



**FLORIDA
DEPARTMENT OF
ENVIRONMENTAL PROTECTION**

**QUALITY ASSURANCE
STANDARD OPERATING PROCEDURES
FOR SAMPLING AT FACILITIES PERMITTED UNDER
CHAPTER 62-737, FLORIDA ADMINISTRATIVE CODE**

**DIVISION OF WASTE MANAGEMENT
BUREAU OF SOLID AND HAZARDOUS WASTE
HAZARDOUS WASTE MANAGEMENT SECTION
NOVEMBER 14, 1997 REVISION
(REFORMATTED APRIL 15, 2010)**

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I. APPLICABILITY AND INTENT

The Department of Environmental Protection has compiled the following document, entitled “Quality Assurance Standard Operating Procedures for Sampling at Facilities Permitted Under Chapter 62-737, F.A.C.” (referred to as “Mercury SOPs” hereinafter) in order to establish standard operating procedures (SOPs) for sampling which address the quality assurance (QA) requirements for processes (operations) and closure of facilities recycling or storing mercury containing lamps or devices identified in Chapter 62-737, Florida Administrative Code (F.A.C.).

Sampling related to process (operations) are only one element of the quality control (QC) plan required in 62-737.800(4)(f), F.A.C. Facilities applying for a permit under Rules 62-737.800 must submit additional elements of a QC plan, such as checklists, logs or other elements which describe how the facility will monitor or evaluate the operational conditions or conformance to Chapter 62-737, F.A.C. These additional elements are specified in 62-737.900(2), F.A.C., “Mercury-Containing Lamp and Device Mercury Recovery and Mercury Reclamation Facility Permit Application Form and Instructions” and briefly summarized in the Appendix section of these Mercury SOPs for your convenience. These additional elements do not involve sampling, are facility specific and shall not be reviewed and approved by the Department’s Quality Assurance Section.

All facilities applying for a permit under Chapter 62-737, F.A.C., must submit a closure plan. Sampling during closure must follow the SOPs specified in these Mercury SOPs.

II. INTRODUCTION

Facilities identified as **Mercury Recovery Facilities** in 62-737.200(13), F.A.C. are required to demonstrate to the Department that their recovery process produces recovered separated glass and metal end caps that contain less than the levels of mercury specified in 62-737.840(3), F.A.C., if these items are to be shipped off site for further recycling or disposal or treatment other than mercury reclamation. Facilities identified as **Mercury Reclamation Facilities** in 62-737.200(12), F.A.C., are required by 62-737.860(4), F.A.C., to demonstrate to the Department that their reclamation process recovers 99% of the mercury introduced into the reclamation process. These two types of facilities must submit a QC plan which meets the requirements of 62-160.300(6), F.A.C., to ensure the integrity of their process (operation).

In accordance with 62-160.300(6), F.A.C., all sampling and analysis for facility process quality control and facility closure shall be conducted in a manner consistent with the Quality Assurance Category 2C requirements. All parties conducting sampling and analysis as required in Chapter 62-737, F.A.C., shall follow these **Mercury SOPs (Quality Assurance Standard Operating Procedures for Sampling at Facilities Permitted Under Chapter 62-737, F.A.C.)** or equivalent procedures as specified in 62-160.300(6), F.A.C. Subsequent analyses of collected samples must be performed by a

laboratory which has a current Department Approved Comprehensive Quality Assurance Plan for the laboratory analysis being performed

The rest of these Mercury SOPs are divided into two sections: Facility Process (Operational) Sampling and Closure Sampling. An Appendix in Section V contains additional Quality Control requirements and explanatory information.

III. FACILITY PROCESS (OPERATIONAL) SAMPLING

A. Chain of Custody (COC) Record

1. The possession or custody of samples must be traceable from the time they are obtained until the time the sample is received at the laboratory for analysis.
2. Sampler must label the sample container with a sample tag (usually an adhesive label). The label must be filled out using waterproof ink at the time of sample collection prior to placing it with other samples and must contain the following information:
 - a. Name of personnel collecting sample*
 - b. Unique sample identification number or code for each sample source and container
 - c. Time and date of the sample collection*
 - d. Location from which sample was taken and process name if facility has more than one process
 - e. Weight or other information identifying quantity of subsample (e.g., one level scoop)*
 - f. Chemical analysis which is to be performed on the sample

*Note: When daily subsamples are being collected in a single sample container in order to produce a “weekly sample composite of process operations” [62-737.840(3)(a)2, F.A.C.], the name of sampler, collection date/military time and quantity of each subsample must be recorded in a permanent daily sampling log.

3. Sampler must complete a COC record concurrent with the sampling event. Two copies of the chain of custody form must accompany the sample to the laboratory. Once the laboratory receives the samples and signs the COC, a copy must be sent to the sample collection organization. All parties accepting the custody of the samples, including the sampler, coordinator, transporter, laboratory custodian, etc. must provide signatures with date and time of sample receipt on the COC form. The COC record or form shall specifically contain the following information:
 - a. Unique sample identification number or code for each sample source and container
 - b. Sampling site name and address
 - c. Name of personnel collecting sample
 - d. Time and date of the sample collection when final weekly sample is filled

- e. Clear indication of number of sample containers
- f. Chemical analysis which is to be performed on the sample
- g. Appropriate places for signatures of sampler and all subsequent persons*
accepting custody and identification of common carriers
- h. Time of day and calendar date of all custody transfers
- I. Comments or remarks section, e.g., unusual ambient conditions

* Note: If samples are transported via integrated intermodal air/ground transporters of small packages, e.g., Federal Express, UPS, etc., the driver is not required to sign the COC. However, the parcel tracking number must be recorded on the COC by the sending party.

4. Copies of the COC records must be maintained for a period of at least 3 years in a loose-leaf binder or bound notebook.

B. Sampling Equipment

Samples shall be obtained using a plastic or stainless steel scoop or shovel. A spatula or similar device shall be used for raking off excess sample. Stainless steel is recommended.

C. Sample Containers

Sample containers may be glass or polyethylene with screw top lids. Polyethylene is recommended.

D. Sampling Equipment and Sample Container Decontamination (Cleaning)

1. Disassemble equipment if possible
2. Wash thoroughly with laboratory grade detergent (Alconox, Liquinox, or equivalent) and hot tap water using a brush to remove any particulate matter or surface film.
3. Rinse thoroughly with hot tap water.
4. Rinse thoroughly with 10% nitric acid (HNO₃) solution.* Note: 10% nitric acid solution is made by adding one part concentrated nitric acid to five parts deionized water.
5. Rinse thoroughly with deionized water and allow to air dry.
6. Wrap completely with plastic wrap to prevent contamination during storage or prior to use.*

*Note: Step #4 is not necessary for ferrous, e.g., stainless steel, sampling equipment. Step # 6 not necessary for screw top sample containers.

E. Sampling Methodology

1. Mercury Recovery Facilities [See Rule 62-737.840, F.A.C.]

Note: Facilities running more than one process line shall conduct required process (operational) sampling on each process line.

a. Daily samples

- (1) Samples may be collected in individual containers and composited later into the weekly composite sample or as equal daily aliquots into a single container which will then become the weekly composite sample. Samples may be collected in containers supplied by the facility or by the laboratory.
- (2) Minimum sample size for the weekly composite sample is 150 grams. A minimum sample size of 50 grams for daily samples is recommended in order to insure that the required 150 gram weekly composite sample is collected during weeks when equipment may not be operational every day. Smaller daily samples may be used as long as the required 150 gram weekly composite sample can be obtained from equal size daily samples. Note: On days when equipment is not operational, no daily sample shall be taken. Daily samples shall not be “doubled up”, i.e., two daily samples collected on the same day, to make up for daily samples when equipment is not operational. The weekly composite shall consist of equal aliquots of daily samples collected on days when equipment is operational. For example, if the equipment is operational for only three days during a particular week, that week’s weekly composite sample shall consist of equal aliquots of the daily samples collected during the three days when the equipment was operational.
- (3) It is recommended that daily samples be collected during maximum daily throughput and when equipment has been operating for at least 30 minutes. However, daily samples shall be collected if the equipment is operated for any period of time during that day. If the equipment is operational for 2 days or less in a particular week, no weekly composite sample shall be required for that week. However, the daily samples for those one or two days shall still be collected and used as part of the next weekly composite sample.
- (4) Representative Sampling
 - (a) Metal endcap assembly processed material: Daily sample material shall be collected using one of the following three procedures.
 - [1] Procedure 1: Representative sampling of endcap assembly processed material as the material exits the process
 - [a] Endcap assembly processed material as they exit the process may not be ground, milled, sized or sieve-separated in any way prior to daily sample collection unless such grinding, milling, sizing or sieve-separation is a part of the facility’s material processing operational procedures.

- [b] Lab aliquots must include portions of all components of the end cap assembly as contained in the 150 gram weekly composite sample required in the SOPs. For example, if the weekly 150 gram composite sample contains at least one whole endcap which includes aluminum outer cap material, pins and filaments, then the lab aliquot must be taken from a whole endcap which includes aluminum outer cap material, pins and filaments and must contain portions of the aluminum outer cap material, pins and filaments.
 - [c] Material shall be collected between the point at which the material exits the equipment and the bin or container which accumulates the material.
- [2] Procedure 2. Representative sampling of endcap assembly processed material after the material exits the process.
- [a] Grind to particle size which passes through a #4 sieve using grinder or mill. This particle size makes the individual parts of the endcap assembly, i.e., aluminum outer cap material, pins and filaments, indistinguishable from each other. Grinding may be performed by either the facility owner/operator or the testing laboratory.
 - [b] The lab aliquot shall be selected from the ground particles.
- [3] Procedure 3. An alternate representative sampling procedure for sampling endcap assembly processed material requested by a mercury recovery facility and approved by the DEP prior to implementation.
- [a] At a minimum, this procedure must include representative sampling of endcap assembly processed material. For example, if the weekly 150 gram composite sample contains at least one whole endcap which includes aluminum outer cap material, pins and filaments, then the lab aliquot must be taken from a whole endcap which includes aluminum outer cap material, pins and filaments and must contain portions of the aluminum outer cap material, pins and filaments.
 - [b] The procedure must comply with the SOPs unless the DEP approves equivalent alternate procedures not specified in the SOPs.
- (b) Other process material, e.g., glass. Material shall be collected between the point at which the material exits the equipment and the bin or container which accumulates the material.
- (5) Sampling equipment and containers shall be decontaminated according to specified protocols in Section III.D. Permanent (non-disposable) sampling equipment shall be decontaminated at least once per week with daily decontamination recommended.
 - (6) Overfill sampling equipment, e.g., scoop, with material collected. Rake off the excess material with a spatula or knife. Empty into precleaned sample container.
 - (7) Repeat Step 5 until the required quantity is obtained. Record subsample information in permanent daily sampling log (see Chain of Custody Record, III.A.2., Note) if daily samples are being collected in a single sample container as part of weekly composite sample.

b. Weekly composite samples

- (1) Place all previous daily subsamples (whether collected in separate containers or as equal daily aliquots collected in a single container) in a stainless steel, glass or aluminum (or plastic, unless sample is too hot) tray. The sample containers shall be cleaned according to protocols listed in the laboratory's current Department Approved Comprehensive Quality Assurance Plan.
- (2) The sample in the tray shall be homogenized thoroughly by alternately mixing, dividing, and remixing the sample.
- (3) After thorough mixing, transfer sufficient sample to the appropriate sample container(s) leaving minimal headspace.
- (4) Clean the outside and rim of the sample container to remove excess material.
- (5) Affix sample label and complete the COC forms.
- (6) Holding time shall not exceed 28 days.

2. Mercury Reclamation Facilities [See Rule 62-737.860, F.A.C.]

Note: Facilities running more than one process line shall conduct required process (operational) sampling on each process line. Facilities processing more than one process feed category, e.g., phosphor powder, inorganic mercury glassware, etc., must conduct process (operational) sampling on each process feed category as per facility permit conditions.

- a. Minimum sample size is 50 grams.
- b. Sampling equipment and containers shall be decontaminated according to specified protocols in Section III.D.
- c. Collect composites of randomly collected samples of material before processing and on the same material once it has been processed. Unprocessed materials should be collected in the same physical and chemical state in which they are fed into the process equipment, e.g., whole lamps or thermostats, broken lamps, mercury-containing phosphor powder, etc. It is recommended that unprocessed materials of homogeneous size and configuration be used for process sampling even though materials of heterogeneous size and configuration may be routinely mixed during normal processing. For example, mercury-containing ampoules should be of similar size, e.g., medium; mercury-containing arc tubes from High Intensity Discharge mercury-containing lamps should be from the same type of lamp, e.g., metal halide, and of similar size, e.g., small. Processed materials should be collected as closely as possible to the point at which the materials exit the equipment and, if possible, before the materials are deposited into the bin or container which accumulates the material. Processed materials shall be collected according to the categories needed to perform the required calculations to demonstrate 99% recovery of the mercury present in unprocessed materials (see Section 62-737.860(4), F.A.C.). For example, if mercury containing glass ampoules removed from thermostats are being processed, separate samples of the processed materials of glass, metal, and mercury shall be collected.

Aliquots to be composited shall be thoroughly mixed following procedures in Section III.E.1.b., “Weekly Composite Samples.”

- d. After thorough mixing (if required), place or transfer the sample to the appropriate sample container(s) leaving minimal headspace. The sample containers shall be cleaned according to protocols listed in the laboratory’s current Department Approved Comprehensive Quality Assurance Plan.
- e. Clean the outside and rim of the sample container to remove excess material.
- f. Affix sample label and complete the chain-of-custody forms.
- g. Holding time shall not exceed 28 days.

IV. FACILITY CLOSURE

A. Notification

The Department District Office Waste Program Administrator must be notified in writing by certified mail that the facility is being closed at least 30 days prior to initiation of any closure activities.

B. Documentation

1. Field Log - A field log must be maintained for all facility closures and made available to the Department upon request. The information must be maintained for a period of at least 3 years in a loose-leaf binder or bound notebook. All entries should be made in the facility or in the field, not back at the office. Entries should be made in waterproof ink and all mistakes should have one line drawn through and initialed. The log should include the following information

a. General Information

- (1) Site name and address and FDEP facility permit number
- (2) Dates and military times of all closure activities
- (3) Names of all personnel on-site and company affiliation
- (4) Ambient conditions, e.g., temperature and humidity inside; weather outside.
- (5) Signature of sampler(s)
- (6) Site sketch indicating location of facility and sampling points

b. Sampling Information

- (1) Date and military time of sample collection
- (2) Specific description of sample location, e.g., process room wall, north edge of paved outdoor storage area, etc.
- (3) Type of sample, e.g., soil to depth of 15 cm, first wash down rinsate, vacuum residuals, etc.
- (4) Unique sample identification number or code for each sample source and container

2. Chain of Custody (COC) Record

- a. The possession or custody of samples must be traceable from the time they are obtained until final disposal of the sample. Sample custody is being followed if:
 - (1) Sample container is secured to prevent tampering or is placed in a designated, secured area, or
 - (2) Sample is in actual physical possession of the sampler.

- b. Sampler must label the sample container with a sample tag (usually an adhesive label). The label must be filled out using waterproof ink at the time of sample collection prior to placing it with other samples and must contain the following information:
 - (1) Unique sample identification number or code for each sample source and container
 - (2) Date and military time of sample collection
 - (3) Chemical analysis which is to be performed on the sample

- c. Sampler must complete a chain of custody record concurrent with the sampling event. Two copies of the chain of custody form must accompany the sample to the laboratory. Once the sample transporter signs out and receiver signs in, one copy must be retained by the laboratory and one retained by the transporter who will deliver it to the party collecting the sample. All parties except a common carrier accepting the custody of the samples, including the sampler, coordinator, transporter, laboratory custodian, etc. must provide signatures with date and time of sample receipt on the chain of custody form. The COC record or form shall specifically contain the following information:
 - (1) Unique sample identification number or code for each sample source and container
 - (2) Sampling site name and address
 - (3) Name of personnel collecting sample
 - (4) Time and date of the sample collection when final weekly sample is filled
 - (5) Clear indication of number of sample containers
 - (6) Chemical analysis which is to be performed on the sample
 - (7) Appropriate places for signatures of sampler and all subsequent persons* accepting custody and identification of common carriers
 - (8) Time of day and calendar date of all custody transfers
 - (9) Comments or remarks section, e.g., unusual ambient conditions

* Note: If samples are transported via integrated intermodal air/ground transporters of small packages, e.g., Federal Express, UPS, etc., the driver is not required to sign the COC. However, the parcel tracking number must be recorded on the COC by the sending party.

C. Sampling Equipment

All liquid residuals, solid residuals and soil sampling equipment (e.g., spoons, scoops, shovels, hand bucket augers, bombs and bailers) shall be constructed of stainless steel or teflon (or plastic, unless sample is too hot) shall be used. Sample mixing trays shall be stainless steel, glass, aluminum (or plastic, unless sample is too hot).

D. Sample Containers

Precleaned glass or polyethylene sample containers with screw tops shall be used. Sample container decontamination will follow the laboratory's current Department Approved Comprehensive Quality Assurance Plan.

E. Sampling Equipment and Sample Container Decontamination (Cleaning)

1. Disassemble equipment if possible
2. Wash thoroughly with reagent grade detergent (Alconox, Liquinox or equivalent) and hot tap water using a brush to remove any particulate matter or surface film.
3. Rinse thoroughly with hot tap water.
4. Rinse thoroughly with 10% nitric acid (HNO₃) solution* Note: 10% nitric acid solution is made by adding one part concentrated nitric acid to five parts deionized water.
5. Rinse thoroughly with deionized water and allow to air dry.
6. Wrap completely with plastic wrap to prevent contamination during storage or prior to use.

*Note: Step #4 is not necessary for ferrous, e.g., stainless steel, sampling equipment.

F. Sampling Methodology

Facility closure involves sampling and analysis of four (4) different types of materials: decontamination solid residuals, decontamination liquid residuals, surface wipes and soils.

1. Decontamination Solid Residuals

- a. General - For the purposes of this subsection, decontamination solid residuals refer to floor and equipment sweepings or vacuum residuals, PPE, wipes, or other solid materials resulting from decontamination of equipment or buildings.
- b. Minimum sample size is 200 grams.
- c. Sampling equipment shall be decontaminated according to specified protocols in Section IV.E

- d. Collect composites of randomly collected samples of residuals. The number of aliquots will vary depending upon the quantity of materials being analyzed and subject to the approval of the DEP project engineer.
- e. Sample collection, e.g., overfill scoop and rake off excess, record subsample information, and mixing procedures shall follow procedures listed in III.E.1. a. "Daily Samples" and III.E.1. b. "Weekly Composite Samples".
- f. After thorough mixing (if required), place or transfer the sample to the appropriate sample container(s) leaving minimal headspace.
- g. Clean the outside and rim of the sample container to remove excess material. Cap sample container.
- h. Affix sample label and complete the chain-of-custody forms.
- i. Holding time shall not exceed 28 days.

2. Decontamination Liquid Residuals

- a. General - For the purposes of this subsection, pits, ponds, and lagoons refer to any basin, pit, or open tank (lined or unlined) which contains or is suspected of containing unknown concentrated liquid, solid, or sludge chemical wastes
- b. Sampling Locations - Sampling locations within pits, ponds, and lagoons shall yield samples which are representative of that section, or of the entire pit, pond, or lagoon being sampled. All phases (floating solids, all liquid phases, and sludge) in the pit, pond, or lagoon shall be sampled.
- c. Safety - Because of the dangers involved with container sampling, the sampling of drums or other containers containing either unknown materials or known hazardous materials shall be considered a hazardous duty assignment. Additional information regarding container sampling is available in the draft EPA *Safety Manual for Hazardous Waste Site Inspections*, US EPA, Draft 1979. Safety procedures for container sampling will be in accordance with the Region IV *Field Health and Safety Manual*, US EPA, Region IV, 1990 Edition (or latest edition).
- d. All equipment shall be decontaminated according to specified protocols in Section IV.E
- e. Holding time shall not exceed 28 days. Water samples shall be preserved with nitric acid (HNO₃). Solid waste and concentrated waste samples have no preservative requirements. Soil samples must be preserved at 4° C.
- f. Sampling Equipment - The following equipment may be used for field use in collecting liquid waste samples from pits, ponds, and lagoons: sampling containers; sampling containers affixed to a piece of conduit pipe, stainless steel scoop affixed to a piece of conduit pipe with a scoop bracket, stainless steel spoon affixed to a conduit pipe, peristaltic pump and tubing arrangement and Bacon-Bomb samplers. For sludge sampling of pits, ponds, and lagoons, stainless steel scoops attached to conduit pipe, stainless steel push tubes and corers are available.

g. Sampling Techniques

(1) **Liquid Waste Sampling**

- (a) Minimum sample size is 100 ml.
- (b) If the sampling technique utilized requires multiple aliquots, or if the final sample will consist of aliquots from several different locations in the pit, pond, or lagoon, all aliquots should be placed into a large glass sample container or other suitable compositing container, and mixed thoroughly before containerization.
- (c) Phase Separation or Stratification of Container Contents
 - [1] When this condition occurs, or is suspected, care must be taken to insure that the sample collected is representative of the container contents. If only one layer or phase is sampled, this should be noted and taken into account when interpreting analytical results.
 - [2] Determine whether phases are present by using a peristaltic pump.
 - [a] The tubing attached to the pump is strapped to a piece of conduit pipe and slowly lowered to the bottom of the unit to be sampled.
 - [b] The pump discharge (and the intake tubing) is examined to determine if phases are present.
 - [3] If phases are present, collect a sample of all phases.
 - [a] The top liquid phase can be sampled by direct dipping with the sample container, or dipping with the sample container attached to the conduit pipe - either directly or by way of a fishing pole type arrangement, or dipping the sample with a stainless steel scoop attached directly to conduit pipe with a scoop bracket.
 - [b] Alternatively, all liquid phases can be sampled with a peristaltic pump and tubing arrangement with the tubing attached to a conduit pipe and held at the desired depth or with the Bacon-Bomb sampler opened at the desired depth. The Bacon-Bomb sampler can be operated directly from the banks of pits, ponds, and lagoons; from piers; or operated by way of a fishing pole type arrangement using a piece of conduit pipe.
 - [4] If phases are not present, samples may be composited by depth (i.e., collected throughout the entire depth of the container or at several different depths) to provide a representative sample. Samples shall not be composited across containers. Composite samples may be collected using a coliwasa sampler or a glass profile tube.
 - [a] Coliwasa Sampler
 - { 1 } The coliwasa sampler is a single use glass sampler, consisting of an outerglass tube with one end tapered, and a separate inner glass tube with a small bulb on one end.

- {2} Slowly lower the outer tube into the pit, pond, or lagoon tapered end first. This must be done slowly for two reasons. One, the drum may contain solid material which might break the tube and injure the sampler. Second, slowly lowering the tube allows the liquid phases in the pit, pond, or lagoon to stay in equilibrium with the coliwasa sampler, ensuring a representative sample.
- {3} Insert the inner glass tube (bulb end first) into the outer tube. This may be done very slowly after the outer tube is fully inserted into the pit, pond, or lagoon or the inner tube may be inserted prior to immersing the outer tube. In the latter case, the bulb tip of the outer tube must be pulled back several inches from the tapered end of the outer tube.
- {4} After both inner and outer tubes are inserted into the pit, pond, or lagoon to be sampled, press the inner tube bulb end gently against the tapered end of the outer tube, forming a seal.
- {5} Withdraw both tubes from the pit, pond, or lagoon and the ends of the tubes are held over the sample container.
- {6} Place the sample in the sample container. By manipulating the inner tube, the sampler can control the rate of flow of the sampled liquid into the sample container.

[b] Glass Profile Tube Sampler

- {1} Samples can also be collected using a four-foot length of glass tube with a ½-inch or less inside diameter
- {2} Insert the tube into the opening of the pit, pond, or lagoon as far as possible.
- {3} Seal the open end either with the thumb or a rubber stopper to hold the sample in the tube while removing the tube from the container.
- {4} Place the sample in the appropriate container.
- {5} Repeat the procedure until an adequate amount of sample is collected.

[c] Optional Samplers - Other sampling procedures including the use of automatic samplers, pumps, siphons, multiple valves and ports, etc., may be used depending on the specific container involved.

(2) **Floating Solids** can be sampled directly or with a stainless steel scoop or spoon attached to a piece of conduit pipe, if necessary.

(3) **Sludge Sampling**

- (a) Minimum sample size is 200 grams.
- (b) If the sampling technique involves multiple aliquots, or if the final sample will consist of aliquots from several different locations in the pit, pond, or lagoon, all aliquots should be placed into a Pyrex dish, a large glass sample container or other suitable container and mixed thoroughly before containerization.
- (c) Push tube or scoop may be used to collect the sample.

[1] Stainless Steel Push Tube

- [a] Push the stainless steel push tube into the sludge
 - [b] Empty the tube contents into a Pyrex dish, a large glass sample container or other suitable container. "Emptying" may include shaking to remove sludge or extrusion of thick or gummy sludges with a new wooden dowel. A disadvantage of this technique is the need for multiple insertions of the tube into the sludge to collect sufficient sample volume.
 - [c] Repeat the procedure until an adequate amount of sample is collected.
- [2] Stainless Steel Scoop
- [a] Insert the scoop into the sludge. Attach a scoop to a piece of conduit pipe with a scoop bracket, if necessary. The scoop bracket has a decided advantage in that it allows sampling personnel to adjust the angle between the scoop and the conduit pipe.
 - [b] Empty the scoop contents into a Pyrex dish, a large glass sample container or other suitable container.
 - [c] Repeat the procedure until an adequate amount of sample is collected.

3. Surface wipes

a. Sampling Equipment

- (1) Wipe filter papers (Whatman Quantitative Grade 41 or 42 Filter Papers or equivalent)
- (2) 5% nitric acid (HNO_3)
- (3) Scintillation vials, 20-ml with polypropylene or Teflon cap liners. Metal cap liners should not be used.

b. Sampling Methodology

- (1) If multiple samples are to be taken at the site, prepare a rough sketch of the area(s) or room(s) which are to be wipe sampled.
- (2) Use a new set of clean impervious gloves with each individual sample. This avoids contamination of the filter by the hand and the subsequent possibility for false positives and prevents contact with the substance.
- (3) Moisten the filter with 5% nitric acid (HNO_3).
- (4) Wipe a section of the surface to be sampled using a template with an opening exactly 100 cm^2 . For irregular surfaces, the wiped area should be as close as possible to 100 cm^2 , estimated as accurately as possible and documented.
- (5) Maximum pressure should be applied when wiping.
- (6) To insure that all portions of the partitioned area are wiped, start at the outside edge and progress toward the center making concentric squares of decreasing size.
- (7) If the filter dries out during the wiping procedure, discard the filter, reduce area to be wiped by half, document the reduced area size and repeat wiping procedure with a new filter.
- (8) Without allowing the filter to contact any other surface, fold the filter with the exposed side in, then fold it over again. Place the filter in a sample vial, cap the vial, number it, place a corresponding number at the sample location on the sketch. Then complete the sample label and COC record.

- (9) At least one blank filter treated in the same fashion, but without wiping, should be submitted for each sample area.

4. Soils

a. General

- (1) All equipment shall be decontaminated according to specified protocols in Section III.D.
- (2) Sample containers shall be glass or polyethylene with screw top lids. Polyethylene is recommended
- (3) Holding time shall not exceed 28 days. The sample containers shall be cleaned according to protocols listed in the laboratory's current Department Approved Comprehensive Quality Assurance Plan.
- (4) Minimum sample size shall be 200 grams.

b. Sample Handling Protocols after Sample Acquisition

- (1) Breakdown the sampler, e.g., split spoon, if necessary. This should be done with the appropriate tools.
- (2) At this time, any portion of the sample that has been disturbed shall be identified, removed with a stainless steel spatula and discarded.
- (3) Slice the sample using a clean, decontaminated spatula from the center portion of the sampler, e.g., corer, split spoon or bucket auger head and place in a stainless steel, glass or aluminum foil-lined tray.
- (4) The sample in the tray shall be homogenized thoroughly by alternately mixing, dividing, and remixing the sample.
- (5) After thorough mixing, transfer the sample to the appropriate sample container(s) leaving minimal headspace.
- (6) Clean the outside of the sample container to remove excess soil.
- (7) The container rim should also be cleaned of soil and sand particles so that the lid can be sealed.
- (8) Affix sample label, seal and complete the chain-of-custody forms.
- (9) Liners
 - (a) If properly used, liners may be inserted into the sampler and used as the actual sample container.
 - (b) Be aware that EPA Test Methods for Evaluation of Solid Waste, SW-846, has mandated that all solid samples must be transported in containers that have screw tops. This also means that all container and lid requirements are still in effect.
 - (c) The ends of the liner must be covered with polyethylene, Teflon or aluminum foil sheeting. The sheeting should be secured by placing an end cap over the sheeting.
 - (d) With any sample containerized this way, specific instructions must be sent with the sample so that the laboratory will know how to handle the sample. All non-volatile samples must be homogenized by the laboratory prior to analyses. Also, any disturbed portions of the sample should be discarded prior to mixing.

- c. Composite Soil Sampling - The following is not a complete discussion regarding development of a sample compositing scheme nor all available sampling protocols. When a large site area is to be investigated for contamination, it is sometimes advantageous to composite soil or sediment samples and thus minimize the number of samples to be analyzed.
- (1) Sample aliquots (of identical size) to be composited shall be placed in a mixing tray and thoroughly mixed with a cleaned spoon, or spatula. The sample shall be thoroughly blended by mixing, and dividing into sections. Each section shall then be mixed separately. Recombine all mixed sections and mix thoroughly. Repeat sectioning and mixing process to ensure proper homogenization.
 - (2) The origin and size of each (sub)sample or aliquot that is used to make the composite shall be documented in the field notebook along with the other important sampling details.
 - (a) Although the size of these subsamples is important and should be documented, it is critical that these subsamples be of equivalent size, so that the composite sample is not biased by unequal aliquoting.
 - (b) There is no level of accuracy here; it is dependent upon the size of the aliquots.
 - (c) Aliquoting should be done in a systematic manner.
 - (3) Clean the outside of the sample container to remove excess soil, affix label, seal (if required), and complete the COC record.
- d. Discrete Soil Sampling - Note: When a relatively small site area to be investigated for contamination, discrete sampling is required.
- (1) Soil sampling locations should be selected such that a representative portion of the soils are collected with minimal disturbance with the concurrence of the DEP closure project engineer.
 - (2) Surface soil sampling (ground surface to 6 inches below ground surface)
 - (a) Leaves, grass and surface debris shall be removed from the area to be sampled using a clean stainless steel spoon or shovel.
 - (b) Surface soil samples can then be collected using a precleaned stainless steel scoop or spoon.
 - (3) Shallow subsurface soil sampling (6 inches to 2 feet below ground surface)
 - (a) Shallow subsurface samples may be collected by digging a hole or trench to the required depth with a stainless steel shovel.
 - (b) Some situations may require a trench or pit to be dug with a backhoe. Depending upon the equipment available at the site or the soil type to be penetrated, this option is acceptable. [Please note that any OSHA requirements for in-trench sampling should be followed.] In these situations, the trench is first dug to the appropriate depth and then the sample is exposed by using one precleaned spoon, spatula, or equivalent to clean away the soil that came in contact with the backhoe bucket and a second precleaned spoon to actually collect the sample.
 - (c) Alternatively, shallow subsurface soil samples may be collected with an 2-4 inch steel hand auger which would minimize the soil to be removed in order to

reach the desired depth. Using this method, a sampling depth of up to 2 feet may be obtained.

- [1] A soil sample is obtained by pushing and rotating the auger into the soil until the bucket is filled.
- [2] The sample can be removed (with some difficulty) from the bucket by pushing or scraping with an appropriate pre-cleaned stainless steel tool.
- [3] Addition of a sleeve may allow an undisturbed soil sample to be obtained.
 - [a] The device consists of a standard auger head with a removable non-contaminating sleeve which is inserted into the auger barrel.
 - [b] The soil sample is obtained in the normal manner by pushing and rotating the auger into the soil. In this case it is the sleeve which fills with soil. After auger retrieval, the sleeve, which is readily removed from the auger, is capped.
- [4] If the auger hole is prone to collapse, due to low cohesion in some soils, a temporary rigid PVC casing should be inserted into the hole. The casing prevents hole collapse and minimizes cross-contamination between soil zones as the auger is advanced. Upon sample collection, the temporary casing (if used) must be removed and the hole filled with the excavated soil. [If a confining layer has been breached during sampling, the hole shall be grouted to land surface with Type-1 Portland cement. NOTE: this requirement may be different throughout Florida. Contact the local Water Management District office for local requirements.]

IV. APPENDIX: ADDITIONAL QUALITY CONTROL REQUIREMENTS

This section contains explanatory information about facility processes and other quality control requirements for mercury recovery and mercury reclamation facilities permitted under Chapter 62-737, F.A.C. Some of this information does not pertain directly to these Quality Assurance Standard Operating Procedures for Sampling. The intent is to provide an overview and explanation of additional quality control requirements for these facilities.

A. Mercury Recovery Facilities

1. Processed Material Testing

Mercury Recovery Facilities are required in 62-737.840(3)(c), F.A.C., to test the processed lamp glass and lamp metal end caps if they intend to deliver these materials to facilities other than Mercury Reclamation Facilities. Facilities are required to take daily physical samples of these materials at the point at which they exit the processing equipment. The daily samples collected during the week shall be consolidated into one weekly composite sample. This shall be performed by thoroughly mixing equal amounts of the daily samples into a single container. The Facility shall submit the weekly sample for analytical testing to determine the total mercury content of the material sampled. This shall be performed for both glass and metal end caps individually.

Facilities are also required in 62-737.840(3)(c), F.A.C., to maintain a 12 week average value of mercury contained in the glass and metal end caps which is an average calculation of the levels reported in the weekly composite sample results. The average is calculated using the most recent 12 weekly sample results obtained from the weekly composite samples.

2. Quality Control Plan

Facilities shall submit a QC Plan which identifies the methods which they shall use for testing of the glass and metal end caps. This plan shall include logs or checklists that includes a space for the person performing the sample collection or the inspector to sign when sample collection is performed. The date and time of the inspection shall be indicated on the report. The Plan shall also include measures that the facility shall use to ensure that material is not shipped off site of the facility until the required testing and analytical results indicate the material is fit for such deliveries. This shall include the corrective measures a facility shall employ when its 12 week average levels approach the ceilings specified in the Rule. The plan must include the sampling protocols identified in the sampling section of this document.

The QC Plan must also include the types and frequencies of equipment inspections which might be required or specified by the equipment manufacturer or

defined by the facility. These inspections shall be used to determine that the processing equipment in use is properly operating. The facility must inspect the equipment daily to determine that the equipment is operating properly. The facility shall use logs or checklists and indicate any levels or meter readings associated with the inspection and shall include a space for the inspector to sign and date the written record of inspection.

B. Mercury Reclamation Facility

1. Determining Categories of Material

Facilities shall determine the types and general formulations of material which they introduce in their process for mercury reclamation and determine the process recovery rates of mercury for each process.

As specified in 62-737.860.(4), F.A.C., Mercury Reclamation Facilities shall demonstrate, using an EPA approved methodology for analyzing total mercury content, an effective reclamation rate of 99 percent of the mercury introduced into the reclamation process, or a resulting total mercury concentration of the processed material which is below the method detection limits for the testing method utilized. Facilities must demonstrate through records and logs the effectiveness of their process. Facilities must submit testing and QC Plans for each type or classification of material introduced into its equipment for reclamation. Types of material can be identified in categories such as phosphor powder resulting from mercury recovery operations, co-mingled crushed or broken lamps, mercury containing glass ampoules removed from thermostats, etc. An example of a category would be the following:

"Category 1 - Mercury containing phosphor powder received from Mercury Recovery operations where the material is a combination of mercury, fragments of broken glass, and phosphor powder and the range of mercury concentration of the material is between 2,000 and 4,000 parts per million of total mercury and where the total mercury does not exceed 4,000 parts per million."

A facility may process various types or categories of mercury containing materials at its facilities but they must segregate these materials into categories, define the quantitative limits and the constituents of a material included in a category, and determine the effective recovery rate for each category of material it processes. When a facility performs testing to determine the effective recovery rate for material processed within a category, it must perform testing on the material as represented in a worst case, or highest concentration of mercury which is defined in that category. In its QC plan, these facilities must also identify how it will carry out on going analytical testing programs to monitor the effectiveness of each process and each category of material processed. The must also include written plans which describe how they will monitor processing equipment during processing activities, to ensure that the process is being performed as indicated in its process definitions.

2. Defining Processes

Facilities must determine the specifics of a process that it shall perform on the categories of materials it will process. An example of a process description is:

"Category 1, Low Level Phosphor Powder;

Process: Load material in not greater than 45 pound quantities into a specially designed container and place into the retort operation which consists of Acme model 1234 Vacuum Retort Machine. Raise the temperature of the material to 1600 degrees F within one hour, maintain a temperature of 1600 F for 9 hours and cool the material for one hour in the device until the temperature of the material is less than 100 F."

A process may contain several variables it might have as in altering the temperatures during the process, or taking material from one piece of equipment and processing it in another device as a secondary process. Facilities shall adequately describe the functional aspects of a process and in its QC plan, define what steps it will take to ensure the process is performed as specified in the process description.

3. Sample and Test to Determine Effectiveness

Once a category of material is defined and a process is engineered, then the facility must demonstrate the effectiveness of the process. To determine the effectiveness of the process, facilities must sample and test the material entering the process for total mercury content, perform the process under the methods identified in the process description, and then sample and test the material exiting the process for total mercury content. The results of the testing must demonstrate a mercury recovery rate of 99 percent or greater.

4. QC Plan for Each Category and Process

When a facility identifies a category of material it desires to process, defines a protocol by which to process the material and recover 99 percent of the mercury of that material entered into the process, then it must define a Quality Control Plan to monitor the effectiveness of the process. The QC plan shall include the testing required and the frequency of testing which it will employ to monitor the process. As specified in 62-737.860(4), F.A.C., the Department shall require, at a minimum, one complete process evaluation test per category and process type semi-annually to determine the continuing effectiveness of the process. This test shall include EPA approved analytical methods defined to identify the maximum mercury concentration contained in the material identified in a category or type and after processing the material, analytical testing to determine the remaining mercury, or the mercury reclamation rate of 99 percent, or the non-detection of mercury in the processed material. The physical sampling procedures shall be defined in the sampling section of this document.

As part of its QC plan, facilities must implement records or checklists that operators or inspectors shall use to monitor the essential elements of a process, such as temperature of the device during use or times or procedures which must be performed during the process. These checklists must include a space for the operator or inspector to initial or sign the document and the times and dates of the inspection once the ongoing inspections are performed.

5. Summary

Mercury Reclamation Facilities must define the types and physical specifications and the maximum mercury concentration of material which it shall process. They then must specify the physical process which they shall use to reclaim the mercury contained in the material. Next, facilities must use analytical testing of the unprocessed and processed material to demonstrate that the applied process can recover 99 percent of the mercury contained in the material. Alternately, facilities may use analytical testing of processed material only to demonstrate a total mercury content below the method detection limit. Last, facilities must use analytical testing on a semi-annual basis on sample or production runs of processing operations and use process checklists and inspections to demonstrate that the process continues to deliver the recovery rates of mercury which were determined during the original testing of the process.