4 DEP SOP LB-027, Standard Operating Procedure for Reporting Qualified Data and Correcting Quality Control Problems.

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14.5 DEP SOP MT-007, Procedure for Cleaning Metals Labware.

14.6 DEP SOP MT-024, Preparation of Water Samples for Metals Analysis by ICP-AES and ICP-MS.

14.7 DEP SOP MT-058, Procedure for Metals Data Review and Authorization.

14.8 DEP SOP MT-060, Hot Block Digestion of Soil, Sediment, Waste, and Tissue Sample for Total Recoverable Metals.

14.9 DEP SOP MT-061, Analysis of Selected Metals by Dual View Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP-AES).

14.10 DEP SOP MT-063, Microwave Assisted Hydrofluoric Acid Digestion of Solid Matrices for Total Metals Determination (Adapted from EPA Method 3052 for the Milestone Ethos EZ microwave system).

14.11 DEP SOP MT-068, Toxicity Characteristic Leaching Procedure (EPA Method 1311) and the Synthetic Precipitation Leaching Procedure (EPA Method 1312).

14.12 DEP SOP MT-069, Metals Standard and Reagent Preparation Procedure.

14.13 Laboratory Quality Manual, DEP.

14.14 Laboratory Health and Safety Manual.

APPENDIX A. ACQUISITION PARAMETERS

Identifier	Dwell time (s)	Channels	Spacing (u)	Measurement mode	Resolution
6Li (STD)	0.1	1	0.1	STD	Normal
9Be (STD)	0.1	1	0.1	STD	Normal
11B (KED)	0.2	1	0.1	KED	Normal
27Al (KED)	0.05	1	0.1	KED	Normal
45Sc (KED)	0.05	1	0.1	KED	Normal
45Sc (STD)	0.1	1	0.1	STD	Normal
47Ti (KED)	0.05	1	0.1	KED	Normal
51V (KED)	0.05	1	0.1	KED	Normal
52Cr (KED)	0.05	1	0.1	KED	Normal
53Cr (KED)	0.05	1	0.1	KED	Normal
54Fe (KED)	0.05	1	0.1	KED	Normal
55Mn (KED)	0.05	1	0.1	KED	Normal
59Co (KED)	0.05	1	0.1	KED	Normal
60Ni (KED)	0.05	1	0.1	KED	Normal
62Ni (KED)	0.05	1	0.1	KED	Normal

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63Cu (KED)	0.05	1	0.1	KED	Normal
65Cu (KED)	0.05	1	0.1	KED	Normal
66Zn (KED)	0.05	1	0.1	KED	Normal
67Zn (KED)	0.05	1	0.1	KED	Normal
68Zn (KED)	0.05	1	0.1	KED	Normal
71Ga (KED)	0.05	1	0.1	KED	Normal
75As (KED)	0.2	1	0.1	KED	Normal
77Se (KED)	0.2	1	0.1	KED	Normal
78Se (KED)	0.2	1	0.1	KED	Normal
82Se (KED)	0.2	1	0.1	KED	Normal
83Kr (KED)	0.05	1	0.1	KED	Normal
88Sr (KED)	0.05	1	0.1	KED	Normal
91Zr (KED)	0.05	1	0.1	KED	Normal
92Mo (KED)	0.05	1	0.1	KED	Normal
94Zr (KED)	0.05	1	0.1	KED	Normal
95Mo (KED)	0.05	1	0.1	KED	Normal
97Mo (KED)	0.05	1	0.1	KED	Normal
98Mo (KED)	0.05	1	0.1	KED	Normal
99Ru (KED)	0.05	1	0.1	KED	Normal
103Rh (KED)	0.05	1	0.1	KED	Normal
105Pd (KED)	0.05	1	0.1	KED	Normal
106Cd (KED)	0.05	1	0.1	KED	Normal
107Ag (KED)	0.05	1	0.1	KED	Normal
108Cd (KED)	0.05	1	0.1	KED	Normal
109Ag (KED)	0.05	1	0.1	KED	Normal
111Cd (KED)	0.05	1	0.1	KED	Normal
114Cd (KED)	0.05	1	0.1	KED	Normal
118Sn (KED)	0.05	1	0.1	KED	Normal
121Sb (KED)	0.05	1	0.1	KED	Normal
123Sb (KED)	0.05	1	0.1	KED	Normal
135Ba (KED)	0.05	1	0.1	KED	Normal
137Ba (KED)	0.05	1	0.1	KED	Normal
159Tb (KED)	0.05	1	0.1	KED	Normal
203Tl (KED)	0.05	1	0.1	KED	Normal
205Tl (KED)	0.05	1	0.1	KED	Normal
206Pb (KED)	0.05	1	0.1	KED	Normal
207Pb (KED)	0.05	1	0.1	KED	Normal
208Pb (KED)	0.05	1	0.1	KED	Normal
209Bi (KED)	0.05	1	0.1	KED	Normal
232Th (KED)	0.05	1	0.1	KED	Normal

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238U (KED)	0.05	1	0.1	KED	Normal

APPENDIX B. METHOD PARAMETERS

Analyte	Measurement Mode	Quantify	Internal Standard	Fit Type	Weighting	Forcing	Use for SemiQuant
6Li	STD	No	Use as Internal Standard	Linear	None	Blank	Yes
9Be	STD	Yes	6Li	Linear	None	Blank	Yes
11B	KED	Yes	Use Interpolation	Linear	None	Blank	Yes
27Al	KED	Yes	Use Interpolation	Linear	None	Blank	Yes
45Sc	STD	No	Use as Internal Standard	Linear	None	Blank	Yes
45Sc	KED	No	Use as Internal Standard	Linear	None	Blank	Yes
47Ti	KED	Yes	Use Interpolation	Linear	None	Blank	Yes
51V	KED	Yes	Use Interpolation	Linear	None	Blank	Yes
52Cr	KED	Yes	Use Interpolation	Linear	None	Blank	Yes
53Cr	KED	Yes	Use Interpolation	Linear	None	Blank	Yes
54Fe	KED	Yes	Use Interpolation	Linear	None	Blank	Yes
55Mn	KED	Yes	Use Interpolation	Linear	None	Blank	Yes
59Co	KED	Yes	Use Interpolation	Linear	None	Blank	Yes
60Ni	KED	Yes	Use Interpolation	Linear	None	Blank	Yes
62Ni	KED	Yes	Use Interpolation	Linear	None	Blank	Yes
63Cu	KED	Yes	Use Interpolation	Linear	None	Blank	Yes
65Cu	KED	Yes	Use Interpolation	Linear	None	Blank	Yes
66Zn	KED	Yes	Use Interpolation	Linear	None	Blank	Yes
67Zn	KED	Yes	Use Interpolation	Linear	None	Blank	Yes
68Zn	KED	Yes	Use Interpolation	Linear	None	Blank	Yes
71Ga	KED	No	Use as Internal Standard	Linear	None	Blank	Yes
75As	KED	Yes	Use Interpolation	Linear	None	Blank	Yes
77Se	KED	Yes	Use Interpolation	Linear	None	Blank	Yes
78Se	KED	Yes	Use Interpolation	Linear	None	Blank	Yes
82Se	KED	Yes	Use Interpolation	Linear	None	Blank	Yes
83Kr	KED	Yes	Use Interpolation	Linear	None	Blank	Yes
88Sr	KED	Yes	Use Interpolation	Linear	None	Blank	Yes
91Zr	KED	Yes	Use Interpolation	Linear	None	Blank	Yes
92Mo	KED	Yes	Use Interpolation	Linear	None	Blank	Yes
94Zr	KED	Yes	Use Interpolation	Linear	None	Blank	Yes
95Mo	KED	Yes	Use Interpolation	Linear	None	Blank	Yes
97Mo	KED	Yes	Use Interpolation	Linear	None	Blank	Yes

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Analyte	Measurement Mode	Quantify	Internal Standard	Fit Type	Weighting	Forcing	Use for SemiQuant
98Mo	KED	Yes	Use Interpolation	Linear	None	Blank	Yes
99Ru	KED	Yes	Use Interpolation	Linear	None	Blank	Yes
103Rh	KED	No	Use as Internal Standard	Linear	None	Blank	Yes
105Pd	KED	Yes	Use Interpolation	Linear	None	Blank	Yes
106Cd	KED	Yes	Use Interpolation	Linear	None	Blank	Yes
107Ag	KED	Yes	Use Interpolation	Linear	None	Blank	Yes
108Cd	KED	Yes	Use Interpolation	Linear	None	Blank	Yes
109Ag	KED	Yes	Use Interpolation	Linear	None	Blank	Yes
111Cd	KED	Yes	Use Interpolation	Linear	None	Blank	Yes
114Cd	KED	Yes	Use Interpolation	Linear	None	Blank	Yes
118Sn	KED	Yes	Use Interpolation	Linear	None	Blank	Yes
121Sb	KED	Yes	Use Interpolation	Linear	None	Blank	Yes
123Sb	KED	Yes	Use Interpolation	Linear	None	Blank	Yes
135Ba	KED	Yes	Use Interpolation	Linear	None	Blank	Yes
137Ba	KED	Yes	Use Interpolation	Linear	None	Blank	Yes
159Tb	KED	No	Use as Internal Standard	Linear	None	Blank	Yes
203Tl	KED	Yes	Use Interpolation	Linear	None	Blank	Yes
205Tl	KED	Yes	Use Interpolation	Linear	None	Blank	Yes
206Pb	KED	Yes	Use Interpolation	Linear	None	Blank	Yes
207Pb	KED	Yes	Use Interpolation	Linear	None	Blank	Yes
208Pb	KED	Yes	Use Interpolation	Linear	None	Blank	Yes
209Bi	KED	No	Use as Internal Standard	Linear	None	Blank	Yes
232Th	KED	Yes	Use Interpolation	Linear	None	Blank	Yes
238U	KED	Yes	Use Interpolation	Linear	None	Blank	Yes

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APPENDIX C. ICPMS iCAP Q PERFORMANCE REPORT LIMITS

STANDARD MODE (STD) PERFORMANCE REPORT LIMITS

^{7}Li	> 50,000 cps
^{59}Co	> 100,000 cps
^{115}In	> 220,000 cps
^{238}U	> 300,000 cps
$^{137}Ba^{++}$ / ^{137}Ba	< 3 %
$^{140}Ce^{16}O$ / ^{140}Ce	< 2 %
Bkg 4.5	< 1 cps
Bkg 220.7	< 2 cps

Mass Calibration Verification:

^{7}Li: minimum peak width: 0.65 amu; maximum peak width: 0.85 amu

^{59}Co: minimum peak width: 0.65 amu; maximum peak width: 0.85 amu

^{115}In: minimum peak width: 0.65 amu; maximum peak width: 0.85 amu

^{238}U: minimum peak width: 0.65 amu; maximum peak width: 0.85 amu

KINETIC ENERGY DISCRIMINATION (KED) PERFORMANCE REPORT LIMITS

^{59}Co	> 30,000 cps
^{115}In	> 30,000 cps
^{238}U	> 80,000 cps
$^{140}Ce^{16}O$ / ^{140}Ce	< 1 %
Bkg 4.5	< 0.5 cps
Bkg 220.7	< 2 cps
KED	≥ 18 cps

Mass Calibration Verification:

^{59}Co: minimum peak width: 0.65 amu; maximum peak width: 0.85 amu

^{115}In: minimum peak width: 0.65 amu; maximum peak width: 0.85 amu

^{238}U: minimum peak width: 0.65 amu; maximum peak width: 0.85 amu

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APPENDIX D. ICPMS iCAP RQ PERFORMANCE REPORT LIMITS

STANDARD MODE (STD) PERFORMANCE REPORT LIMITS

^{7}Li	> 55,000 cps
^{59}Co	> 100,000 cps
^{115}In	> 240,000 cps
^{238}U	> 330,000 cps
$^{137}Ba^{++}$ / ^{137}Ba	< 3 %
$^{140}Ce^{16}O$ / ^{140}Ce	< 2 %
Bkg 4.5	< 1 cps
Bkg 220.7	< 2 cps

Mass Calibration Verification:

^{7}Li: minimum peak width: 0.65 amu; maximum peak width: 0.85 amu

^{59}Co: minimum peak width: 0.65 amu; maximum peak width: 0.85 amu

^{115}In: minimum peak width: 0.65 amu; maximum peak width: 0.85 amu

^{238}U: minimum peak width: 0.65 amu; maximum peak width: 0.85 amu

KINETIC ENERGY DISCRIMINATION (KED) PERFORMANCE REPORT LIMITS

^{59}Co	> 30,000 cps
^{115}In	> 35,000 cps
^{238}U	> 85,000 cps
$^{140}Ce^{16}O$ / ^{140}Ce	< 1 %
Bkg 4.5	< 0.5 cps
Bkg 220.7	< 2 cps
KED	≥ 18 cps

Mass Calibration Verification:

^{59}Co: minimum peak width: 0.65 amu; maximum peak width: 0.85 amu

^{115}In: minimum peak width: 0.65 amu; maximum peak width: 0.85 amu

^{238}U: minimum peak width: 0.65 amu; maximum peak width: 0.85 amu

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APPENDIX E. Marine SRM, NASS-7-SPK3 Concentrations

Element	Mass Concentration, µg/L	NASS-7 Spike, µg/L	NASS-7-SPK3, µg/L	NASS-7-SPK3 diluted by 10, µg/L	NASS-7-SPK3 diluted by 5, µg/L
Boron	3750 ± 120	0	3750	375	750
Cadmium	0.0161 ± 0.0016	5000	50.0161	5.00161	10.00322
Cobalt	0.0146 ± 0.0014	5000	50.0146	5.00146	10.00292
Copper	0.199 ± 0.014	5000	50.199	5.0199	10.0398
Iron	0.351 ± 0.026	5000	50.351	5.0351	10.0702
Manganese	0.75 ± 0.06	5000	50.75	5.075	10.15
Molybdenum	9.29 ± 0.40	5000	59.29	5.929	11.858
Nickel	0.248 ± 0.018	5000	50.248	5.0248	10.0496
Lead	0.0026 ± 0.0008	5000	50.0026	5.00026	10.00052
Uranium	2.87 ± 0.16	5000	52.87	5.287	10.574
Zinc	0.42 ± 0.08	10000	100.42	10.042	20.084
Arsenic*	1.26 ± 0.06	5000	51.26	5.126	10.252
Chromium*	0.107 ± 0.016	5000	50.107	5.0107	10.0214
Vanadium*	1.30 ± 0.08	5000	51.3	5.13	10.26

*Reference values

APPENDIX F. APPENDIX OF SIGNIFICANT CHANGES

01/18/2018 Gadolinium (Gd) added.

03/05/2019 ICAP STD 5 added as well as % Relative Error limits.
Additional PQL standards preparation instructions added
Preparation and analysis instruction for NASS-7-SPK3 added
Gadolinium added as an interferent to IC6020C.
Sections 8 and 9 edited for clarity
Expanded troubleshooting section

02/20/2023 Sections 12 and 13 edited for more clarity.

01/17/2024 Section 10.5 edited to add additional peristaltic pump rinsing to help with aluminum contamination.

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