ANDREW M. CUOMO
Governor

HOWARD A. ZUCKER, M.D., J.D.
Commissioner

SALLY DRESLIN, M.S., R.N.
Executive Deputy Commissioner

New York State Department of Health - Wadsworth Center

Laboratory of Inorganic and Nuclear Chemistry –

NYS ELAP Laboratory ID 10762

Division of Environmental Health Sciences Albany, New York

Metals and Metalloids in Medical Marijuana Products by ICP-MS

NYS DOH LINC-250

Table of Contents

1	**Scope and Application**	**3**
2	**Summary of the Method**	**4**
3	**Definitions**	**4**
4	**Health and Safety Warnings**	**6**
5	**Interferences**	**7**
6	**Instrumentation, Equipment and Supplies**	**9**
7	**Reagents and Standards**	**10**
8	**Preparation of Reagents, Solutions, and Standards**	**11**
9	**Shipping Conditions, Receiving, and Storage**	**14**
10	**Sample Preparation**	**15**
11	**Instrument Tuning and Calibration**	**16**
12	**Quality Control and Assurance**	**20**
13	**Sample Analysis**	**22**
14	**Data Acquisition, Reduction, Analysis, and Calculations**	**22**
15	**Data Assessment, Acceptance Criteria, and Corrective Actions for Out-of-Control Data**	**23**
16	**Method Performance**	**25**
17	**Waste Management/Pollution Prevention**	**25**
18	**References**	**26**

1 Scope and Application

1.1 This method NYS DOH LINC-250 (ELAP Method ID 9984) is used for the determination of trace metals and metalloids in medical marijuana (MM) products by Inductively Coupled Plasma Mass Spectrometry (ICP-MS).

1.2 This method is to be used for the analysis of medical marijuana products as defined under NYS regulation Title 10 (Health), Subpart 55-2.15 and Chapter XIII, Section 1004.14 for the determination of total recoverable concentrations of elemental contaminants, i.e, toxic metals and metalloids. Based on the current regulations, approved medical marijuana products shall be limited to the forms of administration approved by the Department, including but not limited to: metered liquid or oil preparations; solid and semisolid preparations (e.g. capsules, chewable and effervescent tablets, lozenges); metered ground plant preparations; and topical forms and transdermal patches. Medical marijuana may not be incorporated into food products by the registered organization, unless approved by the commissioner.

1.3 This method is to be used by, or under the supervision of, properly experienced and trained personnel, who are knowledgeable in the recognition and correction of spectral, chemical, and physical interferences in ICP-MS. Each analyst must demonstrate the ability to generate acceptable results with this method.

1.4 The analytical portion of this method is similar to that described in the U.S. Environmental Protection Agency (US EPA) Method 200.8.

Note – Use of trade names is for informational purposes only and does not imply an endorsement by the Wadsworth Center or the New York State Department of Health (DOH).

1.5 Currently regulated analytes are detailed in Table 1. The method can also be used to identify other analytes, provided that the method performance is validated for each analyte prior to use. Table 1 summarizes the Limit of Detection (LOD) and Limit of Quantitation (LOQ) for metals in a representative olive oil matrix. The LODs and LOQs are provided as examples. Requirements related to LOD and LOQ are specified in sections 12.1 ad 12.2.

Table 1 – MCT Oil Matrix - Summary of LODs and LOQs

Analyte	CAS number	LOD (µg/L)	LOD (µg/g)	LOQ (µg/L)	LOQ (µg/g)	MRL (µg/g)
Antimony (Sb)	7440-36-0	0.020	0.006	0.050	0.020	2.0
Arsenic (As)	7440-38-2	0.106	0.042	0.319	0.127	0.2
Cadmium (Cd)	7440-43-9	0.079	0.032	0.237	0.095	0.2
Chromium (Cr)	7440-47-3	0.548	0.219	1.64	0.657	2.0
Copper (Cu)	7440-50-8	0.683	0.273	2.18	0.870	2.0
Lead (Pb)	7439-92-1	0.160	0.064	0.060	0.024	0.2
Mercury (Hg)	7439-97-6	0.030	0.012	0.095	0.038	0.2
Nickel (Ni)	7440-02-0	0.145	0.058	0.434	0.474	2.0
Zinc (Zn)	7440-66-6	3.63	1.45	11.6	4.62	20

2 Summary of the Method

2.1 Prior to analysis, samples must be solubilized or digested using an appropriate NYS ELAP approved sample preparation method. Acid digestion can be carried out using microwave assisted heating technologies or by classical open vessel heating. For the current method (NYS DOH LINC-250), all samples are acid digested using a commercial hot block digestion system (Perkin-Elmer SPD 50-48 block digestion system).

2.2 This method describes the multi-elemental determination of analytes by ICP-MS in MM product samples. Sample solutions or digest solutions are introduced into the ICP-MS plasma via a suitable nebulizer/spray chamber arrangement, where the analyte atoms are ionized, extracted and then separated by a quadrupole mass analyzer, based on their mass-to-charge ratio (m/z), and detected by an electron multiplier.

2.3 Several different types of interferences are possible in inorganic mass spectrometry, including isobaric and polyatomic ions that appear at the same m/z as the analyte ion, and subtle matrix effects that occur in the sample introduction system. These interferences must be assessed, and valid correction approaches applied. Interference correction must include compensation for background ions contributed by the plasma gas, reagents, and constituents of the sample matrix. See Section 5 – Interferences for additional detail.

3 Definitions

3.1 Preparation Batch – Samples that are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents. A preparation batch consists of one to twenty samples (not including method blanks, LCS, matrix spikes and matrix duplicates) of the same matrix with a maximum processing time of twenty-four (24) hours between the first and last sample.

3.2 Analytical batch – An analytical batch consists of prepared samples which are analyzed together as a group. An analytical batch can include prepared samples originating from different matrices and can exceed twenty samples.

3.3 Dissolved Analyte – The concentration of analyte in a sample that will pass through a 0.45 µm membrane filter.

3.4 Total Recoverable Analyte – The concentration of analyte determined by analysis of the sample following digestion by refluxing with hot concentrated mineral acids as specified in the method.

3.5 Quality Control (QC) Standard – Any prepared solution that has defined acceptance criteria, and is used to determine if the method is in conformance, or to assess method performance. The QC standards used in this method are: MB, LCS, ICV, MS, MSD, CCV, and CCB.

3.6 Limit of Detection (LOD) – The statistically calculated minimum concentration of an analyte that can be measured with 99% confidence that the value is greater than zero. Also referred to as Method Detection Limit (MDL).

3.7 Limit of Quantitation (LOQ) – The concentration of an analyte that can be reported within the accuracy and precision limits defined by the method. The LOQ can be no lower than the lowest calibration standard used in the analysis.

3.8 Minimum Reporting Limit (MRL) – The level below which the laboratory will not report concentration results for an analyte. The MRL cannot be less than the LOQ, and is determined by the instrument sensitivity and program requirements. Results below this limit are reported as less than that concentration.

3.9 Rinse Blank – A blank solution that is used to rinse the system between samples.

3.10 Calibration Blank (Cal Blank) – A blank solution of the same composition as the calibration standards, used in establishing the calibration curve.

3.11 Primary Standard – A concentrated single- or multi-element standard of method analytes purchased from a certified vendor. Primary standards are traceable to NIST, when available.

3.12 Stock Standard – A primary standard, or a concentrated solution of method analyte(s) prepared from primary standard(s).

3.13 Calibration Standard – A solution of method analytes prepared from stock or higher concentration calibration standard solutions used to calibrate the ICP-MS instrument response with respect to analyte concentration (e.g. Cal 100 µg/L).

3.14 Independent Calibration Verification (ICV) – A solution of method analytes prepared from a second source that is independent of the source used to prepare the calibration standards. It is used to verify the calibration.

3.15 Continuing Calibration Verification (CCV) – One of the primary calibration standards, analyzed periodically to verify that the calibration is still valid.

3.16 Low Level Continuing Calibration Verification (LLCCV) – The lowest primary calibration standard, analyzed at the end of each analytical batch to verify instrument stability at the low end of the calibration range.

3.17 Continuing Calibration Blank (CCB) – The calibration blank solution, analyzed periodically to verify that no contamination has occurred.

3.18 Linear Dynamic Range (LDR) – The concentration range over which the instrument response to an analyte has been demonstrated to be linear.

3.19 Internal Standard – An element that is added to the sample prior to analysis in order to correct for instrument drift. In ICP-MS, an internal standard should be selected for each analyte determined, such that it has a similar 1st ionization potential and an atomic mass close to the analyte. The ratio of the instrument response to the analyte to the response to the internal standard is used as the response variable for generating the calibration curve and concentration data in the samples. Recommended internal standards are 6Li, 45Sc, 72Ge, 89Y, 103Rh, 115In, 159Tb, 193Ir, and 209Bi, but other elements/isotopes may be used at the analyst's discretion, provided they are not found in the sample under analysis.

3.20 Digestion (Method) Blank (MB) – An aliquot of appropriate pure matrix that is carried through the entire sample preparation process, and is treated exactly as a sample including exposure to all glassware, equipment, solvents, and reagents that are used with other samples. The method blank is used to determine whether contamination with method analytes or other interferences are present in the laboratory environment, reagents or apparatus.

3.21 Matrix Spike (MS) –A portion of an actual sample that is first spiked with a known quantity of target analytes, and then carried through the entire sample preparation and analysis process. The sample from which the portion to be spiked was taken must be analyzed separately to determine endogenous background analyte concentrations. The MS is corrected for background concentrations and used to determine whether or not the sample matrix affects the sample results.

3.22 Matrix Spike Duplicate Sample (MSD) – A second portion of actual sample used to prepare the MS that is spiked and processed in the same manner as the MS. The MS and MSD are used together to measure the precision of the methodology.

3.23 Post Digestion Spike (PDS) – A portion of actual sample that is spiked with a known quantity of target analytes after the sample preparation and then analyzed. The sample from which the portion to be spiked was taken must be analyzed separately to determine background analyte concentrations. The PDS is corrected for background concentrations and used to determine whether or not the sample matrix affects the sample results.

3.24 Laboratory Control Sample (LCS) – A portion of appropriate clean matrix that is spiked with known quantities of target analytes and carried through the entire sample preparation process, and treated exactly as a sample, including exposure to all glassware, equipment, solvents, and reagents that are used with other samples. The LCS measures the accuracy of the methodology. The LCS may be prepared from the same source as the calibration standards, or from a second source.

4 Health and Safety Warnings

4.1 Laboratory safety procedures shall be followed at all times. Regulations required by federal, state and local government agencies shall be implemented and followed.

4.2 This method involves the use of concentrated nitric and hydrochloric acids. Both nitric and hydrochloric acids are corrosive and can cause serious burns. They should be used in a chemical fume hood. If eye or skin contact occurs, flush with large volumes of water. Safety glasses or a shield for eye protection must be worn when working with these reagents.

4.3 Always follow the guidelines listed in safety data sheets (SDS) for proper storage, handling, and disposal of solvents, reagents and standards. These guidelines must be made available to all personnel involved in the chemical analysis.

4.4 General safety procedures used in any chemical laboratory must also be followed. Lab coats, safety glasses and gloves must be worn when performing standard or sample preparations, working with instrumentation, disposing of waste, and cleaning glassware.

5 Interferences

5.1 Interferences in ICP-MS may be isobaric or polyatomic species that have the same mass-to-charge ratio (m/z) or matrix effects that occur in the sample introduction system.

5.2 Isobaric (and polyatomic) interferences in ICP-MS are caused by isotopes of different elements, (or molecular species formed in the plasma), forming ions with the same nominal mass-to-charge ratio (m/z).

5.3 They can be reduced by the use of reaction/collision cell technologies, or other, technology, or corrected by using interference equations. Major isobaric/polyatomic interferences are listed in Table 3 below. Additional information on isobaric/polyatomic interferences can be found in EPA Method 200.8 and FDA Elemental Analysis Manual, section 4.7, Table 6.

5.4 Some common isobaric interferences are shown in Table 3. Table 4 shows some recommended interference equations; others may be used at the analyst’s discretion, provided they have been validated.

5.5 Matrix effects may cause either enhancement or suppression of the signal from some analytes. The analyst must be aware of the types of matrix effects, and how to minimize or correct for them.

5.6 High levels of low ionization potential (I.P) elements in the samples can lower the ionization fraction of high I.P. elements, including some internal standards. For samples with very high (generally, > 100 µg/mL) levels of Na or other easily ionizable elements, it may be necessary to add Na or other elements to the calibration standards, in order to better match them to the sample matrix.

5.7 High levels of carbon in the samples may enhance the ionization of high I.P. elements, such as arsenic and selenium, and add to the $^{40}Ar^{12}C$ interference on ^{52}Cr. Some methods recommend adding 2 – 5 % isopropyl or other alcohol to the diluent for samples and standards to boost sensitivity for these elements and reduce the effect of variation in carbon content between samples and standards.

5.8 Other matrix effects may occur, and the analyst should be prepared to counteract them to the extent possible.

Table 3 – Common Isobaric Interferences

Analyte Isotope	Common Isobaric Interferences
^{52}Cr	$^{12}C^{40}Ar$, $^{37}Cl^{15}N$
^{53}Cr	$^{18}O^{35}Cl$, $^{16}O^{37}Cl$
^{60}Ni	$^{44}Ca^{16}O$, $^{23}Na^{37}Cl$
^{63}Cu	$^{23}Na^{40}Ar$, $^{47}Ti^{16}O$, $^{31}P^{16}O_2$
^{65}Cu	$^{49}Ti^{16}O$, $^{40}Ar^{25}Mg$
^{66}Zn	$^{50}Ti^{16}O$, $^{32}S^{16}O^{18}O$
^{68}Zn	$^{34}S^{16}O^{18}O$
^{75}As	$^{40}Ar^{35}Cl$
^{111}Cd	$^{95}Mo^{16}O$, $^{94}Zr^{16}OH$
^{121}Sb	$^{40}Ar^{81}Br$
^{202}Hg	$^{186}W^{16}O$
^{206}Pb	$^{190}Pt^{16}O$
^{207}Pb	$^{191}Ir^{16}O$
^{208}Pb	$^{192}Pt^{16}O$

NYS DOH LINC-250-02 011320 v2 DD

Table 4 – Interference Equations

Analyte Isotope	Interference Equation*
^{75}As	$[^{75}As] = [75] - 3.127*[77] + 2.736*[82] - 2.76*[83]$
^{111}Cd	$[^{111}Cd] = [111] - 1.073*[108] + 0.764*[106]$
^{115}In	$[^{115}In] = [115] - 0.016*[118]$
^{208}Pb	$[^{208}Pb] = [208] + [206] + [207]$
*Here [xx] = counts/sec (CPS) at m/z = xx, and [xxA] = CPS at m/z = xx attributed to isotope xxA	

6 Instrumentation, Equipment and Supplies

6.1 Standard and Sample Preparation Equipment

6.1.1 Pipettes, mechanical, various sizes, fixed or adjustable volume.

6.1.2 Disposable pipette tips, various sizes.

6.1.3 Centrifuge tubes, various sizes, for use as sample cups.

6.1.4 Digestion tubes, 50 mL, Digitube or other Class “A”.

6.1.5 Plastic bottles, various sizes.

6.1.6 Class “A” volumetric flasks with stoppers, various sizes.

6.2 Instrumentation

6.2.1 Agilent 7500ce ICP-MS spectrometer system, or similarly equipped instrumentation with any concentric, or other suitably designed nebulizer and spray chamber arrangement. Liquid argon is used to provide the gas supply necessary to sustain the plasma.

6.2.2 Agilent ASX-500 autosampler, or similar.

6.2.3 Analytical balance, Sartorious A 120 S, or similar, precision 1 mg or better.

6.2.4 Perkin-Elmer SPD 50-48 block digestion system, or similar, with ± 1 °C uniformity.

6.3 Maintaining Labware

6.3.1 All reusable labware will be maintained in a sufficiently clean state to perform ICP-MS analyses at the required concentration levels. If glassware contamination is suspected, it may be checked by preparing a blank acid (1 – 5 % HNO_3) solution in the vessel, letting it stand for at least one hour, and analyzing it. The concentrations of all target analytes must be below the LOQ.

6.3.2 Glassware used for trace metals analysis must not be used for any other purpose and must be maintained in a clean state between analyses. Glassware used for ICP-MS analyses shall be rinsed a minimum of 3 times with purified water (deionized, > 18 MΩ), filled and stored with 2 – 5 % ultra-pure nitric acid (ultra-pure HCl may also be added at 0.5 % concentration), and rinsed a minimum of 3 times with purified water before use.

6.3.3 If glassware becomes contaminated with oily organic substances, it may be necessary to clean it with a laboratory detergent, Citranox® or similar, prior to performing the above cleaning procedure.

6.3.4 Centrifuge tubes, bottles, and other disposable plasticware must be suitable for trace metals analysis. Clean LDPE, HDPE, PP, and Teflon® (PTFE, PFA, or FEP) containers are acceptable.

6.3.5 Pipettors shall be calibrated quarterly, a summary of the procedures is as follows:

6.3.6 Check the accuracy of the analytical balance using NIST traceable certified weights that span the range of masses that will be measured.

6.3.7 Transfer five aliquots of deionized water to a weighing dish and weigh them. Determine the mean and standard deviation of the measurements, and calculate the mean volume, by dividing by the temperature corrected density of water. Calculate the Relative Standard Deviation (RSD) of the measurements. Compare the accuracy and precision with limits established by the laboratory.

6.3.8 All measurements must be recorded, and the results reviewed by a second analyst.

7 Reagents and Standards

7.1 All reagents used shall be of sufficient purity for ICP-MS analysis. The purity is verified by acceptable results for blanks and QC standards. All blanks, standards, and spiking solutions will be prepared from these reagents.

7.2 Primary standards (single or multi-element), shall be NIST traceable, and ICP-MS grade or equivalent. This includes standards used for calibration standards, ICV standards, and internal standards.

7.3 Reagent water used for standard preparations and all dilutions must be high purity, with a resistivity > 18 MΩ, ASTM Type 1 or similar.

7.4 Concentrated nitric acid (HNO_3) will be double distilled, either prepared on site or purchased; the trade name for double distilled acid from different manufacturers will vary.

7.5 Concentrated hydrochloric acid (HCl) will be trace metals grade or higher purity, or double distilled.

7.6 Hydrogen peroxide, 30 %, (H_2O_2) will be A.C.S. reagent grade or better.

7.7 Isopropyl alcohol, A.C.S spectrophotometric grade.

7.8 Liquid Argon, non-graded.

The primary standards listed in Table 5 are currently used. This is not a fully inclusive list, and it is subject to change at any time.

Table 5 – Primary Standards

Manufacturer	**Catalog number**	**Analyte(s)**	**Concentration**
Accustandard	IS-72182	17 metals[a]	10 µg/mL[a]
Accustandard	ICP-MS-200.8-Cal5-1	Hg	10 µg/mL
High-Purity	SM-2326-006	17 metals[a]	10 µg/mL
High-Purity	10 33-1	Hg	10 µg/mL
SCP Science*	140-052-79x	Au	1000 µg/mL
SCP Science**	140-051-031	Li	1000 µg/mL
SCP Science**	140-051-581	Ce	1000 µg/mL
SCP Science**	140-051-391	Y	1000 µg/mL
SCP Science**	140-051-031	Tl	1000 µg/mL

* Au is used as a matrix modifier, and the concentration is not critical, so the primary standard may be used past the manufacturer's expiration date.
**Li, Y, Ce, and Tl are used for the instrument Tuning solution and may be used past the manufacturer's expiration date.
[a]Sb, As, Be, Cd, Cr, Co, Cu, Pb, Mn, Mo, Ni, Se, Tl, U, V @ 1 µg/mL, Ba, Zn @ 10 µg/mL

8 Preparation of Reagents, Solutions, and Standards

8.1 General preparation information

8.1.1 Single and multi-element primary standards that meet the requirements in section 7 will be used to prepare stock solutions, and they must adhere to all regulatory requirements.

8.1.2 Standards preparation steps are for guidance only. These may be interchanged. In addition, different concentrations or alternate stock mixtures may be prepared as necessary.

8.1.3 All reagents, solutions and standards must be traceable to stocks and, if possible, have NIST-traceable documentation. The preparation method, date of preparation, expiration date, and analyst must also be traceable in laboratory documentation.

8.1.4 All solutions will be prepared in clean class "A" glassware, or clean plastic labware.

8.1.5 Unless otherwise specified, in this section HNO_3 refers to concentrated nitric acid, HCl refers to concentrated hydrochloric acid, as described in section 7.

8.1.6 At a minimum, commercial standards/materials are stored per the manufacturer's recommended storage conditions, and expiration dates are as prescribed by the vendor on their Certificate of Analysis.

8.2 Rinse blank (concentration is not critical; volumetric labware is not required): Store at room temperature; expires 1 year after preparation.

8.2.1 Partially fill a plastic container, marked at 3 liters, with reagent water.

8.2.2 Add 60 mL HNO_3 and 3 ml 1000 µg/mL gold primary standard.

8.2.3 Add 15 mL concentrated HCl.

8.2.4 Dilute to mark with reagent water.

8.3 Calibration blank (concentration is not critical; volumetric labware is not required): Store at room temperature; expires 1 year after preparation.

8.3.1 Partially fill a plastic container, marked at 1 liter, with reagent water.

8.3.2 Add 45 ml HNO_3, 5 mL HCl, and 1 ml 1000 µg/mL gold primary standard.

8.3.3 Dilute to mark with reagent water.

8.4 Instrument tuning solution (concentration is not critical; volumetric labware is not required): Store at room temperature; no expiration.

8.4.1 Partially fill a plastic container, marked at 1 liter, with reagent water.

8.4.2 Add 1 ml HNO_3.

8.4.3 Add appropriate amount of primary standard(s) of Li, Y, Ce, and Tl, to obtain final concentrations of 1 µg/L (e.g. 1 ml each of 1000 µg/mL Li, Y, Ce, and Tl). Since the absolute concentrations are not critical, the primary standards may be used past the manufacturer's expiration date.

8.4.4 Dilute to mark with reagent water.

8.4.5 Dilute to final concentration before use ((200 – 500 µg/L), at analyst's discretion), with Calibration Blank Solution.

8.5 Cal 1000 µg/L: Store at room temperature; follow manufacturer’s expiration date.

8.5.1 Use primary standard Accutrace IS-72182.

8.6 ICV 1000 µg/L: Store at room temperature; follow manufacturer’s expiration date.

8.6.1 Use primary standard High-Purity SM-2326-006.

8.7 Hg 10 µg/L: Store at room temperature; expires 5 days after preparation.

8.7.1 Add approximately 50 mL of reagent water to a 100 ml Class A volumetric flask.

8.7.2 Add 4.5 mL HNO_3, 0.5 mL HCl, and 100 µL 1000 µg/mL gold primary standard.

8.7.3 Add primary Hg standard (100 µl ICP-MS-200.8-Cal5-1) to obtain a final concentration of 10 µg/L of Hg.

8.7.4 Dilute to mark with reagent water.

8.8 ICV_Hg 10 µg/L: Store at room temperature; expires 5 days after preparation.

8.8.1 Add approximately 50 mL of reagent water to a 100 mL Class A volumetric flask.

8.8.2 Add 4.5 mL HNO_3, 0.5 ml HCl, and 100 µL 1000 µg/mL gold primary standard.

8.8.3 Add primary Hg standard (second source, 100 µL High Purity 10 33-1) to obtain a final concentration of 10 µg/L of Hg.

8.8.4 Dilute to mark with reagent water.

8.9 Calibration Standards: Store at room temperature: standards containing Hg must be prepared daily when analyzing for mercury, others expire 3 months after preparation.

8.9.1 Add approximately 50 mL of reagent water to a 100 mL Class A volumetric flask.

8.9.2 Add 4.5 mL HNO_3, 0.5 mL HCl, and 100 µL 1000 µg/mL gold primary standard.

8.9.3 Add stock and/or calibration standards as indicated below to obtain the final concentrations shown in Table 6.
A
8.9.3.1 For Cal 100 µg/L, add 10 ml Cal 1000 µg/L and 9 ml Zn 10 µg/mL.

8.9.3.2 For Cal 50 µg/L add 5 ml Cal 1000 µg/L and 4.5 ml Zn 10 µg/mL.

8.9.3.3 For Call 25 µg/L, add 2.5 ml Cal 1000 µg/L and 2.25 ml Zn 10 µg/mL.

8.9.3.4 For Cal 10/1.0 µg/L, add 10 ml Cal 100 µg/L and 10 ml Hg 10 µg/L.

8.9.3.5 For Cal 5.0/2.0 µg/L, add 10 ml Cal 50 µg/L and 20 ml Hg 10 µg/L.

8.9.3.6 For Cal 1.0/0.1 µg/L, add 10 ml Cal 10 µg/L.

8.9.3.7 For Cal 0.5/0.2 µg/L, add 10 ml Cal 5 µg/L.

8.9.4 Dilute to mark with reagent water.

Table 6 – Final Concentrations of Calibration Standards (All standards have Zn concentrations 10x other metals)

Standard	Metals	Zn	Hg
Cal 100 µg/L	100 µg/L	1ooo µg/L	-
Cal 50 µg/L	50 µg/L	500 µg/L	-
Cal 25 µg/L	25 µg/L	250 µg/L	-
Cal 10/1.0 µg/L	10 µg/L	100 µg/L	1.0 µg/L
Cal 5.0/2.0 µg/L	5.0 µg/L	50 µg/L	2.0 µg/L
Cal 1.0 µg/L	1.0 µg/L	10 µg/L	0.1 µg/L
Cal 0.5/0.2 µg/L	0.5 µg/L	5 µg/L	0.2 µg/L

8.10 ICV 10 µg/L (100 ug/L Zn): Store at room temperature; expires 3 months after preparation.

8.10.1 Add approximately 50 mL of reagent water to a 100 mL Class A volumetric flask.

8.10.2 Add 4.5 ml HNO_3, 0.5 mL HCL, and 100 µL 1000 µg/mL gold primary standard.

8.10.3 Add 1 ml ICV 1000 µg/L and 0.9 ml ICV_Zn 10 µg/mL to obtain a final concentration of 10 µg/L each of Cr, Cu, Ni, As, Cd, Sb, and Pb, and 100 µg/L Zn.

8.10.4 Dilute to mark with reagent water.

8.11 ICV_Hg 1.0 µg/L: Prepare daily.

8.11.1 Add approximately 5 mL of Calibration Blank to a 10 mL Class A volumetric flask.

8.11.2 Add 1.0 ml ICV_Hg 10 µg/L.

8.11.3 Dilute to mark with Calibration Blank.

9 Shipping Conditions, Receiving, and Storage

9.1 Sample Shipping Conditions
The medical marijuana products from Registered Organizations (ROs) are shipped as per the manufacturer's specifications and must adhere to all regulatory requirements.

9.2 Sample Receiving
Medical marijuana products from the RO are received, verified and documented ensuring that method, regulatory, and Accreditation Body requirements are met.

9.3 Sample Storage

All medical marijuana products must be stored under the conditions recommended by the manufacturer. The storage is documented.

9.4 Sample Extract Storage
Sample digests may be stored in plastic containers at room temperature, up to 28 days for Hg analysis, and 6 months for other metals, and the storage is documented.

10 Sample Preparation (Preparation of all samples must be documented).

10.1 Weigh approximately 0.125 g (0.110 – 0.150 g.) of sample in a graduated 50 mL digestion tube (section 6.1.4).

10.2 Add 2.25 mL concentrated HNO_3, 0.25 ml concentrated HCl, and 0.50 ml 1000 µg/mL Au. Add required standards according to sample type as shown in the Table 7.

Table 7

Sample Type	Reagents
Method Blank	none
QC 10/1 µg/L (LCS)	0.5 ml Cal 1000 µg/L* 0.5 ml 100 µg/L Hg**
Sample	none
MS/MSD	0.5 ml Cal 1000 µg/L* 0.5 ml 100 µg/L Hg**

*For Cal 1000 µg/L preparation, see section 8.5.
**fresh solution made from 1:100 dilution of 10 µg/mL Hg in 1 % HNO_3/1 µg/mL Au.

10.3 Cover loosely, place in hot block (section 6.2.4) preheated to 90 ± 3 ºC and reflux for 30 minutes.

10.4 Remove samples from hot block and allow to cool.

10.5 Add 1.0 mL 30 % H_2O_2.

10.6 Cover loosely, place in hot block and reflux for 30 minutes at 90 ± 3 ºC.

10.7 Allow to cool to room temperature.

10.8 Fill to 50 mL mark, or fill by weight to 50 g, with DI (> 18 MΩ) water.

11 Instrument Tuning and Calibration

11.1 Instrument Operation and Maintenance

11.1.1 For operation of the Agilent 7500ce ICP-MS instrument, refer to the Agilent 7500 Series ICP-MS Chemstation Operator's Manual and the Agilent 7500 Series ICP-MS Tuning and Application Manual.

11.1.2 For maintenance of the Agilent 7500ce ICP-MS instrument, refer to the Agilent 7500 ICP-MS Hardware Manual.

11.1.3 Typical operating parameters for the Agilent 7500ce are shown in Table 8.

Table 8 – Agilent 7500ce Operating Conditions	
RF Power:	1550 W
Sample Depth:	8.0 mm
Carrier Gas:	1.0 L/min
Aux Gas:	0.2 L/min
Sample flow rate:	0.4 L/min
Spray Chamber Temperature:	2 °C

11.2 Instrument Startup and Tuning

11.2.1 Set up a sequence using the correct calibration block and analysis method for the sample type. Print a copy of the sequence and analysis method for the data packet. Table 9 shows a typical analytical batch. Additional preparation batches and the required QCs may be added as necessary.

11.2.2 Multiple LLCCVs may need to be run since the analytes have different MRLs. With the current instrumentation and MRL, run two (2) LLCCVs, 0.5/0.2 µg/L for As, Cd, Hg, and Pb, and 5.0 µg/L for Cr, Ni, Cu, Sb, and Zn (the Zn concentration in the LLCCV is 50 µg/L).

Table 9 – Typical Analytical Sequence		
Calibration Block		
	Cal Blank	
	Cal 0.5/0.2 µg/L	
	Cal 1.0 µg/L	
	Cal 5.0/2.0 µgL	
	Cal 10/1.0 µg/L	

	Cal 25 µg/L	
	ICV 10 µg/L	
	ICV_Hg 1.0 µg/L	
	CCV 10/1 µg/L	
	CCB	
Sample Block		
	Digestion Blank	MB
	Digestion QC 10/1	LCS
	Sample 1	
	Sample 2	
	Sample 3	
	Sample 4	
	Sample 5	
	Sample 6	
	Sample 7	
	Sample 8	
	Sample 9	
	Sample 10	
	Sample 11	
	Sample 12 spike	MS
	Sample 12 spike dup	MSD
	Sample 13	
	Sample 14	
	Sample 15	
	Sample 16	
	Sample 17	
	Sample 18	
	Sample 19	
	Sample 20	
	CCV 10/1 µg/L	
	CCB	
	LLCCV	

11.2.3 Following the instructions in the Chemstation Operator's Manual, place the instrument in Analysis mode. DI water is used for the probe rinse, and 2 % (v/v) HNO_3 containing 1 µg/mL Au is used for the rinse blank. Depending on the analytes being measured, 0.5 % (v/v) HCl may be added to the rinse blank in order to reduce the washout time.

11.2.4 Allow the instrument to warm up for at least 30 minutes.

11.2.5 Aspirate the tuning solution (1 µg/l Li, Y, Ce, Tl in 1% (v/v) HNO_3).

11.2.6 Tune the instrument and generate a tuning report, and save a copy of the report with the data packet. The RSDs for each mass must be less than 5 %. The peak widths must be less than 0.9 amu at 10 % of peak height. The oxide ratio (Ce/CeO) and the double charge ratio (Ce2+/Ce+) must both be less than 5 %.

11.2.7 Where isobaric/polyatomic interferences are likely to occur, multiple isotopes of an analyte may be monitored, if they exist and are of sufficient natural abundance to yield adequate sensitivity. For a list of recommended isotopes, see Table 10.

11.2.8 Lead is calibrated and reported as the sum of the major stable isotopes ^{206}Pb, ^{207}Pb, and ^{208}Pb, due to isotopic variation in nature that results in different isotope ratios in different substances.

11.2.9 Internal standards are used, either by spiking the diluent used for calibration standards, QC solutions, and samples, or by addition using a separate channel of the peristaltic pump and a mixing block. A 200 µg/L solution is generally used, but higher concentration solutions may be necessary and used at the analyst's discretion. When possible, internal standards should be close in mass and/or 1st ionization potential to the target analyte.

11.2.10 Table 10 shows some recommended analyte and internal standard isotopes, but others may be used at the analyst's discretion, provided they are included in a validation study. After performing a validation study, it was determined that the ^{53}Cr isotope is not suitable for use when operating the ICP-MS spectrometer without using the collision/reaction cell.

If using the mixing block method, aspirate the internal standards solution and verify that the signal strength is sufficient and the RSDs are < 5 %.

Table 10

Recommended monitored isotopes	**Recommended Internal Standards**	**Recommended reporting isotope**
^{52}Cr, ^{53}Cr	^{45}Sc, ^{72}Ge, ^{89}Y, ^{103}Rh	^{52}Cr
^{60}Ni	^{45}Sc, ^{72}Ge, ^{89}Y, ^{103}Rh	^{60}Ni
^{63}Cu, ^{65}Cu	^{45}Sc, ^{72}Ge, ^{89}Y, ^{103}Rh	^{65}Cu
^{66}Zn, ^{68}Zn	^{45}Sc, ^{72}Ge, ^{89}Y, ^{103}Rh	^{66}Zn
^{75}As	^{45}Sc, ^{72}Ge, ^{89}Y, ^{103}Rh	^{75}As

^{111}Cd	^{89}Y, ^{103}Rh, ^{115}In	^{111}Cd
^{121}Sb, ^{123}Sb	^{103}Rh, ^{115}In, ^{159}Tb	^{123}Sb
^{200}Hg, ^{202}Hg	^{193}Ir, ^{209}Bi	^{202}Hg
$^{206, 207, 208}Pb$	^{193}Ir, ^{209}Bi	sum of isotopes

11.3 Calibration and Standardization

11.3.1 Start the automated analysis sequence.

11.3.2 Calibrate the instrument.
A zero point (Calibration Blank) and at least two calibration standards, one of which must be at or below the MRL, are used to generate the calibration curve. Typically, three or more calibration standards are used. A linear calibration curve is used, and a calibration correlation coefficient of ≥ 0.998 is required. The measured concentration of each analyte in the calibration standards must be within ±10 % of the prepared value and the calibration blank must be < MRL. The internal standard response of calibration standards and samples must be within 60 – 140 % of the response in the calibration blank.

11.3.3 Establish an LDR.
An LDR must be established for each analyte annually by analyzing a standard prepared at a suitable concentration. The measured concentration must be within ±10 % of the prepared value. See DEHS-014 for additional details.

11.3.4 After the calibration standards have been acquired, check the calibration curves versus the acceptance criteria; if any are not in conformance, the following changes are allowed to bring them into conformance.

Standards may be removed from the calibration curve for 1 or more elements in accordance with "Calibration Requirements for Routine Inorganic and Organic Chemistry Analyses" (DEHS-014-01) the resulting calibration curve for that analyte satisfies the above requirements. A standard may be removed from the calibration curve for one or more elements if it is the lowest or highest concentration calibration standard and one of the following conditions occurs:

1. The measured concentration is more than ±10 % different from the prepared concentration.

2. Inclusion of the standard causes the correlation coefficient to be < 0.998.

3. The standard is the highest calibration standard for that analyte, inclusion of that standard skews the slope so that lower concentration standards are more than 10 % different from the prepared value.

4. Any other reason as described in DEHS-014.

A standard may be removed from the interior of a calibration curve for all analytes if conditions 1 or 2 above occur for any analyte as long as compliance with DEHS-014 is maintained.

At the discretion of the analyst, a repeat analysis of a removed calibration standard may be run, either with the same solution, or a new preparation, depending on the reason(s) for the removal as long as compliance with DEHS-014 is maintained.

A change in the specific internal standard assigned for a specific analyte may be sufficient to bring the calibration curve standard(s) into conformance.

If, after the above corrections, the calibration curve still does not meet the QC criteria, the root cause must be determined and rectified before analyzing samples. This may entail making all new calibration standards, and/or performing instrument cleaning or maintenance.

11.3.5 Print a calibration report and save it with the data packet.

12 Quality Control and Assurance

12.1 Limit of Detection (LOD) and Limit of Quantitation (LOQ)

12.1.1 An initial LOD study for each method must be completed and documented for all target analytes in each representative matrix (see **MML-301-SOP, Section 7.3**), on each instrument used to analyze sample extracts. If the laboratory intends to report results below the LOQ, an ongoing LOD verification is also required.

12.1.2 Based on the LOD, the laboratory shall select an LOQ that is greater than the LOD (typically 3-5x the LOD) and consistent with the needs of its client. An LOQ is required for each representative matrix, method and analyte combination. For each method, the lowest calibration standard concentration must be at or below the corresponding LOQ.

12.1.3 An initial LOQ study for each method must be completed and documented for all target analytes in each representative matrix. The initial LOD samples may be used for this purpose as long as the concentration used is at or below the LOQ. The mean recovery shall be within 70-130% of the spiked value.

12.1.4 On an ongoing basis, the laboratory shall prepare and analyze a minimum of one LOQ verification sample spiked at the same concentration as the initial LOQ verification study on each instrument during each quarter in which samples are being analyzed for each representative matrix, method, and analyte combination. The recovery of the LOQ verification samples shall be within 70-130%.

12.1.5 12.1.5 For specific procedures, see *Determination of Method Detection Limit (MDL/LOD) and Minimum Reporting Limit (MRL/LOQ) Procedure* (DEHS-013-SOP) which describes the MDL/LOD procedure required through the 2017 Method Update Rule. The 2017 Method Update Rule finalized in the Environmental Protection Agency's (EPA's) Federal Register on August 28,

2017, prescribes a revised approach to Method Detection Limit (MDL)/LOD data collection and calculation per Part 136 Appendix B. The New York State (NYS) Environmental Laboratory Program (ELAP) requires that the revised procedure detailed within the EPA's document *Definition and Procedure for the Determination of the Method Detection Limit, Revision 2, December 2016* be implemented for all NYS ELAP accredited methods.

12.1.6 Note that due to the high sensitivity of ICP-MS and the sample range of interest to the program and Department, the laboratory may establish a MRL that is significantly higher than the statistically determined LOQ. MRL may not be lower than the lowest calibration standard used in the analysis and will be used to meet relevant LOQ requirements appliable to ongoing analyses.

12.2 Demonstration of Capability (DOC)

12.3.1 Each analyst must perform an initial demonstration of capability (DOC) using the procedures described in this method for each target analyte. The initial DOC must consist of the analysis of four matrix spike samples that have been fortified with all analytes of interest at a concentration 1 to 4 times the MRL. For each analyte, the mean recovery value must be within ±20 % of the spiked value. The precision of the measurements, calculated as relative standard deviation (RSD), must be < 10 %.

12.3.2 Each analyst must complete a continuing DOC annually. This may be accomplished either by analyzing a blind sample, which may be an external performance test sample, or an internal sample prepared by another analyst, or by performing an initial DOC as described above at any concentration within the calibration range. All initial and continuing DOCs must be documented.

12.3.3 If there is a change in the ICP-MS instrumentation, or other major change to the method, or if the laboratory or analyst has not performed the method in a 12-month period, the analyst must perform another initial DOC. Minor changes are validated by the analysis of the LCS samples.

12.3 Quality Control Standards

12.3.1 An ICV standard must be analyzed to verify the initial calibration. The concentration range must be within the calibration range for each analyte, preferably near the midpoint of the range. The ICV must be recovered within ± 10% of the prepared concentration.

12.3.2 A CCV and CCB will be analyzed before beginning to analyze samples, after each analytical batch of 20 samples or less, and at the end of the analysis. The CCV concentration must be ≤ ½ the highest calibration standard and must be recovered within ± 10% of the predicted concentration. The CCB must be < MRL.

12.3.3 An LLCCV will also be run at the end of each analytical batch of 20 samples or less. The measured value must be within ± 30 % of the prepared value.

12.3.4 For each preparation batch, at least one MB and one LCS must be carried through the entire sample preparation process and analyzed. The measured concentration of each analyte in the MB must be < MRL, and the LCS recovery must be within ± 15% of the prepared concentration.

12.3.5 At least one MS/MSD pair must be analyzed with each preparation batch of 20 samples or less. The spike amount should be near the midpoint of the calibration range for each analyte, where possible. The recovery of each analyte must be ± 30 % of the spiked value. If the spike amount is less than 20 % of the endogenous amount of an analyte in the sample, the spike recovery requirement for that sample is not applicable. The relative percent difference (RPD) between the MS and MSD must be < 20 %.

12.3.6 Any other quality control standards required by state regulations and standards.

13 Sample Analysis

Samples must be acid digested prior to analysis to remove any organic matrix, and ensure all analytes are in solution (section 10). If the instrument calibration is acceptable (section 11), continue to analysis.

13.1 Transfer a minimum of 5 mL of each sample digest solution to a 15 mL centrifuge tube and place it in the assigned position in the autosampler.

The instrument software monitors IS response and QC sample performance. If any of these parameters fall outside of the acceptable range (section 12), follow the corrective actions described in the section 15.

13.2 When the analysis sequence is complete, rinse the system with a rinse acid (e.g. 2 % HNO_3, 0.5 % HCl) for a minimum of 5 minutes, followed by reagent water for a minimum of 5 minutes.

13.3 Place the system in standby mode.

14 Data Acquisition, Reduction, Analysis, and Calculations

14.1 Perform calculations.
The instrument software (e.g., Agilent Masshunter) calculates the concentrations (aqueous units), spike recoveries, and duplicate precision results, using the following formulae:

$$\text{MS recovery} = (C_{MS} - C_{sample})/C_{spiked}$$
$$\text{RPD} = (C_{MSD} - C_{MS})/\text{mean}(C_{MSD}, C_{MS})$$

where C_{MS} = concentration in the matrix spike, C_{sample} = concentration in the sample, C_{spiked} = spiked amount (concentration), and C_{MSD} = concentration in the matrix spike dup

The aqueous concentrations (µg/L) must be converted to mass fraction units (µg/g) by the analyst before reporting, using the following formula:

Mass fraction (µg/g) = concentration (µg/L)*sample volume (L) / sample mass (g)

14.2 If the instrument software doesn't do the conversion, the sample aqueous concentrations (µg/L) must be converted to mass fraction units (µg/g) by the analyst before reporting, using the following formula:

Mass fraction (µg/g) = concentration (µg/L)*sample volume (L) / sample mass (g)

Enter valid results into the laboratory's database (e.g., CLIMS) after the analysis is complete.

14.3 Review and evaluate the results. This is performed by a second analyst or supervisor.

Data are reviewed to ensure that calculations and transcriptions are correct, and that all QC criteria are met. All final reports must be reviewed for accuracy.

Analytes not detected, or detected at a concentration below the MRL, are reported as less than (<) the MRL value. Analytes detected at a concentration at or above the MRL are reported to 3 significant figures.

For those analytes that are monitored at more than one m/z, the isotope used for reporting is up to the analyst's discretion, but must be used for all samples, standards, and controls, and have been validated. While the reported isotope used may be different for different matrices or samples, that same isotope must be used for the respective controls and standards. For example, if Cr is monitored at m/z 52 and 53, and sample A and B are analyzed, yet Cr in sample A is reported using m/z 52, while in sample is reported using m/z 53, the former must be based on calibration and control data at m/z 52. Likewise, the data for sample B reported based on m/z 53 must be based on calibration and control data at m/z. 53. The purpose here is to preserve the flexibility to handle different sample matrices,

14.4 Report the results to the client after they have been reviewed and approved.

15 Data Assessment, Acceptance Criteria, and Corrective Actions for Out-of-Control Data

Data which are unacceptable according to the QC criteria stated below shall not be reported, with the exception of MS/MSD failures. The sample(s) shall be reanalyzed after determining and correcting the source of the problem.

15.1 If the calibration curve fails the QC criteria in section 11.3, the cause will be determined and corrected, and the instrument recalibrated before analyzing any samples.

15.2 If the ICV or LCS fails the recovery check, of +/-10% or +/-15% respectively, it may be rerun once. A fresh solution may prepared and analyzed, if necessary. If the ICV or LCS fails a second time, the cause will be determined and corrected, and the instrument will be recalibrated. An ICV and LCS must pass acceptance criteria before analyzing samples.

15.3 If the concentration of an analyte in a sample is above the LDR, the sample will be diluted and rerun. Samples within the LDR may also be diluted and rerun if significant matrix effects are suspected.

15.4 If carryover from a sample with a high concentration is suspected, the suspect sample(s) will be rerun after sufficient rinse time has elapsed. The absence of contaminants may be verified by running a rinse blank, or by the results of samples analyzed subsequent to the first analysis of the suspect sample, with concentrations of the affected contaminants < MRL.

15.5 If the matrix spike recovery (± 30%) for an analyte is out of the control range, but the QC results for that analyte are in control, the recovery problem is assumed to be matrix related. If the sample concentration is within or above the LDR, a portion of the sample can be diluted appropriately, an aliquot of the diluted portion spiked, the diluted sample and PDS analyzed, and the results compared with the QC criteria to verify the concentration. If the undiluted sample concentration divided by the spike recovery is less than the MRL, this procedure is not necessary.

15.6 Some samples may be nonhomogeneous in metals contents, causing an MS or MSD failure. If a sample of a type known to be nonhomogeneous has an MS and/or MSD result is outside of the acceptable range, a post digestion spike (PDS) of the sample should be analyzed to verify the recovery of the analytes.

15.7 If the above procedure doesn't meet the MS recovery criteria, the data user will be informed that the result for that analyte is suspect due to either a non-homogeneous sample or an uncorrected matrix effect.

15.8 If an MS/MSD fails the precision criterion (RPD > 20 %), the cause of the failure will be determined and corrected, and the MS/MSD pair reanalyzed. A new MS/MSD pair may be prepared if necessary.

15.9 If a continuing calibration verification standard (CCV) falls outside the control range (± 10 %) for continuing analysis, it may be repeated, optionally after running a blank. If it still fails, assess and correct the cause if possible. Repeat the CCV analysis. If the result is in conformance, continue with the analysis, otherwise recalibrate the instrument or end the sample run.

15.10 If a CCB fails (≥ MRL), it will be rerun once with the same solution. If the contamination persists, it will be rerun with new solution. If the contamination persists, the source of the contamination will be determined and eliminated, if possible. Sample analysis will not continue until the contamination has been eliminated. If it is likely that the contamination has affected the prior samples significantly, they must be reanalyzed for all affected analytes.

15.11 If a CCV falls outside the control range, and a change of the internal standard used for measuring that analyte brings the concentration into conformance, the data for the previous 10 samples are considered valid providing that all previous QC checks are in conformance when reprocessed using the changed internal standard. Sample analysis may continue if the QC standards are within the control limits.

15.12 If an LLCCV falls outside of the control range (± 30%), and cannot be brought into control by the above procedure for CCVs, any samples in the preceding or subsequent block with concentrations less than 3 times the LLCCV control range will reanalyzed for the affected analytes.

15.13 If an analysis is stopped due to a CCV failure, but the CCV result is within ± 15 % for all analytes, the data may be reported, with the CCV exceedance flagged. If it is outside of that range, all of the samples analyzed after the last in control CCV will have to be reanalyzed.

15.14 If an internal standard falls outside of the control range, 60 - 140%, any analytes which are normalized to that internal standard may be reprocessed using a different (but suitable) internal standard. If all the prior QCs for those analytes pass when reprocessed using the alternate internal standard, sample analysis may continue, and the concentration data may be reported. If this is not possible, dilution and reanalysis of the sample may restore the internal standard conformance. If the subsequent CCV/CCB/LLCCV pass the QC criteria, the samples within that block that have acceptable IS response may be reported. If neither of these

procedures are successful, it may necessary be to reanalyze the sample(s) with calibration standards better matched to the sample matrix.

16 Method Performance

The sensitivity of the method is indicated by the LODs for each analyte. The accuracy and precision are indicated by the demonstration of capability for each analyte. In order to assess analytical uncertainty at different analyte concentrations, the analyst may select a calibration standard, independent calibration verification standard, or other QC standard at the appropriate concentration level. The standard is matrix matched for the appropriate analysis, and, if applicable, acid digested in the same manner as the samples. A minimum of seven measurements of the selected standard will be used, either from historical data or from a batch run specifically to determine the measurement uncertainty. The measurement uncertainty will be expressed as the 95 % confidence interval of the selected analyses.

17 Waste Management/Pollution Prevention

17.1 It is the responsibility of the laboratory to comply with all federal, state and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions.

17.2 Minimize digest, chemical, reagent, and standard use whenever possible to reduce the amount of hazardous waste generated.

17.3 Dispose of digested waste in an appropriate waste container properly labeled.

17.4 Dispose of non-hazardous aqueous waste in the laboratory sink followed by flushing with tap water.

17.5 Dispose of glassware in appropriately labeled boxes. Be sure that, whenever possible, the glass has been thoroughly rinsed and is contaminant-free before disposal.

17.6 Consult federal, state and local regulations for additional information or for information on the disposal of products not described in this method.

18 References

18.1 Agilent 7500 Series Chemstation Operator's Manual

18.2 Agilent 7500 ICP-MS Hardware Manual

18.3 Agilent 7500 Series Tuning & Applications Handbook

18.4 EPA Method 200.8: Determination of trace elements in waters and wastes by inductively coupled plasma – mass spectrometry. Revision 5.4 (1994). Environmental Monitoring Systems Laboratory, Office of Research and Development, U. S. Environmental Protection Agency, Cincinnati, OH 45268

18.5 Inductively Coupled Plasma-mass Spectrometric Determination of Arsenic, Cadmium, Chromium, Lead, Mercury and Other Elements in Food Using Microwave Assisted Digestion. http://www.fda.gov/downloads/Food/FoodScienceResearch/LaboratoryMethods/UCM377005.pdf

18.6 Public Health Law, section 502 of the Public Health Law ("PHL"), Title 10 (Health) of The Official Compilation of Codes, Rules and Regulations of the State of New York (NYCRR) subpart 55-2 (Approval of Laboratories Performing Environmental Analysis). http://w3.health.state.ny.us/dbspace/NYCRR10.nsf/56cf2e25d626f9f785256538006c3ed7/c9252587bc832b 3485256c390055920a?OpenDocument&Highlight=0,section,55)

18.7 Manual for the Certification of Laboratories Analyzing Drinking Water (5th Edition), Environmental Protection Agency (EPA). EPA 815-R-05-004. January 2005.