Standard Operating Procedure for the Screening of Marijuana for Toxic Metals by Inductively Coupled Plasma Mass Spectrometry

(Toxic Metals by ICP-MS)

Method ECLS-I-MM-1

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Federal DEA Registration # PD112627
New Jersey CDS Registration # CA00027600

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SCREENING FOR TOXIC METALS IN MARIJUANA BY INDUCTIVELY COUPLED PLASMA-MASS SPECTROMETRY

1.0 Identification of the Test Method

1.1. This method is a screening procedure for select toxic metals in marijuana.

1.2. The DOH-ECLS method number is ECLS-I-MM-1.

1.3. Based on US EPA Method 200.8 (Analysis) and SW846 Method 3050B (sample preparation).

2.0 Applicable Matrix and Matrices

2.1. This method is applicable to plant matrices, specifically marijuana.

3.0 Detection Limits

3.1. Results are reported to Reporting Limits (RLs). RLs for each analyte of interest were defined as the lowest, reasonably achievable level at which an analyte can be reliably detected in a marijuana, or a level below which quantitation is not practical. MDLs are not utilized in this method.

3.2. The reporting limits for each analyte are as follows:

Analyte	Symbol (Quantitation Mass)	RL (On Instrument)	RL (In Samples*)	(CAS #)
Arsenic	As (75)	1.00 µg/L	0.50 µg/g	7440-38-2
Cadmium	Cd (111)	0.20 µg/L	0.10 µg/g	7440-43-9
Chromium	Cr (52)	4.00 µg/L	2.00 µg/g	7440-47-3
Iron	Fe (57)	5.00 µg/L	2.50 µg/g	7439-89-6
Lead	Pb (208)	0.20 µg/L	0.10 µg/g	7439-92-1
Manganese	Mn (55)	5.00 µg/L	2.50 µg/g	7439-96-5
Mercury	Hg (202)	0.50 µg/L	0.25 µg/g	7439-97-6
Nickel	Ni (62)	0.50 µg/L	0.25 µg/g	7440-02-0
Selenium	Se (82)	1.00 µg/L	0.50 µg/g	7782-49-2
Zinc	Zn (66)	10.0 µg/L	5.00 µg/g	7440-66-6

* Assumes that 0.1 g of sample is digested and diluted to a final volume of 50 mL.

4.0 Scope and Application

4.1. This method provides a screening procedure for the determination of select, total recoverable elements by Inductively Coupled Plasma – Mass Spectrometry (ICP-MS) in total quant mode for marijuana. All samples are digested prior to analysis.

4.2. The total recoverable sample digestion procedure given in this method will make soluble and hold in solution the analytes of interest. Literature suggests that some forms of mercury may be lost in this digestion procedure, but our studies thus far have yielded acceptable recoveries for mercury (22.0).

4.3. Use of this method is limited to analysts experienced in the use of ICP-MS, with the interpretation of interferences, and procedures for their correction. It is recommended that an analyst have a minimum of six months of closely supervised experience with commercial instrumentation.

4.4. Users of the method data should state the data-quality objectives prior to analysis. Users of this method must document and have on file the required initial demonstration of performance data.

5.0 Summary of Method

5.1. A 0.1-0.2 g aliquot of well blended, homogeneous plant material is measured in the Chemical Terrorism (CT) laboratory and placed in a quartz microwave tube with 7 mL of ultra-trace nitric acid. This is done for the dual purposes of beginning the sample preparation process and to destroy any cannabinoids in the sample, rendering

it a non-controlled substance. This tube is then transferred to the Metals Laboratory for sample digestion via microwave (ECLS-I-MD-1).

5.2. The method describes the multi-elemental determinations by ICP-MS in a quantitative method by Agilent 8900 QQQ (8900). Introduction of the sample solution is by pneumatic nebulization, into radio-frequency plasma where energy transfer processes cause desolvation, atomization, and ionization. The ions are extracted from the plasma through a differentially pumped vacuum interface and separated based on their mass-to-charge ratio by a quadrupole mass spectrometer having a minimum resolution capability of 1 amu peak width at 5% peak height. The ions transmitted through the quadrupole are detected by an electron multiplier or Faraday detector and the ion formation processed by a data handling system.

6.0 Definitions

6.1. Calibration Blank (CB) – A volume of reagent water acidified with the same acid matrix as the samples. Use of the calibration blank is as a zero standard and to auto-zero the instrument (12.1.1).

6.2. Calibration Standard (S1-S4) – A solution acidified with the same acid matrix as the samples and prepared from the dilution of stock standard solutions. Use calibration standards to calibrate the instrument response with respect to analyte concentration (10.6).

6.3. Daily Performance Check Solution (DPC) – A solution used to tune and determine acceptable instrument performance prior to calibration and sample analysis. For this method, the Daily Performance Check solution for the 8900 contains 1 ppb of Ce, Co, Li, Mg, Tl, and Y (10.13).

6.4. Dual Detector Calibration Solution – A solution of analytes at various concentrations used for normalizing the instrument's pulse and analog detectors. Most elements are at a default concentration of 200 ppb.

6.5. Instrument Calibration Verification (CV) – A solution of analytes used to evaluate the performance of the instrument with respect to a defined set of method criteria. For this method, rerun S3 to evaluate instrument performance (12.2).

6.6. Internal Standard (IS) – Pure analytes added to a sample, extract, or standard solution in known amounts and used to measure the relative responses of other method analytes that are components of the same sample or solution. The internal standard must be an analyte that is not a sample component (10.12).

6.7. Laboratory Duplicates (DUP) – Two aliquots of the same sample taken in the laboratory and analyzed separately with identical procedures. An analysis of a sample and its duplicate indicates precision associated with laboratory procedures, but not with sample collection, preservation, or storage procedures (12.4).

6.8. Blank Spike (BS) – An aliquot of BLK (6.10) to which known quantities of analytes are added in the laboratory and analyzed as a sample. The purpose of the BS is to determine whether the methodology is in control and whether the laboratory is capable of accurate and precise measurements (Sections 12.1.3).

6.9. Matrix Spike (MS) – An aliquot of an environmental sample to which known quantities of analytes are added in the laboratory. Analyze the MS exactly like a sample. Its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the elements in the sample matrix must be determined in a separate aliquot and the measured values in the MS corrected for background concentrations. This sample is also prepared in duplicate for every batch of 20 samples or fewer; the duplicate is designated the Matrix Spike Duplicate (MSD) (Section 12.5).

6.10. Laboratory Reagent Blank (BLK) – An aliquot of blank acid matrix that is treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, and internal standards that are used with the samples. Use of the BLK is to determine if analytes or interferences are present in the laboratory environment, reagents, or apparatus (Sections 12.1.2).

6.11. Linear Range (LR) – The concentration range over which the instrument response to an analyte is linear. A series of solutions with concentrations greater than your highest calibration standard must be analyzed. The highest

concentration that is determined to be ±15% recovery is the LR. Samples above 85% of the LR must be diluted.

6.12. Quality Control Sample, Low Calibration Verification (LCV) and High Calibration Verification (HCV) - A solution of analytes of known concentrations used to fortify blank acid matrix. Obtain the QC stock from a second source from the source of the calibration standards. It is used to check laboratory and instrument performance. The recovery of the LCV and HCV must be ±25% (Section 10.7).

6.13. Reporting Level Check (RLC) - A solution of analytes used to verify the accuracy of the reporting level. The acceptance limits are ± 50% from the true value listed in 3.2 (Section 18.8).

6.14. Rinse Solution – Contains 2% trace metals grade HNO_3, 2% trace metals grade HCl, 200 µg/L Au, and 0.02% Triton-X 100 in reagent water.

6.15. Stock Calibration Standard Solution – A concentrated solution containing one or more analytes prepared in the laboratory using assayed reference materials or purchased from a reputable commercial source.

6.16. Total Recoverable Analyte – The concentration of analyte determined to be in a solid sample following digestion by refluxing with hot dilute mineral acid(s) as specified in the method.

6.17. Sample – An aliquot of marijuana that represents a composite of the individual samples submitted. The composite is homogenized and ground into a fine powder prior to analysis. All sample preparation must be performed by microwave digestion (ECLS-I-MD-1).

7.0 Interference

7.1. Several interferences may cause inaccuracies in the determination of trace elements by ICP-MS. Many of these interferences are inherently controlled by the functionality of the triple quad ICP-MS. Potential interferences are:

7.1.1. Isobaric elemental interference – Caused by isotopes of different elements that form singly or doubly charged ions of the same nominal mass-to-charge ratio and which cannot be resolved by the mass spectrometer in use. All elements determined by this method have, at a minimum, one isotope free of isobaric interference. Of the analytical isotopes used with this method only selenium-82 (krypton) has an isobaric interference. If alternative analytical isotopes having higher natural abundance are selected to achieve greater sensitivity, an isobaric interference may occur. All data obtained under such conditions must be corrected by measuring the signal from another isotope of the interfering element and subtracting the appropriate signal ratio from the isotope of interest.

7.1.2. Abundance sensitivity – A property defining the degree to which the wings of a mass peak contribute to adjacent masses. The abundance sensitivity is affected by ion energy and quadrupole operating pressure. Wing overlap interferences may result when a small ion peak is being measured adjacent to a large one. The 8900 ICP-MS will recognize the potential for this interference and adjust the spectrometer resolution to minimize the interference. The results of the tuning will be documented on the Tune Summary Report. An example of this is the shadowing effect of Be mass 9 over the background mass 8.5.

7.1.3. Isobaric polyatomic ion interference – Caused by ions consisting of more than one atom that have the same nominal mass-to-charge ratio as the isotope of interest and cannot be resolved by the mass spectrometer. These ions commonly form in the plasma or interface system from support gases or sample components. The instrument software identifies most of the common causes of interference of this type for the analytes affected and inter-element correction equations are entered for each interference. Such interferences must be recognized in method development, and when they cannot be avoided by the selection of alternative analytical isotopes, appropriate corrections must be made to the data. Establish equations for the correction of data at the time of the analytical run sequence, as the polyatomic ion interference will be highly dependent on the sample matrix and chosen instrument conditions. Reduce the common 82 Kr interference that affects the determination of selenium with the use of high purity krypton free argon. In addition, the use of alternate isotopes for certain elements can monitor this type of interference.

7.1.4. Physical Interference – Associated with the physical processes that govern the transport of sample into the plasma, sample conversion processes in the plasma, and the transmission of ions through the plasma-mass

spectrometer interface. This type of interference may result in differences between instrument responses for the sample and the calibration standards. Physical interference may occur in the transfer of solution to the nebulizer (e.g. viscosity effects), at the point of aerosol formation and transport to the plasma (e.g. surface tension), or during excitation and ionization processes within the plasma itself. High levels of dissolved solids in the sample may contribute deposits of material on the skimmer cones reducing the effective diameter of the orifice and therefore ion transmission. Dissolved solids levels in samples not exceeding 0.2% (w/v) reduce such effects. Effective use of internal standardization compensates for many physical interference effects. Internal standards ideally should have similar analytical behavior to the elements being determined. Use of matrix matching of acid concentrations in samples and standards in this method helps to minimize most transport effects.

7.1.5. Memory interference – Results when isotopes of elements in a previous sample contribute to the signals measured in a new sample. Memory effects can result from sample deposition in the sample introduction lines, on the sampler, skimmer or hyper cones and from the buildup of sample material in the plasma torch and spray chamber. The site where these effects occur is dependent on the element and is minimized by flushing the system with a rinse blank between samples. Recognize the possibility of memory interference within an analytical run and add suitable rinse times to reduce them. Estimate the rinse times necessary for each analyte prior to analysis. Achieve adequate rinse times by aspirating a standard containing analytes corresponding to ten times the upper end of the linear range for a normal sample analysis period, followed by analysis of a rinse blank at designated intervals. Note the length of time required to reduce analyte signals to within a factor of ten of the method detection limits. Examine the analyte concentration in the previous sample to identify if this was high. If the previous sample showed a high concentration of the analyte in question, suspect a memory interference. Reanalyze the sample after a longer rinse period. In the determination of mercury, which suffers from memory effects, the addition of 200 µg/L of gold will effectively rinse out 5 µg/L of mercury in approximately two minutes. Higher concentrations will require a longer rinse time.

8.0 Safety

8.1. Lab coats are mandatory for all personnel in a laboratory area. Gloves and safety glasses are mandatory for all personnel when working with samples and/or chemicals.

8.2. Treat each chemical used in this method as a potential health hazard. From this viewpoint, reduce exposure to these chemicals to the lowest possible level by whatever means available, (e.g. chemical fume hoods, protective aprons, lab coats, safety glasses, face shields, and gloves).

8.3. Safety Data Sheets (SDS) and Hazardous Substance Fact Sheets (HSFS), which provide detailed health & safety information on individual chemicals, are available for reference in the SDS PHEAL program.

8.4. Handle concentrated solutions of toxic, flammable, reactive, and corrosive chemicals safely. Use safety carriers when transporting in 2-liter or larger bottles. Handle chemicals in a fume hood using gloves. Wear a face shield and rubber apron when pouring from one container to another. Keep containers tightly closed. Protect from physical damage. Chemicals must be stored in a cool, dry, well-ventilated area away from sources of heat and incompatible chemicals.

8.5. Take appropriate precautions (e.g. chemical fume hood, protective apron, face shield and gloves) when handling samples to prevent exposure to potentially harmful chemicals that could be present in the samples.

8.6. Nitric Acid (HNO_3):

- Contact with combustible material may cause fire.
- Health Effects: Can cause severe irritation and burns to the eye, skin, nose and throat, leading to permanent eye damage, shortness of breath, and pulmonary edema
- Target Organ: Teeth
- Incompatibilities: bases, amines, alkali metals, copper, and organic materials

8.7. Hydrogen Peroxide (H_2O_2):
- May cause spontaneous combustion in the presence of flammable materials.
- Health Effects: Can cause severe irritation and is corrosive to the eye, skin, nose and throat, leading to permanent eye damage, shortness of breath, and pulmonary edema
- Target Organs: Skin, Respiratory Tract
- Incompatibilities: oxidizing agents, reducing agents, combustible materials, organic materials, metals, acids, alkalis.

8.8. The acidification of samples containing reactive materials may result in the release of toxic gases, such as cyanides or sulfides. Acidification of samples should be done in a fume hood.

8.9. It is the responsibility of the user of this method to comply with relevant disposal and waste regulations.

8.10. The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Regard each chemical as a potential health hazard. Exposure to these compounds should be as low as reasonably achievable. Each laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material data handling sheets should also be made available to all personnel involved in the chemical analysis. Specifically, concentrated nitric acid presents various hazards and are moderately toxic and extremely irritating to skin and mucus membranes. Use these reagents in a fume hood whenever possible and if eye or skin contact occurs, flush with large volumes of water. Always wear safety glasses or a shield for eye protection, protective clothing and observe proper mixing when working with these reagents.

8.11. Only view the plasma when wearing UV eye protection to prevent exposure to damaging ultraviolet emissions.

8.12. Take extra care when working in the radiation and CT labs as those labs work with dangerous materials.

9.0 Equipment, Supplies, and Maintenance

9.1. Inductively Coupled Plasma – Mass Spectrometer (ICP-MS), Agilent 8900 QQQ – in-house ID# is ICPMS (4). Note: Agilent 8900 Hardware and Software Manuals are in Room L-445 or in the help menu of the Masshunter software.

9.2. Agilent nebulizer, MicroMist, P.N. G3266-80005.

9.3. Agilent spray chamber, P.N. G3280-80008.

9.4. Agilent sampling cone, P.N. G3280-67036.

9.5. Agilent skimmer cone, P.N. G8400-67201.

9.6. Agilent platinum shield, P.N. G1833-65419.

9.7. Agilent torch bonnet, P.N. G1833-65421.

9.8. Agilent ICP-MS torch, P.N. G3280-80053.

9.9. Low-pressure, High Purity (99.99%) liquid argon is required with a tank output of 100 psi.

9.10. High Purity O_2 gas is required with an output of 10 psi. Regulator and tubing safe for O_2 gas.

9.11. Agilent sample tubing, white/white, SCP Science P.N. 022-033-009 or equivalent.

9.12. Agilent internal standard pump tubing, orange/blue, SCP Science P.N. 023-130-003 or equivalent.

9.13. Agilent drain pump tubing, yellow/blue, SCP Science P.N. 022-033-419 or equivalent.

9.14. Roughing pump oil, P.N. X3760-64004 with a drain pan, funnel and waste container.

9.15. PolyScience Recirculator and 1 L coolant (#WE01-6558) and PolyScience Chiller and 1 L coolant (#WE01-6558)

9.16. Low-pressure, High Purity (99.99%) liquid argon is required with a tank output of 70-120 psi.

9.17. A variable speed peristaltic pump is required to deliver both standard and sample solutions to the nebulizer.

9.18. Air displacement pipettes capable of delivering volumes ranging from 10 -10000 µL with an assortment of high-quality disposable pipette tips.

9.19. A computer capable of running Masshunter software, monitor, and printer.

9.20. A microwave digester able to achieve, control, and maintain 200°C for 10 minutes in each individual vessel without over-pressurizing.

9.21. Preventive Maintenance – All maintenance performed is recorded in the maintenance logbook located in L-445.

9.22. Change all peristaltic pump tubing after every 2-3 days of use, or as needed (See Section 14.2.3). Other circumstances that would require a need to change pump tubing would be the development of flat spots or after approximately eight hours of use. Firmly attach the drain tubing to the spray chamber to ensure that liquid flows smoothly through the pump. Keep the drain tubing clear of debris and empty the drain bottle as needed.

9.23. Rinse the sample introduction system thoroughly before and after each use of the instrument. The 8900 will perform the warm-up procedure automatically upon lighting the plasma and the analyst will put two rinses in at the end of each analytical batch.

9.24. Inspect the peristaltic pump rollers before each use to make sure they are clean and move freely. Clean the exterior of the pump with a cloth moistened with water if needed.

9.25. Before analysis, check that the solution flows freely through the nebulizer and that the sampling capillary is clean and in good condition. If clogged or wetting of the spray chamber is not uniform, then rinse the nebulizer system with 5% HNO_3 for 15 minutes followed by water and re-check. Change or clean the sampling capillary or probe if necessary.

9.26. Vacuum system – The Masshunter software will monitor total vacuum hours and recommend times to change the filter and the oil. If clogged, the cone orifice pressure is lower. If worn, the cone orifice pressure is high. This procedure gives an indication of the cone condition. If the daily performance check is not acceptable and there has been a vacuum pressure change, the cones made need to be cleaned or replaced. Cracked, hardened, brittle or used O-rings can also lead to unacceptable daily performance checks. Check and replace the O-rings as necessary prior to replacing the cones.

9.27. Pumps – Visually check the condition and level of the pump oil for the appropriate vacuum pumps. Check that the oil is not dirty and is at the proper level. The 8900 has an interface roughing pump and a turbo backing pump. Compare the appearance of the oil with a small sample of new oil. Change the oil if it has unusual color, is dark, contains particles, or appears dirty or turbid. Pump oil the color of tea is acceptable but will soon need to be changed. Pump oil the color of coffee needs to be changed. For further information and the procedure to change the oil, see the Masshunter help function.

9.28. Torch Assembly and RF Load Coil – Perform a weekly inspection of the torch and aerosol injector tube. Clean the torch when needed to remove accumulated deposits. Inspect the RF coil for any deformation or carbon build up. Replace the coil if there is any sign of pitting. The glassware should be clean, with no heavy deposits or signs of melting. If the analyst observes a significant drop (> 10%) in element intensity, consider replacing the torch assembly. If the torch needs replacement, see the Masshunter hardware guide. Torches, nebulizer chambers, and injectors can be cleaned by soaking in 10% HNO_3 or Citranox followed by rinsing with reagent water. A mild detergent may be added if necessary. Never sonicate these items as the glass will become compromised and brittle.

9.29. Clean or replace sampler and skimmer cones as needed. Deposits on the cones can be removed with gentle wiping with a Kimwipe and 2% HNO_3 or sonicating in a Citranox bath. Allow to dry before reassembling. If cone orifice is heavily pitted, then replace.

10.0 Reagents and Standards

10.1. Ultra-pure Nitric Acid (HNO_3), concentrated.

10.2. Hydrogen Peroxide, 30%.

10.3. Reagent Water – Obtain reagent 18.2 MΩ water from the Barnstead Reagent Diamond point-of-use water purification system unit in Room L-445B (in-house # WS-2).

10.4. Calibration Stock Standards - Purchased from a reputable commercial source. Standard Certificate of Analysis forms are stored in Element.

10.5. The multi-element stock standard solution is a mix of the following: Aluminum (Al), Antimony (Sb), Arsenic (As), Barium (Ba), Beryllium (Be), Cadmium (Cd), Chromium (Cr), Cobalt (Co), Copper (Cu), Lead (Pb), Manganese (Mn), Molybdenum (Mo), Nickel (Ni), Selenium (Se), Silver (Ag), Thallium (Tl), Thorium (Th), Uranium (U), Vanadium (V) and Zinc (Zn) at a concentration of 20 mg/L.

10.6. The single element stock solutions for As, Cd, Cr, Pb, Fe, Ni, Mn, Se, Zn, Ge, In, and Au are at a concentration of 1,000 mg/L.

10.7. The single element stock for Hg is at a concentration of 20 mg/L.

10.8. The single element stock solutions for Fe and Sc are at a concentration of 10,000 mg/L.

10.9. The ICP-MS calibration standards are prepared bimonthly or as needed from these stock solutions or their intermediates. Prepare standards as follows: Into Class A Digitubes labeled BLK through S4 with the analysts initials and preparation and expiration dates, add approximately 20 mL of reagent water, 7 mL of trace metals grade HNO_3, 10 µL of Au stock (10.7), and the appropriate amount of the multi-element stock, bring to 50 mL final volume, cap, and thoroughly mix. See Appendix B for standard preparation.

10.10. Quality Control Samples (LCV and HCV)

10.10.1. The LCV and HCV are made from a reputable source different from the vendor used for the calibration standards. The LCV and HCV are custom made with the same elements (10.6) at a concentration of 20 mg/L.

10.10.2. Single element stock solution for Hg at a concentration of 20 mg/L from a different source than 10.8.

10.10.3. Single element stock solution for Fe at a concentration of 10,000 mg/L from a different source than 10.9.

10.10.4. The preparation of the LCV and HCV is the same as 10.10. See Appendix B for preparation.

10.11. Internal Standards – Scandium (Sc), Indium (In), Bismuth (Bi), and Terbium (Tb) are the elements utilized for internal standardization during the analysis. Introduce the internal standard solution into all blanks, calibration standards, quality controls, and samples via a second channel of the peristaltic pump and a mixing manifold. The prepared internal standard solution has a concentration of 0.5 ppm of Sc, In, Bi, and Tb in 1% HNO_3. Monitor the internal standard response throughout the sample set analyzed. See Section 12.7 for monitoring criteria and Appendix B for preparation.

10.12. Daily Performance Check Solutions (14.6) – Prepared in-house for use with the 8900 for Torch Axis, EM, Plasma Correction, Standard Lens Tune, Resolution/Axis, and Performance Report optimizations. The 8900 daily performance check solution is Ce, Co, Li, Mg, Tl, and Y at 1 µg/L.

11.0 Sample Collection, Preservation, Shipment and Storage

11.1. Collect samples in new, single use plastic bags only.

11.2. The volume collected must be sufficient to ensure a representative sample and to allow for replicate analysis. An individual analysis may be completed with a minimum of 0.1 g; however, to allow for repeat analysis, dilutions, and quality control procedures, ECLS requires that a minimum of 0.5 g be collected.

11.3. Samples are to be received and maintained in accordance with the sample receiving SOP for the Medicinal Marijuana Program.

11.4. There is no established holding time for metals analyses in marijuana. Every effort is made to prepare and analyze these samples as soon as possible.

11.5. Chain-of-custody is maintained for all marijuana samples. All samples are stored at room temperature in a designated safe in the CT laboratory. The sample aliquots given to the Metals Laboratory must be acidified by the CT laboratory before the transfer is made. This process destroys the regulated ingredient in marijuana, rendering it no longer a controlled substance.

12.0 Quality Control

12.1. Blanks - Four types of blanks are required for this method. A calibration blank is used to establish the analytical calibration. The laboratory reagent blank (BLK) is used to assess possible contamination from the sample preparation procedure and to assess spectral background. The blank spike (BS) is used to assess routine laboratory performance, and a rinse blank is an instrument automated task that is used to flush the instrument sample introduction system between analyses.

12.1.1. The Calibration Blank consists of HNO_3 and Au in reagent water. Analyze the calibration blank during the calibration of the instrument and following the CV in the analysis run. The calibration blank acceptance limit is ± the RL of each analyte.

12.1.2. The BLK contains all the reagents in the same volumes as used in processing the samples. The BLK is carried through the same preparation scheme as the samples, including sample digestion. Analyze a BLK once every 20 or fewer samples. Use of the BLK data assesses contamination from the laboratory environment. Suspect laboratory or reagent contamination if BLK values exceed the RL. When BLK values are above the RL of the analyte, the batch will be re-prepared for that analyte and subsequently re-analyzed. Analyze the BLK following the analysis of the HCV.

12.1.3. The BS is a spike of the BLK with all method analytes at a known concentration. Analyze a BS once every 20 or fewer samples and spike at the following concentrations: 10 ppb for Hg, 20 ppb for As, Cd, Cr, and Pb, 60 ppb Se, 300 ppb for Ni and Mn, and 1000 ppb Fe. Carry the BS through the same preparation scheme as the samples, including sample digestion. Analyze the BS following the analysis of the BLK. If the recovery of any analyte falls outside the required control limits of 75 – 125 %, that analyte is judged to be out of control. The batch of samples associated with that BS must be re-prepared and/or re-analyzed for the analyte in question. Results of the BS percent recovery are available to produce control charts for all analytes for a select date range dates through Element.

12.1.4. The rinse blank is prepared by adding 40 mL of trace metals HNO_3, 40 mL of trace metals HCl, 200 µL of 20% Triton-X, and 400 µL Au stock to 2 L of reagent water and mixing thoroughly. Perform this preparation directly in a 2-Liter Teflon bottle.

12.2. Instrument Calibration Verification – To verify the calibration throughout the run, analyze S3 and CB immediately following the calibration, after every 10 samples, and at the end of the run for the Calibration Verification (CV). The CV for all analytes within the standard solutions must be within ± 25% of the calibration standard analyzed. If not confirmed within ± 25%, reanalyze the 10 samples prior to the failure after a recalibration is performed in a subsequent analysis. If sample matrix is responsible for the calibration drift, analyze the previous 10 samples in groups of five or fewer between calibration checks to minimize the calibration drift.

12.3. Quality Control Samples (LCV and HCV) – Analyze the LCV and HCV for daily verification of the accuracy of calibration standards and instrument performance. Obtain the QC stock from a second source, different from the standard stock solutions source, and prepared in the same acid mixture as the calibration standards. Analyze the LCV and HCV at the beginning of the run following the analysis of the calibration verification (12.2) and RLC (12.6). The LCV is analyzed at a concentration of 5 µg/L for Cr, Mn, Ni, Zn, As, Se, Cd, Hg, and Pb, 50 µg/L for Fe. The HCV is analyzed at a concentration of 100 µg/L for Cr, Mn, Ni, Zn, As, Se, Cd, and Pb, 20 µg/L for Hg, and 1000 µg/L for Fe. The acceptance limits are ±25%.

12.4. Laboratory Duplicate (DUP) – One sample is prepared and analyzed in duplicate for every batch of 20 samples or fewer. Carry the sample and its duplicate through the entire analytical process, treating them as separate samples. Analyze the DUP immediately following the original sample (which is the first sample analyzed in the batch of 20). The acceptance limits for samples by this method are ≤ 30% Relative Percent Difference (RPD) if

both sample concentrations are greater than the Reporting Level (RL). In instances where one or both samples are below the RL, use a control limit of ± the RL.

12.5. Matrix Spike (MS) and Matrix Spike Duplicate (MSD) – These are matrix spikes that are aliquots of a sample spiked with a known concentration of targeted analytes at the same level as the BS (12.1.3). These spiked samples undergo the same sample preparation, digestion procedure, and analysis as the non-spiked samples in the associated batch. An MS and MSD are prepared for every batch of 20 or fewer samples. Use the same sample for the DUP as for the MS and MSD. Perform spike recovery calculations using the result of the original sample analysis. When the initial sample concentration is below the RL, zero will be used in the calculation of the percent recovery. The percent recovery acceptance criterion is 70 to 130%. The relative percent difference (RPD) acceptance criterion between the MS and MSD is ≤30%. Recovery calculations are not required if the concentration added is less than 30% of the sample background concentration.

12.6. Reporting Level Check (RLC) – Analyze an RLC for daily verification of the RL. Analyze the RLC following the calibration verification routine outlined in Section 12.2 and at the end of the run. See Appendix B for RLC preparation and concentration. The acceptance limits are ± 50% from the true value.

12.7. Internal Standard Response – The analyst must monitor the internal standard response throughout the run. The information is used to detect potential problems caused by mass dependent drift, errors incurred in adding the internal standards or increases in the individual internal standards caused by background contributions from the sample.

13.0 Calibration and Standardization

13.1. Calibration is performed daily (24-hour period) for all elements analyzed by this method. Outlined in Appendix B is the standard preparation and concentrations. The calibration consists of a blank and four standards (BLK-S4). The computer software is programmed with the calibration information (i.e. concentrations and autosampler position). Place the calibration blank in the auto-sampler rack, followed by the calibration standards. The software creates a calibration based on the responses observed in the calibration standards.

13.2. Electronic Pipette Calibration Check – Perform a calibration check on all electronic pipettes used in this method quarterly as per Appendix 21 of the Lab QA Manual. File completed pipette calibration check forms in Room L-440 and forward a copy to OQA.

14.0 Analytical Procedure

14.1. Sample Preparation – All samples will be digested per ELCS-I-MD-1 prior to analysis by this procedure.

14.1.1. Obtain a worklist by querying the Element LIMS system. A sample preparation list is prepared from the worklist and recorded in the sample preparation logbook. Include one each of a DUP, MS, MSD, BLK and BS for each set of twenty samples or fewer.

14.1.2. All samples require chain-of-custody. These samples are stored in a safe in the CT laboratory until they are to be analyzed. A representative from the CT laboratory will dispense approximately 0.1 g of each sample into quartz microwave digestion tubes followed by 7 mL of trace metals HNO_3. This serves to destroy any cannabinoids rendering the sample a non-controlled substance and performs the first step in the digestion process. The exact mass provided is supplied to the metals analyst by the CT laboratory representative for use in calculating sample results once analyses are completed.

14.1.3. Before use, thoroughly rinse sample digestion tubes with reagent water. Label the tubes for the associated QC, which includes the DUP, MS, MSD, BLK, and BS.

14.1.4. All samples and batch QC are processed according to the digestion procedure outlined in ECLS-I-MD-1 prior to analysis.

14.1.5. Summary of microwave digestion: the vessels are weighed prior to being given to the CT Lab. ~0.1 g of sample is added to its respective vessel. 7 mL of HNO_3 is added to each vessel prior to the sample leaving the CT Lab. Add 1 mL 30% H_2O_2 and 10 µL of 1000 ppm Au to each tube. The BS, MS, and MSD are spiked,

and the vessels are sealed and placed in the digestion rack. The digestion protocol is a 10-minute ramp to 200°C, a 10-minute hold at 200°C, and a 30-minute cooling step. Samples are brought to 50 g with reagent water and the final mass is recorded.

14.2. ICP-MS Instrument Set-up:

14.2.1. Instrument Set-up – All methods are pre-programmed with all the necessary information to analyze for the selected parameters.

14.2.2. Inspect the condition of the torch, injector, and the load coil. If the torch has severe deposit discoloration, clean or replace it. If the injector is clogged or has deposit, clean or replace it. Replace any torch or injector if damaged in any way. Check that the load coil is clean, dry, and free of pitting. Never sonicate glassware.

14.2.3. Check the condition of the sample and skimmer cones for severe degradation due to deposits or corrosion. If there are signs of degradation or carbon deposits, clean or change the cones. Take note of the analyzer pressure on the Instrument window. If the analyzer pressure is high, suspect clogged cones. If the analyzer pressure is low, suspect enlarged or damaged cone orifices and replace.

14.2.4. Cleaning sample and skimmer cones – If ICP-MS plasma is on, stop plasma using instrument control panel or the plasma button on the instrument and allow chamber to cool for several minutes. Open outer instrument lid and open chamber. Use cone tools to remove sample and skimmer cones. Wet a Kimwipe with reagent water and gently clean by wiping off residue from surface. Avoid contact with the fragile tips of the cones. Sonicate cones in a solution of reagent water and Citronox until they look clean. Rinse cones with reagent water and wipe off surface with a dry Kimwipe and spray with compressed air. Using cone tools, replace cones into position. Close the chamber and instrument lid, turn on plasma, and allow to plasma to stabilize before going through the tune process with the tune solution. Clean cones as necessary especially if initial performance check fails due to low counts and high oxide ratio.

14.2.5. Replace damaged peristaltic pump tubing. Clip the fasteners onto the tubing stops on the peristaltic pump and center the tubing in the middle of the clamp levers. Engage the tubing clamps for each channel and swing the clamp levers over to apply tension to the clamps. The tubing to install for the 8900 is white/white for the sampling, orange/blue for the internal standard, and blue/yellow for the waste. Verify appropriate loop is correctly attached to the fast autosampler mount.

14.2.6. Ensure that there is sufficient argon available at the correct pressure of 100 psi. Ensure there is sufficient oxygen available at the correct pressure 10 psi.

14.2.7. Check the level of coolant in the chiller and turn on if not already on.

14.2.8. Check that the oil level is adequate and the color of the oil in the roughing pumps is honey brown. If the oil has become a coffee color, change the oil before proceeding. Never add oil to the pumps, drain completely and change.

14.2.9. Open the Masshunter software if it is not already open. Select the Instrument icon on the toolbar and then click the Front Panel tab to open the control panel.

14.2.10. If necessary, start the vacuum by clicking the down arrow next to the Hardware icon and selecting Vacuum On. Upon achieving sufficient vacuum, the vacuum indicator in the top right corner of the screen will turn green. The plasma is now ready to be lit.

14.2.11. To light the plasma, click the arrow to the right of the Plasma button and select the ‘Plasma On’ button to ignite the plasma. A dialog box will pop up prompting the analyst that the plasma on sequence will take place. To edit the plasma on sequence, click the ‘Plasma’ button and check or uncheck boxes as necessary. Otherwise, click ‘OK’ on the dialog box to begin the sequence. The peristaltic pump will start automatically when a plasma has been achieved. The instrument will warm up for about twenty minutes and begin the optimization sequence automatically.

14.3. Instrument Optimization – The 8900 will go through an optimization sequence as part of the warm up. The

default optimization sequence is Torch Axis, EM, Plasma Correction, Standard Lens Tune, Resolution/Axis, and Performance Report. If necessary, switch the tune solution with the P/A Factor solution, uncheck the all optimizations under the Plasma button, check the P/A Factor optimization, and select Add to Queue. A copy of the performance report is saved in the run folder.

14.4. Calibration and Sample Analysis:

14.4.1. Click the small arrow next to the Batch icon, select Open Batch Folder, and select the most recent medicinal marijuana run. Save the batch as YYMMDD (ElementBatch#) MM.

14.4.2. Load the samples into the autosampler.

14.4.3. Click the Sample List tab and edit the sample list to match the bench sheet for the current run. Ensure the sample names and file names match and ensure the vial #s match the sample locations in the autosampler. Save the batch.

14.4.4. Click the Add to Queue to begin analyzing samples. All samples are flagged if the RSDs are too high which may indicate issues with the run. Issues with RSDs can indicate old tubing, dirty cones, dirty nebulizer or spray chamber, or contamination from the prep.

14.4.5. Click the Overwrite if Data File name is same if necessary and click Plasma Off at End if the analyst will not be present when the run is finished.

14.4.6. Click the arrow next to the Plasma button and select Plasma Off after run is finished. Unclamp the tubing from the peristaltic pump and cap all samples for disposal.

14.5. Shutting down Agilent 8900 completely and restarting the instrument in the case of a power outage or major instrument repair:

14.5.1. To shut down: click the small arrow next to the Hardware button and select Vacuum Off. Wait about ten minutes (or until the LED on the instrument stops flashing) and close the Masshunter software. Press the power button on the front of the 8900 to shut off the instrument.

14.5.2. To power on: Press the power button on the front of the 8900 to boot up the instrument. Wait a few minutes before opening the Masshunter software. Click the small arrow next to the Hardware button and select Vacuum On. The plasma can be lit once the LED stops flashing.

14.6. Analyst's Data Review:

14.6.1. The analyst reviews all data and verifies that all QC meets the acceptance criteria as outlined in the SOP.

14.6.2. If the QC meets all the acceptance criteria, the analyst signs the Data Report and gives it to the Metals Laboratory supervisor or designee for final review and reporting.

14.7. Data Reporting:

14.7.1. Sample data are in units of µg/L or ppb for all elements analyzed by this method and are reported to three significant figures. The LIMS will convert these results to µg/g or ppm, accounting for the weight of sample used, and the final volume.

14.7.2. When a sample value is at, or above, RL it is reported with no qualification.

14.7.3. When a sample value is below the RL, it is reported as (Sample Value) ND for Not Detected.

14.7.4. Data Reporting and Reviewing – In the Online Data Analysis window, click the Report tab to bring up the drop-down menu and select LIMS and Configure LIMS Settings. Ensure output folder is "Medical Marijuana", change the file name to match "YYMMDD (ElementBatch#) MM", and click OK. Select all samples to be exported to Element, click the Report tab, select LIMS, and select Export Selected Samples. Copy the file that is created in the "Medical Marijuana" folder into the ICP-MS (4) folder on the Z drive. The data are now ready to be uploaded into Element using DataTool.

14.7.5. The analyst uploads the data into Element using DataTool. After verifying the results, the analyst updates

the status to "Analyzed".

14.8. The supervisor will review the run documentation to ensure it meets all the QC requirements as outlined in the SOP (Section 18). The items for review are as follows: all CV, QCS (LCV and HCV), RLC, BLK, BS, DUP, MS, MSD and Internal Standard recoveries. Consider an analytical run valid when all QC items meet the assigned acceptance limits.

15.0 Calculations

15.1. Calibration – The Masshunter software provided with the instrument calculates the calibration based on the response from each standard. The software provides a tabular listing of the calibration standard for the response factor and the associated theoretical and calculated concentrations. Print the calibration summary to include with the report.

15.2. The Masshunter software performs the response factor calculation. See the manufacturer hardware manuals for the details behind calculating the response factor.

15.3. Percent (%) Recovery Calculation – BS, MS, and MSD – The software performs this calculation and it is contained in the raw data.

$$\% \text{ Recovery} = \frac{(R1 - R2)}{C} x100 \text{ where:}$$

R1 = Concentration of spiked sample in ppb
R2 = Concentration of original sample in ppb
C = Concentration of spike addition in ppb

15.4. Relative Percent Difference (RPD) – DUP, MS/MSD. Contained in the raw data, the software performs this calculation.

$$\text{RPD} = \text{absolute value} \{ - \frac{R1 - R2}{(R1 + R2)/2} x100 \} \text{ where:}$$

R1 = First Sample Value (original)
R2 = Second Sample Value (duplicate)

Note: The RPD is an absolute value, reported as a positive number.

16.0 Method Performance

16.1. Demonstration of Capability (DOC) – Prior to analyzing samples using this method the analyst must demonstrate their capability to perform the analysis. Perform the DOC at any time there is a change in instrument type, personnel or test method. The analyst must analyze four replicate Blank Spike samples at the same concentration. Analyze the DOC samples either concurrently or over a period of days. The acceptance criterion for the DOC sample will be ±25%. If all parameters meet the acceptance criteria, the analysis of actual samples may begin. If any one of the parameters does not meet the acceptance criterion, the performance is unacceptable for that parameter and the analysis must be repeated.

16.2. The raw data are maintained on file in room L-440.

17.0 Pollution Prevention

17.1. Sample Disposal –The residual sample digestate left in the sample tube may be safely disposed of through the building's sanitary sewer system by the analyst. Copious amounts of tap water are used to dilute the acidity of the residual digestates during disposal. Spiked blanks, spiked samples, and samples high in hazardous analytes are to be poured into the Hazardous Waste container under the hood in Room L-445.

17.2. Standard Disposal – External standards, QCs, and other laboratory spiked samples are to be poured into the Hazardous Waste container under the hood in Room L-445.

17.3. Kits for spill clean-up are available in Room L-445. Kits available are for clean-up of mercury, acids, and bases.

17.4. For information about pollution prevention that may be applicable to laboratories and research institutions consult "Less is Better: Laboratory Chemical Management for Waste Reduction, American Chemical Society's Department of Government Relations and Science Policy", 1155 16th Street NW, Washington D.C. 20036, (202) 872-4477.

18.0 Data Assessment and Acceptance Criteria for Quality Control Measures

18.1. CV:

18.1.1. To verify instrument performance (12.2), S3 is analyzed immediately following the calibration, after every 10 samples, and at the end of the run.

18.1.2. The CV must be within ±25% of the true value. If not verified within these limits, the instrument must be recalibrated and any samples that are not bracketed by passing CVs must be reanalyzed.

18.2. CB:

18.2.1. The analysis of the CB is performed following the calibration, after every 10 samples, and at the end of the run. The CB must yield a result of +< RL, if not reanalyze the CB.

18.2.2. If reanalysis still yields a CB value +> RL, the analysis must stop, and the source of the problem must be identified. If the problem is not contamination of the element in question, the instrument may have to be recalibrated. Sample data are reportable up to the last acceptable CV and CB.

18.2.3. In some instances, certain elements will not meet the above criteria and must be reanalyzed. Only report results for elements that meet the acceptance criteria.

18.3. BLK:

18.3.1. Analyze a minimum of one BLK for every 20 samples in an analytical batch.

18.3.2. Evaluate the BLK using a limit of BLK +< RL.

18.3.3. If the BLK is outside this limit, verify that there was no laboratory contamination introduced by the apparatus. Verify that the laboratory water is of good quality. If the BLK does not meet this criterion, the associated analytical batch must be re-prepared and analyzed for the element in question.

18.4. BS:

18.4.1. Analyze a minimum of one BS containing all the elements of interest for every 20 samples in an analytical batch.

18.4.2. Evaluate the BS using the limits ±25% of the true value.

18.4.3. If the BS does not meet this criterion, the associated analytical batch must be re-prepared and analyzed for the element in question.

18.5. DUP:

18.5.1. Analyze a minimum of one DUP for every 20 samples in an analytical batch.

18.5.2. Calculate the relative percent difference as in Section 15.4.

18.5.3. If both initial and replicate results are above the RL, evaluate the duplicate difference using the limit ≤ 30% RPD.

18.5.4. If the duplicate RPD is within the limits, no further action is required.

18.5.5. If the duplicate RPD is not within the limits, the results associated with the affected sample, and any associated sample (from the same source or sampling episode), must be qualified with a “J”, to signify an estimated or approximate value. Optionally, the affected samples can be re-prepared and analyzed for the element in question.

18.5.6. If either the initial or the replicate result is above the RL, evaluate the difference using the limit RPD ± RL.

18.5.7. If both the initial and replicate results are below the RL, the RPD is not calculated and the DUP is not evaluated.

18.6. LCV and HCV:

18.6.1. Evaluate the LCV and HCV using the acceptance criterion of ±25% from the true value.

18.6.2. If the LCV and HCV are within the acceptance limits, no further action is required.

18.6.3. If either LCV or HCV are outside the acceptance limits, reanalyze all samples associated with the analytical batch for the analyte(s) in question.

18.7. MS and MSD:

18.7.1. Evaluate the MS and MSD using the following criteria: ± 30% recovery and ≤ 30% RPD.

18.7.2. Biased MS and MSD results do not necessarily invalidate the entire analytical run. If either the MS or the MSD results are outside the acceptance criteria and the remaining quality control in the run meets acceptance criteria, a matrix effect for this sample may be suspected. In this case, qualify the affected sample and any associated samples (samples from the same source or sampling event) by assigning a "J" for an estimated or approximate value. Optionally, the affected samples can be re-prepared and analyzed for the element in question. Recovery calculations are not required if the concentration added is less than 30% of the sample background concentration.

18.7.3. If additional quality control problems are evident, rerun the analysis or qualify the data.

18.8. RLC

18.8.1. Analyze the RLC following the instrument performance (12.2) routine at the beginning and end of the run. The RLC acceptance limit is ± 50 % of the true value.

18.8.2. If the RLC is outside the acceptance criterion, stop the analysis and perform a new instrument calibration or repeat the analysis for the element in question.

19.0 Corrective Action for Out of Control Analyses

19.1. The analyst is responsible for monitoring the outcome of quality control measures during the analytical process. Suspend sample runs producing unacceptable quality control. Identify the problem and take corrective action. The analyst should thoroughly document the problem and the corrective action(s) taken. The analyst should also notify their immediate supervisor of the nature of the problem and any corrective actions taken. The supervisor will determine if the analytical process may be resumed at that point or must be repeated.

19.2. Should the analyst be unable to identify and correct the problem, the analyst must immediately notify the supervisor. The supervisor will assist in identifying and correcting the problem. Resume or repeat the analysis at the discretion of the supervisor. Thoroughly document the problem and corrective action.

19.3. Notify the QAO should the supervisor be unable to identify and correct the problem. Once the QAO is involved, suspension of the analysis is necessary until performing a corrective action. The QAO notifies any clients affected by the suspension of the analysis and requests that they refrain from submitting any samples requesting the analysis in question until they are notified to resume. Once a corrective action is made, the QAO may choose to require the analysis of a quality control sample to verify that the corrective action was successful. When the QAO is satisfied that the appropriate corrective action(s) have been taken and properly documented, the analyst and supervisor are notified that the analysis may be resumed.

19.4. When evaluating quality control measures, one must consider the following factors and corrective actions:

19.4.1. The conditions of the pump tubing: If it is crimped or flattened, change the tubing.

19.4.2. Sample introduction system: Rinse thoroughly with rinse solution or 20% HNO_3.

19.4.3. Cleanliness of the sampling capillary probe: Clean or replace the capillary probe.

19.4.4. A sample matrix may cause carryover effects. Rinse system and analyze a series of blanks to ascertain and

rectify the problem.

19.4.5. Degradation of the LCV or HCV controls: Re-prepare the QC sample in question. If all pre-digestion spikes (i.e. MS, MSD, and BS) were out of control, the most likely reason would be degradation of the intermediate or stock solution or improper spiking technique. Investigate and reanalyze or re-prepare the batch, if necessary.

20.0 Contingencies for Handling Out of Control or Unacceptable Data

20.1. Isolated Problem

20.1.1. If quality control acceptance criteria are not met throughout the run, the run is rejected, the problem is diagnosed and corrected, and the entire run is repeated.

20.1.2. If only part of the run is unacceptable, that portion is repeated. The rest of the run can be reported if the sample values to be reported are bracketed by acceptable CVs and CBs and the quality control measures associated with the portion of the run to be reported have met the acceptance criteria.

20.2. Persistent or ongoing problem:

20.2.1. When the quality control measures for an analysis fail repeatedly, the QAO is informed. The supervisor or QAO may decide that the analysis is no longer in control and the method must be shut down until the problems are corrected.

20.2.2. The QAO informs the ECLS Director (or designee) who will inform laboratory clients that the analysis is being shut down.

21.0 Waste Management

21.1. Aim: To ensure that minimal harm will come to people, other organisms, and the environment from the disposal of waste laboratory chemicals.

21.2. Waste Removal Policy: Disposal of hazardous waste chemicals down the drain or adding them to mixed refuse for landfill burial is unacceptable. Hoods should not be used as a means of disposal for volatile chemicals. Disposal by recycling or chemical decontamination should be used when possible.

21.3. All outdated and waste chemicals must be removed from the laboratories and properly disposed through the Employee Health and Safety Program.

21.4. Waste Removal Procedure:

21.4.1. Arrangements to dispose of waste chemicals must be made through the Laboratory Safety Officer, Frank Gordon. He can be reached at (609) 406-6809.

21.4.2. Quantities of waste chemicals greater than 1 gallon or 10 pounds are not to be accumulated in the laboratory at any time.

21.4.3. Large quantities of liquid waste chemicals must be transferred to appropriate 55-gallon drums (in the hazardous waste cage). All drums are labeled. When transferring flammable liquids, ensure that the drum and the safety are bonded and grounded.

21.4.4. Small quantities of other chemicals should be tightly sealed, labeled, and deposited in appropriate (labeled) storage bins (in the hazardous waste cage). The hazardous waste removal company will not accept unidentified chemicals.

21.4.5. The chemical name, the amount being disposed, and initials of the employee must be entered in to the "Hazardous Waste Log" provided by the Lab Safety Officer.

22.0 References

22.1. U.S. Environmental Protection Agency, Method 200.8: “Determination of Trace Elements in Water and Wastes by Inductively Coupled Plasma-Mass Spectrometry”, Revision 5.4, 1994.

22.2. Quality Manual, NJDHSS, PHEL, ECLS, 2007.

22.3. "Employee Guide to Working Safely with Hazardous Materials", 2nd Edition, 1997 NJDHSS Health and Safety Program.
22.4. Biohazard Control Plan; Revised: February 1997, NJDHSS Health and Safety Program
22.5. EPA Method 3050B, "Acid Digection of Sediments, Sludges, and Soils.
22.6. EPA Method 3052, "Microwave Assisted Digestion of Siliceous and Organically Based Materials".
22.7. "Environmental Contaminants in Hops", R. Schmidy, P. Andegress, M. Biendl.
22.8. " TotalQuant Analysis of Teas and Wines by ICP-MS". Perkin Elmer.
22.9. "Advantages and limitations of the semi-quantitative operation mode of an inductively coupled plasma-mass spectrometer for multi-element analysis of wines", C. Marisa Almeida, M. Teresa S.D. Vasconcelos.

23.0 Appendices

23.1. Appendix A – Demonstration of Capability
23.2. Appendix B – Standard and QC Table