If this SOP is accessed electronically, it is an uncontrolled copy and will not be updated.

STANDARD OPERATING PROCEDURE

for

SAMPLE PREPARATION OF BIOLOGICAL TISSUE FOR METALS ANALYSIS BY GFAA, ICP-OES, AND ICP-MS

SOP No.: MET-TDIG
Revision: 3

Effective date: February 13, 2012

Approved by:	Supervisor	1/17/12 Date
	QA Manager	1-17-12 Date
	Laboratory Director	1/17/12 Date

COLUMBIA ANALYTICAL SERVICES, INC.
1317 South 13th Avenue
Kelso, Washington 98626

© Columbia Analytical Services, Inc. 2012

DOCUMENT CONTROL	
NUMBER:	
Initials:	Date:

If this SOP is accessed electronically, it is an uncontrolled copy and will not be updated.

SAMPLE PREPARATION OF BIOLOGICAL TISSUE FOR METALS ANALYSIS BY GFAA, ICP-OES, AND ICP-MS

1. SCOPE AND APPLICATION

This procedure describes techniques used for sample preparation and acid digestion of biological tissue samples. This procedure is applicable to the analysis of biological tissue for heavy metals. The procedure provides a convenient and efficient digestion/dissolution technique which allows for the simultaneous or sequential analysis of the sample for metals. The digestates may be analyzed by graphite furnace atomic absorption (GFAA), Inductively Coupled Plasma-Optical Emission Spectroscopy (ICP-OES), or Inductively Coupled Plasma-Mass Spectrometry (ICP-MS). The procedure includes, but is not restricted to, the metals listed in Table 1.

2. METHOD SUMMARY

A representative tissue sample is lyophilized, blended, then sub-sampled for conventional oven digestion. Oxidation is brought about by the use of concentrated nitric acid in a Teflon closed vessel. The digestate is then analyzed for metallic constituents by GFAA, ICP-OES, or ICP-MS methods

3. DEFINITIONS

3.1. **Batch -** A batch of samples is a group of environmental samples that are prepared and/or analyzed together as a unit with the same process and personnel using the same lot(s) of reagents. It is the basic unit for analytical quality control.

3.1.1. Preparation Batch - A preparation batch is composed of one to twenty field samples, all of the same matrix, with a maximum time between the start of processing of the first and last samples in the batch to be 24 hours.

3.2. **Method Blank (MB) -** The method blank or Preparation Blank (PB) is an artificial sample is designed to monitor the introduction of artifacts into the analytical process. The method blank is carried through the entire analytical procedure.

3.3. **Laboratory Control Samples (LCS)** – The LCS is laboratory created sample to which known amounts target analytes are added. The LCS is prepared and analyzed in exactly the same manner as the samples. The percent recovery is compared to established limits and assists in determining whether the batch is in control.

3.4. **Matrix Spike/Duplicate Matrix Spike (MS/DMS) -** In the matrix spike analysis, predetermined quantities of target analytes is added to a sample matrix prior to sample preparation and analysis. The purpose of the matrix spike is to evaluate the effects of the sample matrix on the method used for the analysis. Samples are split into duplicates, spiked, and analyzed as a MS/DMS pair. Percent recoveries are calculated for each of

If this SOP is accessed electronically, it is an uncontrolled copy and will not be updated.

the analytes detected. The relative percent difference (RPD) between the duplicate spikes (or samples) is calculated and used to assess analytical precision. The concentration of the spike should be at levels specified by a project analysis plan.

3.5. **Laboratory Duplicates (DUP)** – Duplicates are additional replicates of samples that are subjected to the same preparation and analytical scheme as the original sample. The relative percent difference (RPD) between the sample and its duplicate is calculated and used to assess analytical precision.

3.6. **Standard Reference Material (SRM)** – A material with specific certification criteria and is issued with a certificate or certificate of analysis that reports the results of its characterizations and provides information regarding the appropriate use(s) of the material. An SRM is prepared and used for three main purposes: (1) to help develop accurate methods of analysis; (2) to calibrate measurement systems used to facilitate exchange of goods, institute quality control, determine performance characteristics, or measure a property at the state-of-the-art limit; and (3) to ensure the long-term adequacy and integrity of measurement quality assurance programs.

4. INTERFERENCES

Refer to the determinative method for a discussion of interferences.

5. SAFETY

5.1. Follow all CAS safety practices as described in the CAS Safety Manual.

5.2. Each chemical compound or reagent should be treated as a potential health hazard. Exposure to these chemicals should be reduced to the lowest possible level. A reference file of material safety data sheets is available to all personnel involved in these analyses. CAS also maintains a file of OSHA regulations regarding the safe handling of the chemicals specified in this method.

5.3. Nitric Acid is extremely corrosive. Care should be taken while working with this chemical. Personal protective equipment shall include safety glasses (with side shields), gloves and a lab coat. Follow normal precautions as per the CAS Safety Manual.

6. SAMPLE, COLLECTION, PRESERVATION AND STORAGE

Samples are typically collected in plastic containers and iced or refrigerated at 4 ± 2°C from time of collection until preparation or analysis. Sample may be frozen at ≤-10ºC or as specified by project requirements.

7. APPARATUS AND EQUIPMENT

7.1. Lyophilizing apparatus, LABCONCO Model 7948040 freeze dryer.

If this SOP is accessed electronically, it is an uncontrolled copy and will not be updated.

7.2. Conventional laboratory oven capable of precise temperature control at 105°C.

7.3. Analytical balance capable of weighing to 1 mg.

7.4. 50 mL graduated poly tubes.

8. STANDARDS AND REAGENTS

8.1. Reagent grade chemicals shall be used in all tests. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lowering the accuracy of the determination. The preparation for all laboratory prepared reagents and solutions must be documented in a laboratory logbook. Refer to the SOP *Reagent/Standards Login and Tracking (ADM-RTL)* for the complete procedure and documentation requirements.

8.1.1. Reagent water - ASTM Type I or Type II water

8.1.2. Concentrated nitric acid.

8.2. Standards

8.2.1. Stock standard solutions may be purchased from a number of vendors. All reference standards, where possible, must be traceable to SI units or NIST certified reference materials. The vendor-assigned expiration date is used.

8.2.2. Metals spiking solutions: Five solutions are needed to prepare the matrix spiking standards: SS1, SS2, SS3, SS4, and SS5.

8.2.3. Follow the formulations laid out in Table 2. These solutions are prepared in acid rinsed Class A volumetric flasks using purchased custom mixed standards or 1000 ppm single analyte standards. Aliquots are made using acid rinsed Class A volumetric pipettes of the appropriate size.

8.2.3.1. SS1 (Al, Ag, Ba, Be, Cd, Co, Cr, Cu, Fe, Pb, Mn, Ni, Sb, V, and Zn): Fill a 1000 mL volumetric flask approximately half full with reagent water, add 50 mL of nitric acid and mix. Next add 100 mL of the custom mixed standard (CAS-CAL-14) purchased from “Inorganic Ventures”. In addition add 50 mL of 1000 ppm Antimony. Dilute to volume with reagent water, mix thoroughly and transfer to a 1000 mL Teflon bottle for storage. The solution expiration date is determined by the earliest expiration date of any single component in the solution.

If this SOP is accessed electronically, it is an uncontrolled copy and will not be updated.

8.2.3.2. SS2 (As, Cd, Pb, Se, Tl and Cu): Fill a 500 mL volumetric flask approximately half full with reagent water, add 25 mL of nitric acid and mix. Next add 2.0 mL each of 1000 ppm Arsenic, Cadmium, Lead, Selenium, Thallium and Copper. Dilute to volume with reagent water, mix thoroughly and transfer to a 500 mL Teflon bottle for storage. The solution expiration date is determined by the earliest expiration date of any single component in the solution.

8.2.3.3. SS3 (As, Se, and Tl): Fill a 500 mL volumetric flask approximately half full with reagent water, add 25 mL of nitric acid and mix. Next add 50 mL each of 1000 ppm Arsenic, Selenium, and Thallium. Dilute to volume with reagent water, mix thoroughly and transfer to a 500 mL Teflon bottle for storage. The solution expiration date is determined by the earliest expiration date of any single component in the solution.

8.2.3.4. SS4 (B, Mo): Fill a 500 mL volumetric flask approximately half full with reagent water, add 25 mL of nitric acid and mix. Next add 50 mL each of 1000 ppm Boron and Molybdenum. Dilute to volume with reagent water, mix thoroughly and transfer to a 500 mL Teflon bottle for storage. The solution's expiration date is determined by the earliest expiration date of any single component in the solution.

8.2.3.5. SS5 (K, Na, Mg, Ca): Fill a 1000 mL volumetric flask approximately half full with reagent water, add 10 mL of nitric acid and mix. Next add 100 mL of the custom mixed standard (CAS-CAL-14) purchased from "Inorganic Ventures". In addition add 20 mL of 10000 ppm Phosphorus, Sodium, Magnesium, Calcium. Dilute to volume with reagent water, mix thoroughly and transfer to a 1000 mL Teflon bottle for storage. The solution expiration date is determined by the earliest expiration date of any single component in the solution.

9. PREVENTIVE MAINTENANCE

9.1. All maintenance activities are recorded in a maintenance logbook kept for the freeze drying instrument. Pertinent information (serial numbers, instrument I.D., etc.) must be in the logbook. Maintenance entries should include date, symptom of problem, corrective actions, and description of maintenance, date, and name. The log should contain a reference to return to analytical control.

9.2. Centrifuge tubes must be thoroughly pre-cleaned with 1:4 HCl, and rinsed with DI water. All laboratory equipment used for trace metals analysis shall be stored in the clean room, and shall not be used for any other purpose.

9.3. Routine cleaning of the sample handling and digestion apparatus is necessary. Refer to the SOP for Metals Laboratory Glassware Cleaning.

10. RESPONSIBILITIES

10.1. It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Analysis and interpretation of the results are performed by personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP. This demonstration is in accordance with the training program of the laboratory. Final review and sign-off of the data is performed by the department supervisor/manager or designee.

10.2. It is the responsibility of the department supervisor/manager to document analyst training. Documenting method proficiency, as described in the SOP *Documentation of Training* (ADM-TRANDOC), is also the responsibility of the department supervisor/manager.

10.3. When new or unfamiliar situations develop, it is also the responsibility of the employee to immediately notify the section supervisor, project manager, or the laboratory director for quick resolution of all issues. Due to the nature of the work, it is important that all work be documented, department supervisors, project managers and when necessary, the client, be contacted immediately when questions arise.

11. PROCEDURE

11.1. Obtain a representative tissue sample that will yield ~ 300 mg of freeze-dried solids.

Note: This is designed to be a general guideline. Approximately 300 mg of dry sample is typically required to obtain the desired detection limits that often are necessary for tissue analysis.

11.2. Slice the sample into thin pieces prior to filling the drying vessel. Homogenize the sample before freeze drying.

11.3. Cap the vessel and freeze the sample in a conventional freezer. Note: Using the Labconco Model 7948040 freeze dryer, the samples do not need to be frozen prior to freeze drying.

11.4. When the sample is frozen, remove from freezer and follow the manufacturer's instructions for operation of the freezer dryer.

11.5. Blend the dry solids to obtain a homogeneous sample. The sample may be stored dry until digestion.

If this SOP is accessed electronically, it is an uncontrolled copy and will not be updated.

11.6. Sample Digestion

11.6.1. Transfer 300 mg of dried sample, weighed to the nearest 0.1 mg, to a 50 mL decomposition vessel.

11.6.2. Add 4.5mL concentrated nitric acid to the vessel.

11.6.3. Follow the manufacturer's instructions for the use of the decomposition vessels. Place the vessel in a conventional oven at 105°C for a minimum of 12 hours. If using the conventional oven option, monitor oven temperatures for each batch and record this data onto the appropriate benchsheet.

11.6.4. Cool the vessel, open to relieve pressure and vent the gases. If Antimony, Silver or Tin are target analytes, then add 10 ml of deionized water to the cooled vessel. Re-cap and tighten the vessel. Return to the oven for an additional hour.

11.6.5. Cool the vessel, open to relieve pressure and vent the gasses. Transfer the sample to a volumetric container and dilute to 30 ml. The sample is ready for analysis.

12. QA/QC REQUIREMENTS

12.1. Ongoing QC Samples required are described in the CAS-Kelso Quality Assurance Manual and in the SOP for Sample Batches. Additional QC Samples may be required in project specific quality assurance plans (QAPP). General QC Samples are:

12.1.1. Method Blank

12.1.1.1. A method blank is prepared in an empty digestion vessel and digested with every batch of 20 (or fewer) samples to demonstrate that there are no method interferences. If the method blank shows any hits above the reporting limit, corrective action must be taken. Corrective action includes recalculation, reanalysis, system cleaning, or re-extraction and reanalysis. For some project specific needs, exceptions may be noted and method blank results above the MRL may be reported for common lab contaminants.

12.1.2. Lab Control Sample (LCS)

12.1.2.1. The laboratory control sample is composed of both a SRM and a laboratory created sample. The laboratory created LCS is created by spiking the elements defined in the project plan into a digestion vessel. The LCS is designed to monitor the accuracy of the procedure.

12.1.2.2. Prepare one laboratory control samples (LCS) and a duplicate laboratory control sample (DLCS) with every batch of 20 (or fewer) samples whichever is more frequent.

12.1.2.3. Analyze a standard reference material (SRM) at 5% frequency or one per batch, whichever is more frequent. Standard Reference Material (SRM's) should be representative of the tissue sample being analyzed.

12.1.3. Sample duplicates

12.1.3.1. Samples analyzed by methods 6010, 6020 or GFAA require one sample duplicate at with every batch of 20 (or fewer) samples. Methods 200.7 and 200.8 require one sample duplicate at with every batch of 10 (or fewer) samples.

12.1.4. Matrix Spike

12.1.4.1. A matrix spike (MS) is prepared and analyzed with every batch of 20 (or fewer) samples if analyzed by methods 6010, 6020 or GFAA. Methods 200.7 and 200.8 require one sample duplicate at with every batch of 10 (or fewer) samples.

12.1.4.2. The MS is prepared by adding a known volume of the matrix spike solution to the sample and determining the spiked sample concentration. Spikes should be added directly to the dry sample. Spike solutions should be multi-element with analyte concentrations high enough to minimize volume added to sample.

13. REPORTING

13.1. Refer to the SOP for *Data Reporting and Report Generation* for reporting guidelines.

14. DATA REVIEW AND ASSESSMENT

14.1. Digestion data sheets, including weights and volumes used are completed and a batch lot number is assigned and attached to the data sheet. The Manufacturer's lot numbers for the reagents used are added to the digestion data sheet. (See appendix A)

14.2. Spiking sheets are completed including all spike data and volumes of spiking solutions used.

14.3. Following primary data interpretation and calculations, all data is reviewed by a secondary analyst. Following generation of the report, the report is also reviewed. Refer to the *SOP for Laboratory Data Review Process* for details. The person responsible for final review of the data report and/or data package should assess the overall validity and quality of

the results and provide any appropriate comments and information to the Project Chemist to inclusion in the report narrative.

15. CORRECTIVE ACTION

15.1. Refer to the SOP for *Corrective Action*. Personnel at all levels and positions in the laboratory are to be alert to identifying problems and nonconformities when errors, deficiencies, or out-of-control situations are detected.

15.1.1. Handling out-of-control or unacceptable data

On-the-spot corrective actions that are routinely made by analysts and result in acceptable analyses should be documented as normal operating procedures; specific documentation need be made other than notations in laboratory maintenance logbooks, runlogs, for example.

15.1.2. Documentation of nonconformity must be done using a Corrective Action Report (NCAR) when: a) corrective action is not taken or not possible b) corrective action fails to correct an out-of-control problem.

16. POLLUTION PREVENTION AND WASTE MANAGEMENT

16.1. It is the laboratory's practice to minimize the amount of solvents, acids, and reagents used to perform this method wherever feasibly possible. Standards are prepared in volumes consistent with methodology and only the amount needed for routine laboratory use is kept on site. The threat to the environment from solvents and/or reagents used in this method can be minimized when recycled or disposed of properly.

16.2. The laboratory will comply with all Federal, State, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions as specified in the CAS Environmental Health and Safety Manual.

17. TRAINING

17.1. Training outline (See the SOP for *Documentation of Training*)

17.1.1. Read and understand the SOP/Method. Also review the applicable MSDS for all reagents and standards used. Following these reviews, observe the procedure as performed by an experienced analyst at least three times.

17.1.2. The next training step is to assist in the procedure under the guidance of an experienced analyst. During this period, the trainee is expected to transition from a role of assisting, to performing the procedure with minimal oversight from an experienced analyst. This incremental step in training shall be documented at

each step by use of the Training Plan or a modified version of the Training Plan outlined in the SOP for *Documentation of Training.*

17.1.3. Perform an initial precision and recovery (IPR) study for tissue samples. Summaries of the IPR are reviewed and signed by the Technical Director. Along with the completed Training Plan, copies are forwarded to the employee's training file. For applicable tests, IPR studies should be performed in order to be equivalent to NELAC's Initial Demonstration of Capability.

17.2. Training is documented following the SOP *for Documentation of Training*.

NOTE: When the analyst training is documented by the supervisor on internal training documentation forms, the supervisor is acknowledging that the analyst has read and understands this SOP and that adequate training has been given to the analyst to competently perform the analysis independently.

18. REFERENCES

Recommended Guidelines for Measuring Metals in Puget Sound Marine Water, Sediment, and Tissue Samples; April 1997.

19. CHANGES SINCE THE LAST REVISION

19.1. Sec 3.1 is new
19.2. Sec 3.6 is new
19.3. Sec 8.1 is new
19.4. Sec 8.2 – updated standards and added detail to procedure for spiking standards prep
19.5. Sec 9.2 – is new
19.6. Sec 12.1.2 and 12.1.4– added QC for 200.7 & 200.8
19.7. Sec 12.1 – removed reference to DoD QSM and added detail to entire section

If this SOP is accessed electronically, it is an uncontrolled copy and will not be updated.

Table 1

Selected Elements and Analysis Procedures for Tissue Samples

Element	GFAA	ICP-OES	ICP-MS
Aluminum		√	√
Antimony		√	√
Arsenic	√		√
Barium		√	√
Beryllium		√	√
Boron		√	
Cadmium	√	√	√
Calcium		√	
Chromium		√	√
Cobalt		√	√
Copper		√	√
Iron		√	
Lead	√	√	√
Lithium		√	
Magnesium		√	
Manganese		√	√
Molybdenum		√	√
Nickel		√	√
Potassium		√	
Phosphorus		√	
Selenium	√		
Silver		√	√
Sodium		√	
Strontium		√	
Thallium	√	√	√
Tin		√	√
Vanadium		√	
Zinc		√	√

If this SOP is accessed electronically, it is an uncontrolled copy and will not be updated.

Table 2

METALS SPIKING SOLUTIONS CONCENTRATIONS FORM

Solution Name	Element	mLs of 1000ppm Solution	Final Volume	Solution Conc. mg/L	Concentration in the digest mg/L
SS1	HNO3	50.0	1000ml	-	
	Al	100*	1000ml	200	2
	Ag	100*	1000ml	5	0.05
	Ba	100*	1000ml	200	2
	Be	100*	1000ml	5	0.05
	Cd	100*	1000ml	5	0.05
	Co	100*	1000ml	50	0.5
	Cr	100*	1000ml	20	0.2
	Cu	100*	1000ml	25	0.25
	Fe	100*	1000ml	100	1
	Pb	100*	1000ml	50	0.5
	Mn	100*	1000ml	50	0.5
	Ni	100*	1000ml	50	0.5
	Sb	50	1000ml	50	0.5
	V	100*	1000ml	50	0.5
	Zn	100*	1000ml	50	0.5
SS2 GFAA SPIKE	HNO3	25.0	500ml	-	
	As	2.0	500ml	4	0.04
	Cd	2.0	500ml	4	0.04
	Pb	2.0	500ml	4	0.04
	Se	2.0	500ml	4	0.04
	Tl	2.0	500ml	4	0.04
	Cu	2.0	500ml	4	0.04
SS3	HNO3	25.0	500ml	-	
	As	50.0	500ml	100	1
	Se	50.0	500ml	100	1
	Tl	50.0	500ml	100	1
SS4	HNO3	25	500ml	-	
	B	50	500ml	100	1
	Mo	50	500ml	100	1
SS5	HNO3	10.0	200ml	-	
	K**	20	200ml	1000	10
	Na**	20	200ml	1000	10
	Mg**	20	200ml	1000	10
	Ca**	20	200ml	1000	10

Operation of the Freeze Drier Labconco 7948040

Operation Checklist

The following checklist should be followed after each use of the Stoppering Tray Dryer.

1. Wipe out the interior of the Stoppering Tray Dryer chamber with a paper towel to remove any moisture or debris.
2. Wipe the interior of the collector chamber (base unit)of the freeze dry system with a paper towel to remove any accumulated moisture.
3. Check the collector chamber drain hose on the freeze dry system (base unit) to ensure that the hose is free of moisture and that the drain plug is securely installed.
4. Using a paper towel, wipe the freeze dry system collector chamber (base unit) lid gasket and the Stoppering Tray Dryer door gasket to remove any dirt and contaminants that could cause a vacuum leak. Vacuum grease is not required on the door gasket or collector lid gasket to obtain proper vacuum seal.

Starting the Freeze Dry cycle

1. On the left side of the Stoppering Tray Dryer unit flip the black switch to the on position. On the left side of the base unit flip the black switch to the on position. On the base unit that the condensing coil is located push the manual button. The collector temperature must reach -40°C before the vacuum pump can be turned on.

2. Place the containers holding the pre-frozen samples onto the trays in the Stoppering Tray Dryer unit on top of the base unit. When closing the door pick up left hand bottom corner of door while closing. Turn the door handle all the way to the right. The door should be covering the entire black gasket attached to the Stoppering tray unit. Make sure that the vacuum release valve on Stoppering Tray Dryer unit is turned to the closed position. Press the mode (see attached diagram) button until the green light is on for Automatic on the Stoppering Tray Dryer unit. Press the run stop button on the Stoppering Tray Dryer unit P1 will appear in the display and the amber led button by the run stop button will light. Once the Stoppering Tray Dryer unit reaches -40°C it will hold at that temperature for one hour and forty eight minutes. After one hour and forty eight minutes the vacuum pressure in the display should decrease. If the vacuum is not decreasing press the vacuum on the collector unit button and open and close the door lifting up the bottom left hand corner of the door while closing. Turn the door handle all the way to the right. Press the vacuum button to restart the vacuum.

3. When the vacuum pressure (mBar) is the same on the Stoppering tray dryer and the base unit the samples have finished the freeze drying process (typically after 48 hours, depending on mass of tissue). Press the vacuum button on the collector unit to turn the vacuum pump off. Flip the switch on the side of the base unit to off. Flip the switch on the side of the Stoppering Tray Dryer unit to off. Open the chamber by moving the vacuum release control on the front of the

Stoppering Tray Dryer unit to the open position. When the sound of air through the back fill port is no longer audible, the chamber door is ready to open.

Alarms

A number of unusual events may occur during a lyophilization procedure that can adversely effect the operation of the Stoppering Tray Dryer. If an event occurs, the alarm indicator will flash and the beeper will sound. The beeper will automatically mute itself after one minute. The specific alarm can be identified observing the display. The following "out of specification" conditions will initiate an alarm:

System Temperature Variations
Once the system temperature has stabilized for 20 minutes, if the manual set point temperature or automatic hold temperature varies more than ±2°C as measured by the system temperature sensor, the Red Alarm indicator and word "TMP" on the display will flash until the end of the run.

Vacuum
Once the system vacuum is low and stabilize at a point where it changes less that 0.020mBar in 5 minutes if the vacuum changes more than 0.500mBar , the red Alarm indicator and the word "VAC" on the display will flash until the end of the run.

System Temperature Set Point
If during a ramp mode the system temperatures stabilize without reaching the set point temperature, the control will enter the next Hold mode. The Red Alarm indicator will flash and the program indicator "Px" on the display will flash until the end of the run.

Power Failure
If a power failure occurs while a run in is progress, the Red Alarm indicator and Run/Stop indicator will flash when the power in restored. Once the power is restored, the process will continue as programmed until completion. Pressing Run/Stop cancels the flashing warning.

If this SOP is accessed electronically, it is an uncontrolled copy and will not be updated.

Vacuum Pump

The oil in the Vacuum pump should be checked before every use. It must be changed if it is cloudy, shows particles or is discolored. The useful life of the vacuum pump oil can be extended if the vacuum pump is operated for an extended period of time after a freeze dry run.

Changing the Vacuum pump oil:

Pump oil is located in the ICPMS Instrument Laboratory.

1. Make sure the Vacuum pump is turned off
2. Remove the front cover of the bottom unit of the freeze drying unit by pressing up on the metal clasp located on the bottom of the front cover in the middle. Use a small screwdriver.
3. Slide the Pump out far enough to allow a 1 L plastic container to placed under the drain on the front of the Vacuum pump.
4. Remove the Grey cap on top of the Vacuum pump.
5. Remove the Grey plug on the bottom of the Vacuum pump.
6. After all of the used oil has drained out of the pump, pour a small amount of the unused oil into the vacuum pump with the drain plug removed to rinse out the oil reservoir. Place the used oil in the **Used Oil** container located in the ICPMS Instrumentation Laboratory.
7. Place the drain plug on the vacuum pump (hand tighten) fill the pump up with approx 400 mls of oil the visib should be 1 inch from the top mark on the outside of the vacuum pump. Screw the grey cap back onto the top vacuum pump (hand tighten).