Industrial and Hazardous Waste Sampling and Shipping Procedures Introduction

Sampling is generally conducted to verify the identity of a waste or to identify releases of hazardous wastes or hazardous constituents to the environment. The investigator must determine what conditions or activities may necessitate sampling. The following are examples of situations for which sampling may be required:

- A potentially hazardous waste is being handled as a non-hazardous waste. Sampling may be required to verify waste classification.
- Waste handling practices indicate that mislabeling or misidentification of waste has occurred or is likely to occur. Sampling may be required to demonstrate that the facility is mislabeling or misidentifying wastes.
- There is evidence of possible releases of hazardous wastes from waste management units, satellite storage areas, waste generating areas, etc. Sampling media and wastes may be required to document that a release has occurred or is occurring.
- Wastes may be managed improperly, i.e., in an inappropriate treatment or disposal unit. Sampling may be required to verify that the correct wastes are being managed in the facility's various waste management units.

During the investigation, the investigator should identify the types and locations of samples that may need to be collected. The investigator should identify:

- The media or wastes to be sampled.
- The physical locations to sample.
- The steps within a treatment process to sample.
- The physical characteristics of the medium to be sampled.
- Other relevant information that would be helpful in developing a sampling plan.

The investigator must then decide whether to sample during the investigation or to conduct a follow-up or case development investigation at a later date. A number of factors enter into the sampling decision making process:

- Can sampling wait until a future date or time without jeopardizing documentation of potential violations?
- Is there potential for an imminent threat to human health or the environment?
- Does the investigator have appropriate sampling equipment (including personal protection and safety equipment)?
- Can the investigator develop an adequate sampling plan with available information?

Risks are always involved when sampling potentially hazardous wastes or media. If the investigator believes that immediate sampling is necessary, he may want to call his manager or other experienced personnel for assistance.

In general, sampling requires the collection of adequately sized, representative samples of the wastes or contaminated media. Sampling situations vary widely and therefore no universal sampling procedure can be recommended. This chapter outlines several procedures for sampling different types of wastes in various physical states and containers.

These procedures require a plan of action to maximize safety of sampling personnel, minimize sampling time and cost, reduce errors in sampling, and protect the integrity of the samples after collection.

Preparation for Sampling

A. Background Information About the Waste

Accurate background information about the waste to be sampled is very important in planning any sampling activity. The information is used to determine the required types of protective equipment, sampling precautions to be observed, as well as the types of samplers, sample containers, container closures, and (when needed) preservatives. Generally, information about the waste determines the kind of sampling scheme to be used.

Often, the information about the waste is incomplete. In these instances, as much information as possible must be obtained by examining any documentation pertaining to the wastes, such as the shipping manifest, material safety data sheets on chemicals used and standard references on industrial processes. When documentation is not available, information must be obtained from the generator, transporter, disposer, or processor.

B. Sampling Plan

The primary purpose of the sampling program is to obtain representative samples of waste, soil, water, and any other media possibly containing or contaminated by hazardous wastes. Representative samples will aid in the evaluation of the nature and extent of hazardous waste deposits present at each site. The first step in developing a site-specific Sampling Plan is to decide what is to be accomplished by the data gathered during the sampling program. Based on this overall goal, specific sampling requirements can be set. The investigator must decide whether preliminary qualitative or detailed quantitative analysis is needed; what media should be sampled; what parameters should be analyzed; what type of equipment should be used; and what types of field measurements are required.

C. Sampling Safety

Proper safety precautions must always be observed when sampling hazardous wastes. In all cases, the investigator must be aware that the waste can be a strong sensitizer and can be corrosive, flammable, explosive, toxic and capable of releasing extremely poisonous gases. The background information obtained about the waste should be helpful in deciding the extent of sampling safety precautions to be observed and

in choosing protective equipment to be used. In some situations obtaining a sample using normal sampling procedures may be too dangerous and should not be attempted. The following general safety rules and practices should be followed whenever sampling:

- Each sample must be treated and handled as though it were extremely hazardous, sample procedures should minimize the risk of personnel exposure.
- If special handling of a sample would be appropriate, you must also warn the laboratory which receives the sample.
- Approved and appropriate safety equipment, such as gloves, eye protection, foot protection and respirators must be worn in areas where hazardous conditions are suspected. In addition, eye protection should be worn when handling acidic, caustic, or other hazardous liquids (including preservative chemicals).

D. Selection of Sampler

Hazardous wastes are often complex, multi-phase mixtures of liquids, semisolids, sludges, or solids. The liquid and semisolid mixtures vary greatly in viscosity, corrosivity, volatility, explosivity, and flammability. The solid wastes can range from powders to granules to big lumps. The wastes may be in drums, barrels, sacks, bins, vacuum trucks, ponds, or other containers.

Sampling these diverse types of wastes requires different types of samplers. Sampling devices are described in this chapter. Table 6-1 is a general guide to the types of waste that can be sampled by each of the samplers described.

Sampling Equipment

This section describes EPA-approved equipment and procedures for obtaining representative samples of a solid waste. The information in this section is general in nature. Since each specific sampling situation is unique, the equipment and procedures described must be modified appropriately in an actual use situation to ensure that representative samples are collected. There are several things to be aware of concerning sampling equipment:

- Continued exposure to waste materials will shorten the life of most equipment.
- Exposure to sometimes harsh decontamination solutions will add to the above chemical effects.
- The useful life of a piece of equipment can be decreased by inadequate storage.

A regular schedule of sampling equipment maintenance should be established. This will ensure that the next sample event will have reliable and clean operating equipment. A person should be designated to repair and order equipment as needed.

A. Pond Sampler

Scope and Application:

The pond sampler consists of a glass or a plastic beaker clamped to the end of a 2 or 3 piece telescoping aluminum or fiberglass pole which serves as the handle. This instrument samples liquids and free-flowing slurries.

General Comments and Precautions:

- Do not use a nonfluorocarbon plastic beaker to sample wastes containing organic materials.
- Do not use a glass beaker to sample wastes of high pH or wastes that contain hydrofluoric acid.
- Paint aluminum pole and clamp with a 2-part epoxy or other chemical-resistant paint when sampling either alkaline or acidic wastes.

Procedure:

- Clean beaker, clamp, and handle.
- Assemble sampler by bolting adjustable clamp to the pole. Place the beaker in the clamp and fasten shut.
- Turn sampler so the mouth of the beaker faces down and insert into waste material. Turn beaker right side up when the dipper is at desired depth. Allow the beaker to fill completely as shown by the cessation of air bubbles.
- Raise pond sampler and transfer sample to the container.

B. Oil Thief

Scope and Application:

An oil thief is a brass sampler somewhat similar in construction to a Kemmerer sampler except that the upper and lower seals are spring activated. The brass construction prevents the possibility of creating a spark when it is operated. It is used to sample oil and sludges which are not high in solids.

Procedure:

- Clean sampler.
- Set the sampler by pulling the upper and lower stoppers until the spring trigger is held in the open position.
- Lower the sampler to the desired depth and activate the spring trigger. The stoppers will then close by the spring action.
- Raise sampler and transfer sample to container.

C. Trier

Scope and Application:

A trier consists of a tube cut in half lengthwise with a sharpened tip that allows the sampler to cut into sticky solids and loosen soil. A trier can be used to sample moist or sticky solids with a particle diameter less than one-half the diameter of the trier. It may be useful for sampling waste piles.

Procedure:

- Clean trier.
- Insert trier into waste material 0 to 45 degrees from horizontal. Rotate trier to cut a core of the waste. Remove trier with the concave side up and transfer the sample to the container.

D. Bucket Auger

Scope and Application:

A bucket auger consists of two dull cutting bits welded to an open-ended cylinder bucket. A central metal T-shaft is attached to the other end of the cylinder. It is used to pull an undisturbed core of soil.

General Comments and Precautions:

- This auger cannot be used on rocky soils.
- It may be difficult to extract the sample from the bucket intact. This will take some practice to get right.

Procedure:

- Clean sampler.
- Rotate the auger clockwise with downward pressure. When the cylinder bucket is full, the auger is lifted and twisted out of the hole.
- Push the sample from the opening near the shaft. It should scrape the cutting bits slightly as it is extracted.

E. Scoop and Shovel

Scope and Application:

Scoops are disposable sampling tools used to sample granular material or surface soil. Shovels are reusable and are used to sample subsurface soils.

Procedure:

- Clean the shovel.
- Obtain a full cross section for the waste material using a scoop or shovel that is large enough to contain the waste collected in one cross section sweep.

F. Bailer

Scope and Application:

A bailer is a tube made of PVC, stainless steel, teflon, etc., which is open at the top. The bottom is sealed with a ball check valve. This sampler is used in collecting water from wells. The TNRCC has PVC, stainless steel, and acrylic (hydrocarbon surface sampler) types of bailers.

Procedure:

- Clean the bailer.
- Lower the sampler down a well until it touches the water surface. Lower the sampler slowly until the top of the bailer is approximately 12 inches below the water or waste surface. Allow the bailer to fill. Pull up and raise to surface. Ball valve will automatically seal the bottom opening.

G. Glass Tubing

Scope and Application:

Glass tubing is used to collect liquid samples. Its advantage is that representative samples of stratified wastes can be taken and it is simple to use. The surface tension of the waste across the opening helps hold the sample in the tubing. Organic wastes usually have lower surface tension than water and it may be difficult to retain the sample volume in the tubing.

Procedure:

- Place a wide mouth jar on the ground near the sampling location.
- Vertically lower the tubing to the desired depth. Make certain that a freeboard of at least 6 inches of tubing is kept above the waste.
- Seal the opening with your thumb. Wearing gloves will help assure a tight seal at the opening. With a steady and smooth motion lift the tubing into position above the wide mouth jar. Carefully release your thumb from the opening and allow the sample to flow into the bottle.
- Dispose of the tubing in the container which was sampled.

H. Coring/Sampling Device

Scope and Application:

The recommended method of sample collection for both low and high concentrations of volatiles in soils, sludges or sediments is to collect the sample using a coring device and to quickly extrude the sample core into a *tared* 40-mL VOA vial that does not contain preservative but does contain the stir bar, if applicable. This prevents aerobic bacterial breakdown and volatilization from sample handling.

Procedure:

- Prior to sampling check the tared weight of the VOA vial, pre-tared by the lab, to ± 0.1 g.
- Take the sampler, i.e., En-CoreTM, syringe, and take a core sample of the soil, sediment.
- Place the core sample into the tared VOA vial.
- Inspect and wipe the threads of the vial clean. Seal the vial and chill immediately. Hold at 4±2EC, and ship to the laboratory.

The vial remains unopened until after the analysis is complete. The laboratory should analyze the sample within 48 hours from the time of collection. Alternatively, the laboratory can preserve the sample within the 48 hours to extend the holding time to 14 days (See Section 9.0 below). The manual addition of any water, surrogates, and/or internal standards and all additions of preservatives should be made using a 22-gauge or thinner needle through the septum seal. This collection procedure does not require the use of preservatives in the field or balances in the field.

Another Option:

Collection of the sample in an approved coring device that can serve as an intermediate hermetically sealed sample container should be used according to the manufacturer's instructions.

Procedure:

- Take the sampler, i.e., En-CoreTM, syringe-type, and take a core sample of the soil, sediment.
- Wipe the outside of the sampler to remove anything that would prevent an airtight seal and quickly seal the sampler and chill to 4±2EC, and ship to the laboratory.

I. Field Instruments

Field instruments will normally be used for environmental sampling. Immersing the probe of a field instrument into a hazardous waste sample will probably destroy the probe. Field instruments should be calibrated prior to field use and recalibrated in the field before measuring each sample. The sampling plan should describe these procedures and the field notebook must document the calibration.

Operation of field instruments varies depending on the manufacture. Consult the operation manual for specific instruments for operation and use. Field instrument measurements should be made immediately after the sample is collected or may be performed in-situ.

J. Sample Storage Containers

The most important factors to consider when choosing containers, for hazardous waste samples are compatibility, resistance to breakage, and volume. Containers must not melt, rupture, or leak as a result of handling or chemical reactions with the samples. Containers with wide mouths are easiest to work with. Also, the containers must be large enough to contain the required volume of the sample.

The plastic containers available for use by Commission personnel are constructed of linear polyethylene with a polypropylene cap. These cubitainers are available in 1 liter and 5 liter sizes. They should be used to collect and store aqueous samples which do not contain oily residues, pesticides, or halogenated hydrocarbons.

Glass containers are inert to most chemicals and can be used to collect and store all hazardous waste samples except those that contain hydrofluoric acid or strong alkali. Wide mouth 1-liter jars and 40 ml

Volatile Organics Analysis (VOA) vials are available. These are provided with a rigid plastic or metal cap and a teflon liner. The VOA vials are used to collect samples for analysis of volatile organics or very concentrated hydrocarbon samples which are to be analyzed by GC or GC/MS. The 1-liter glass jars are used to collect samples containing semi-volatile hydrocarbons or halogenated organics to be analyzed by GC and GC/MS.

Sampling Various Media

The following procedures are recommended for sampling different types of hazardous wastes in various containers.

A. Sampling a Drum

Drums containing liquid wastes can be under pressure or vacuum. A bulging drum usually indicates that it is under high pressure and a heavily corroded or rusted drum can readily rupture and spill its contents when disturbed. Opening the bung on a drum can produce a spark that might detonate an explosive gas mixture in the drum. These situations are difficult to predict and must be taken into consideration every time a site is inspected. The need to leave an unopened drum in an undisturbed state cannot be overemphasized. *The investigator should never open a sealed drum!*

If a leaking drum is discovered at a site, the contaminated soil near the drum should be sampled. If an open drum is discovered at a site, collect a sample through the opening with a glass tube. The investigator should mark unlabeled or improperly labeled drums. Numbering drums with spray paint helps to identify the drum and will discourage improper disposal of the drum while waiting for analysis.

B. Sampling a Barrel, Fiberdrum, Can, Bag, or Sack Containing Powder or Granular Waste

The proper protective respirator in addition to the other protective gear, must be worn when sampling dry powdered or granular wastes in these containers. These wastes tend to generate airborne particles when the containers are disturbed. Collect a composite sample from the container with a trier or scoop.

C. Sampling a Surface Impoundment

Storage or evaporation ponds for hazardous wastes vary greatly in size. It is difficult to collect representative samples from the large ponds without incurring large expenses and assuming excessive risks. Any samples desired beyond $3.5 \text{ m} (11 \frac{1}{2} \text{ ft.})$ from the bank may require the use of a boat, which is very risky, or the use of a crane or a helicopter, which is very expensive. The information sought must be weighed against the risk and expense of collecting the samples. The pond sampler can be used to collect samples as far as $3.5 \text{ m} (11 \frac{1}{2} \text{ ft.})$ from the bank. The investigator may also want to sample the surface impoundment sludges from exposed areas. The pond sampler can be used to scoop up the sludges while still remaining a safe distance from the pond. Collect a composite sample with a pond sampler, as described

in this chapter.

D. Sampling Soil

Soil samples may be collected for different purposes. A grab of soil contaminated with spilled material may be appropriate to document contamination.

If the investigator's purpose is to determine levels of contamination at a site, either prior to or after a cleanup, the following procedure should be followed. Soil samples are taken in a grid pattern over the entire site to ensure a uniform coverage.

- Divide the area into an imaginary grid.
- Sample each grid.
- To sample up to 8 cm (3 in.) deep, collect samples with a bucket auger.

E. Sampling a Waste Pile

Waste piles can range from small heaps to large mounds of wastes. The wastes are predominantly solid and can be a mixture of powders, granules, and chunks. A number of core samples have to be taken at different angles and composited to obtain a sample that, on analysis, will give average values for the hazardous components in the waste pile. Collect the samples with a waste pile sampler such as a trier, shovel, auger, or scoop.

F. Sampling a Storage Tank

Sampling a storage tank requires a great deal of manual dexterity. Usually it requires climbing to the top of the tank through a narrow vertical or spiral stairway while wearing protective equipment and carrying sampling paraphernalia. At least two people must be involved in the sampling: One should collect the actual samples and the other should stand back, usually at the head of the stairway, and observe, ready to assist or call for help. The investigators should be accompanied by a representative of the company, to open the sampling hole, usually on the tank roof.

- Collect one sample each from the upper, middle, and lower sections of the tank contents with a weighted bottle sampler.
- Combine the samples in one container and submit it as a composite sample and/or analyze each phase separately.

G. Sampling a Well

Wells are sampled with bailers and various types of pumps. Well sampling can be as simple as collecting a grab with a bailer to as detailed as groundwater monitoring for CME investigations. The following publications should be consulted for well sampling procedures.

• RCRA Ground Water Monitoring Technical Enforcement Guidance Document.

• TNRCC Manual: RCRA Comprehensive Ground Water Monitoring Evaluation Training Session.

H. Equipment Decontamination

All reusable sampling equipment should be properly cleaned before going into the field. It is recommended that the cleaned equipment be wrapped to help distinguish the cleaned equipment and to protect it from dust or other contaminants. Butcher paper or plastic sacks are recommended as the wrapping material. Sample containers for some organic analyses should also be cleaned. The sampling plan should detail cleaning procedures for sampling equipment and containers. When the sampling and field activities are completed, all equipment including safety equipment and field instrumentation should be decontaminated before leaving the site. The purpose of the field decontamination procedures is to protect the field equipment from gross contamination. It will also make it easier to clean the equipment before the next site investigation. The field decontamination procedures should provide a quick method of removing most sample residues from the equipment. Rinsing and/or wiping with paper towels is usually sufficient for field decontamination.

The investigator should consider designating some non-disposable samplers as "clean" or "environmental" sampling equipment. Generally, low (ppm) concentrations of contaminants occur in contaminated ground or surface waters. If some samplers are designated for this type of sampling, there will be less chance of cross contamination from inadequately decontaminated sampling equipment.

- Sample Equipment and Containers: Sampling equipment should be cleaned with a nonphosphate detergent in hot water, tap water rinse, solvent rinse if necessary, and a final distilled water rinse. Some authorities suggest the solvent be the last rinse but cross-contamination may be a problem. The best solvent to be used in the cleaning will depend on the parameters that are requested. Methylene chloride, acetone, hexane, and methanol are all appropriate solvents if semivolatile organics are to be analyzed. If volatiles are to be analyzed, ultra pure methanol is the solvent of choice. Acetone may also be used if it is not an analyte of interest.
- *Instruments*: Decontamination of meters should consist of a distilled water rinse of the probe or portion of the probe which was in contact with the sample. If the contamination is difficult to remove, then consult the owner's manual for specific instructions. Obviously, grossly contaminated samples probably should not be measured for field parameters if possible. Between use, probes should be stored according to manufacturers' instructions.
- *Safety Equipment*: Personnel equipment should be decontaminated in the field. Most dealers of rubberized products have their own mild detergents.

Quality Assurance/quality Control Procedures

A. Number of Samples

Collecting one representative sample of a given waste is usually adequate to determine the average concentrations of the hazardous components. Unless you are testing for volatiles, this sample can be

collected from either a single sampling point with a composite sampler, or several samples can be collected from various sampling points and combined into one composite sample.

When gathering evidence for possible legal action, multiple samples of a waste are sometimes collected. Two identical samples are desirable: one sample is given to the person or entity responsible for the waste for its own analysis; the second sample is submitted to our laboratory for analysis. A third sample can be kept in storage for possible use as a referee sample if desired. Subdividing a waste sample is not recommended unless it is homogeneous.

Quality assurance rules require samples to be collected in number and nature sufficient to represent the total site. In a complex solid waste site, this requirement, strictly enforced, can require an impractical number of samples. Seldom is it necessary to know every major chemical in a site. If a suspected source of the waste can be determined, likely components of the waste can be assumed. By limiting the components of interest, the number of samples required to determine their presence or absence is decreased.

B. Volume of Sample

Sufficient volume of the sample, representative of the main body of the waste, must be collected. This sample must also be adequate in size for all analytical needs. The concentration of the contaminant, the type of analysis, and the sample medium determine the volume requirements. Table 6-2 and Table 6-2a gives general guidelines for volume requirements.

C. Quality Control Samples

- *Environmental Sample* (or field sample) is a representative sample of any material (aqueous, nonaqueous, or mixed matrix) collected from any source for which determination of composition or contamination is requested or required.
- *Field blanks* should be collected at least one in ten samples (1:10) collected. Field blanks are aliquots of metal and/or organic-free water that contact sampling equipment under field conditions and are analyzed to detect any contamination from sampling equipment, cross contamination from previously collected samples, or contamination from conditions during sampling (e.g., airborne contaminants that are not from the waste being sampled).
- *Trip blanks* must be incorporated with all volatile sample collections. This is to check silicone Teflon membrane permeability. Trip blanks are not opened in the field. They are incorporated to check sample contamination that may originate from sample transport, shipping and from site conditions.
- *Equipment Blanks*: These blanks must be collected after each sample is taken. Opened in the field, collected as a rinsate blank to demonstrate any cross contamination between each sample collected, and returned to the laboratory as a sample. Cross contamination can occur with the repeated use of the same sampling equipment. Equipment blanks are not necessary when disposable sample equipment is used.

- *Field duplicates* are to be collected one in ten samples (1:10) to document precision. These are two samples taken at the same time, and at the same location. The precision resulting from field duplicates is a function of the variance of waste composition, the variance of the sampling technique, and the variance of laboratory sample selection.
- *Matrix Duplicates*: These samples are restricted to organic sample collection only. These are samples used by the laboratory to prepare a matrix spike and matrix spike duplicate for each analytical method employed. One sample during a sampling event must be collected in triplicate for volatiles using 40-mL vials, and semi-volatiles in duplicate using one liter (1-L) containers. The 1-L containers used for semi-volatile collection must be pre rinsed with reagent grade methylene chloride and baked in a drying oven to remove any remaining residual solvent. This procedure is currently performed by the laboratory.
- *Sample Duplicates*: All water samples for VOA analysis should be collected in duplicate. All soil. Sludge or sediment samples for VOA analysis should be collected in triplicate into 40-ml VOA glass vials. Duplicate samples should also be collected for other parameters such as pesticides, semi-volatile organics, and trace metals. The purpose of collecting sample duplicates is to verify that the sampling procedures are reproducible and representative. It is recommended that duplicates be collected at a frequency of once for every ten samples (1:10), with the exception of VOA analyses for water samples noted above.

D. Analytical Parameter Selection

Once the sample media and sampling points have been selected, it is necessary to select the parameters for analysis which will provide the desired information. Those parameters which are known or suspected to be in the waste will be the primary focus of the analytical program. Cases where no information about the waste is known present the largest problems. Deciding which parameters to request for analysis. It is impossible to make guidelines for predicting which parameters to request which will hold true for every case. The final decision is left to the investigator. Tables 6-3 and 6-4 present some guidelines which may help in developing the analysis request.

E. Sample Handling

After a sample is transferred into the proper sample container, the container must be tightly capped as quickly as possible to prevent the loss of volatile components and to exclude possible oxidation from the air.

The use of a preservative or additive is not usually recommended for solid waste samples. However, if only one or two components of a waste are of interest, and if these components are known to rapidly degrade or deteriorate chemically or biochemically, the sample may be refrigerated at $4E\pm 2EC$ (39. $2E\pm 3.6EF$.) or treated with preservatives according to EPA approved guidelines. Remember it is important to keep the samples in a secure place until the shipper picks them up.

F. Sample Identification

Each sample must be labeled and sealed properly immediately after collection. Sample seals are used to preserve the integrity of the sample from the time it is collected until it is opened in the laboratory. Gummed paper seals are used as official sample seals. The seal must carry information such as the collector's name and the date of collection.

The seal must be attached in such a way that it is necessary to break it in order to open the sample container. The seal should drape over the cap/lid and down the side of the container.

G. Field Log Book

Information pertinent to a field survey and/or sampling event must be recorded. Entries in the log book should include the following types of information:

- Purpose of sampling (e.g., waste classification, environmental impact, ground-water monitoring, etc.).
- Location of sampling (e.g., transporter, disposal site, etc.) and address.
- Name and address of field contact.
- Generator of waste and address.
- Type of process (if known) producing waste.
- Type of waste (e.g., sludge, wastewater, soil, oil, etc.).
- Declared waste components and concentrations.
- Number and volume of sample taken.
- Description of sampling point.
- Date and time of collection.
- Collector's sample identification number(s).
- Sample distribution (e.g., laboratory, transporter, etc.).
- References such as maps or photographs of the sampling site.
- Field observation.
- Any field measurements made such as pH, flammability, explositivity, etc.
- Calibration record of field equipment.

Sampling situations vary widely. No general rule can be given as to the extent of information that must be entered in the log book. A good rule, however, is to record sufficient information so that someone can reconstruct the sampling situation without relying on the collector's memory.

The log book must be retained in accordance with TNRCC policy. See Chapter 11 for additional guidance.

H. Chain of Custody Procedures

Chain of custody (COC) requires permanent records of all sample handling and shipment. COC procedures must be used to ensure sample integrity and legally and technically defensible data. One member of the investigation team should be responsible for initiating COC procedures and documenting the sample source.

The sample should be kept in view or within limited access, locked storage until custody is relinquished and formal documentation of the transfer is completed. The person collecting a sample will start the chain of custody procedure.

TNRCC Chain Of Custody Procedures

The following instructions will explain the different sections on the TNRCC COC record. Consult your laboratory for chain of custody forms.

Copies of the Commission's COC are included in this chapter's appendix (FIGURE 6-1). Each COC has a unique preprinted number in the upper right-hand corner. The form is printed on carbonless paper and has four copies.

Tear-off copies:

- The *green* copy will be kept by the person submitting the samples.
- The *yellow* copy will be retained by the laboratory.
- The *pink* copy will be sent to Austin (laboratory contract manager).
- The top *white* copy will be submitted to the laboratory along with the yellow copy. After the analyses are complete, the white copy will be sent by the laboratory to Austin as part of the original laboratory report.

Top of Form:

The top of the form has pertinent information for the investigator and laboratory. This information is identified on the example (Table 10-5) by number and includes:

- The location where the sample(s) was collected and the permit number. This information should not be filled out if the sample is sent to a contract laboratory. This information should be in the field notebook of the investigator and can be written on the green copy retained by the investigator.
- The region.
- The investigator's organization number (needed for TNRCC LIMS).
- PCA Code.
- Program such as water or waste.
- The telephone number where the sampler can be reached if the laboratory has questions (important for contract labs).
- The email ID of the person receiving the report.
- Samplers' signature, this is required.

• Samplers' name printed legibly so the laboratory or the laboratory contract manager knows who should receive the report.

Sample Information:

The next part of the form includes the sample information for use by the investigator and the laboratory.

- The shaded area with Lab ID Number is for use by the laboratory only.
- Sample ID will consist of the COC #-01, -02, etc. The -01, -02 has been preprinted in for the sampler. Additional sample information can be added such as Outfall 001, Drum 152, etc., but do not include the terms "blank", "dup" or other words which could theoretically give the lab clues regarding the presence or absence of analytes. This COC can be used for a maximum of ten samples from the sample location/site. A sample can consist of more than one container, if different container types or preservatives are used. The labeling of the container is discussed below.
 - * Date: the date the sample is collected.
 - * Time: time the sample is collected in military time.
 - * # of bottles: number of containers for a particular sample as described in 10. The sample would be from the same point of collection but may have different containers requested. The total number of containers for that sample would be entered in this place.
 - * Grab/Comp.: enter either G or C if the sample is a grab or composite. If the sample is neither, then leave blank. For a composite, the duration and number of aliquots must be included such as 24/12 for 24 hours and 12 aliquots.
 - * Sample Type: enter either L (Liquid), S (Solid), M (multi phase), T (tissue), or O (Oil).
 - * Cl_2 : enter the residual chlorine, if it was measured.
 - * pH: enter the pH of the sample, if it was measured.
 - * Cond.: enter the conductivity of the sample, if it was measured.
 - * Anal. Req.: enter 'See RFA' if one is attached. The analyses can be written here but it must be legible so that the laboratory can determine what the investigator is requesting.
 - * Remarks: any additional comments about the sample, additional data such as flow measurements can be added here, or special directions.

Chain of Custody:

The signature must include all persons who handled the sample. If a shipper is used the shipper name and tracking number are entered at the bottom, the courier does not need to sign the form. The form should be signed with the date and time the samples were transferred to the shipper and the date and time the

samples were received by the laboratory from the shipper. There must not be any breaks in the custody, not even one minute. When shipping, the COC should be tapped under the lid of the ice chest in a ziplock bag.

- C The first 'relinquished by' must be the sample collector. The shipper must be filled in as received and relinquished.
- C The date must include the year.
- C The time must be in military time.
- C The remainder of the form is for use by the laboratory.

Sample Label:

Each container will have a sample label (FIGURE 10-3) to identify the sample. The label must be filled out and attached to the container before the container is filled with the sample. If the label is put on after the sample has been cooled, condensation will prevent it from sticking.

The sample ID is the COC form #-01, -02, etc., (e.g., 0123456-01, 0123456-02.). If there are multiple containers for one sample, each container will have the same sample number. The preservative must be indicated on each label.

Analysis/Comments:

This section must have the hand written analysis and any other comments about the sample. If cyanide or some other toxin is expected, or if the sample is particularly hazardous, comments must be added to the label. In completing the label, care should be utilized to insure that all necessary information is correct and is legibly entered onto the label with a black waterproof ink pen.

Request For Analysis:

The request for analysis (RFA) is program specific. The example (FIGURE 6-4 & 4a) is for the IHW program and contains analyses that are requested.

The COC number is written at the top of the form, so that the laboratory can match the analysis with the samples.

In completing the RFA, care should be utilized to insure that all necessary information is correct and is legibly entered onto the sheet with a black waterproof ink pen.

I. Shipping of Samples

Samples should be delivered to the laboratory for analysis as soon as possible - usually within 1 or 2 days

after sampling. The samples must be accompanied by the chain-of-custody (COC) (FIGURE 6-2). Also, a request-for-analysis sheet (FIGURE 6-4 & 4a) may accompany the COC when needed. In addition, when applicable (DOT; Exceptions For Limited Quantities Of Hazardous Materials, Not Classified As Consumer Commodities), shipping papers, noting the hazardous material in limited quantities must be prepared. Please be aware that there is a potential for the shipper to be held accountable for the shipment. The samples must be delivered to the person in the laboratory authorized to receive samples (often referred to as the sample custodian).

For TNRCC's purposes, there are two broad categories which affect this agency's shipping of samples. The first category will be called Transportation of Hazardous Materials, Not Offered for Commerce. This is the least restrictive of DOT's hazardous materials regulations when a hazardous material is not offered for commerce but, rather, transported in agency vehicles. Samples transported in state owned or leased vehicles are not subject to the hazardous material regulations, because the material is not being offered "in commerce." Use of a personal vehicle, for which an employee receives compensation from the state, would be considered by the US DOT the same as a state-owned vehicle. TNRCC employees may personally transport the samples known or suspected to be hazardous to a local lab.

For example, a regional office sampled numerous used oil facilities. Many of the oil mixtures contained gasoline and solvents. The sample jars were sealed inside plastic bags that were inside ice-filled coolers. However, one was able to detect the smell of gasoline outside the closed coolers. These samples were transported directly to contract labs by TNRCC personnel. Had they been offered for shipment to a commercial carrier, the samples would not have been compliant with DOT's regulations (Wastewater samples are exempted per 40 CFR 136.4).

When a sample is transported by TNRCC personnel to a laboratory, it must be packaged in a proper shipping container (e.g., an igloo-ice chest) to avoid leakage and/or breakage. It is recommended that the individual shipping the sample use "POLY-NET," bubble wrap on all glass containers. If you are concerned about melting ice leaking out of the ice chest, use a heavy-gage, plastic garbage bag. Place the bag in the ice chest and put the sample containers inside the bag. Open the ice bags and distribute the ice over and around the samples. Tie the top of the trash bag in a knot or use reinforced tape, string, rubber bands, etc., to seal the top of the bag. Don't forget to place the COC inside a ziplock bag and tape it to the bottom of the cooler lid.

The second category will be called Exceptions For Limited Quantities Of Hazardous Materials, Not Classified As Consumer Commodities. Samples that are known to be hazardous must be shipped as a hazardous material. The shipping paper must state the type of material being shipped and that it is being shipped in "limited quantity." It should be noted that, unlike a hazardous waste manifest, a COC does not serve as a shipping paper. Therefore, in addition to preparing a COC that will be packed inside the cooler with the sample, a shipping paper, noting the hazardous materials in limited quantities, must also be prepared. To qualify for the limited quantity exception, the quantity of the material must be packaged in an inner container, secured inside a strong outer packaging.

In the used oil example, mentioned earlier, the samples would have been securely packed in absorbent, non-reactive material, inside an inner container, which, in turn, would need to be securely packed in a strong outer packaging. The package would then have been labeled (FIGURE 10-5) and shipped in accordance with DOT's regulations applicable to hazardous materials in limited quantities. If samples are shipped on Wednesdays or Thursday, be sure to confirm that they will reach the laboratory by Friday morning.

Two types of samples taken by an IHW investigator could probably be categorized as ignitable (DOT flammable) or corrosive. Their proper shipping names, which must appear on the shipping papers, are as follows:

Flammable liquids, n.o.s., 3, UN 1993, PG III Corrosive liquids, n.o.s., 8, UN 1760, PG III

NOTICE: If you know or reasonably suspect the samples to be flammable or corrosive you must label (See FIGURE 6-5) and ship in accordance with DOT requirements.

Other samples may present a hazard during transportation, however, they may not meet the definition of any other hazard class. In these situations, the proper shipping names for these types of materials are as follows:

Environmentally hazardous substances, liquid, n.o.s., 9, UN 3082, PG III Environmentally hazardous substances, solid, n.o.s., 9, UN 3077, PG III

Unless offered or intended for transportation by aircraft, limited quantities of miscellaneous hazardous material (Class 9) are excepted from labeling and placarding requirements as long as they are packaged in combination packages, not exceeding 30 kg (66 pounds), as follows:

- For liquids, inner packaging not over 4.0 L (1 gallon) net capacity each, packed in strong outer packaging.
- For solids, inner packaging not over 5.0 kg (11 pounds) net capacity each, packed in strong outer packaging.

TNRCC samples (Class 9) appear to meet the above-mentioned requirements.

The samples must be packed in a combination package. Appropriately completed shipping papers that identify the type and quantity of material must accompany the shipment. Samples packaged to meet limited quantity requirements must be indicated with the works "limited quantity" or "Ltd Qty" following the proper shipping name.

The quantities and types of packaging stated above are for ground transportation only. Therefore, samples that we know or reasonably suspect to be hazardous should be packaged appropriately and specified for ground transportation only. Remember if samples are shipped on Wednesdays or Thursday, be sure to confirm that they will reach the laboratory by Friday morning.

It is important to note that 49 CFR 173.24 prohibits hazardous material leaking in or from a package. Such conditions warrant a DOT official placing the vehicle, carrying the package, out of service until the condition is resolved. A DOT investigator may assume that a leaking cooler, containing even a limited quantity of hazardous material, to be a violation of 49 CFR 173.24 and place the vehicle out of service. Therefore, extra care should be taken when packing the cooler so the melted ice does not leak out.

Remember all packages must be accompanied by the COC. A complete address of the sender and the receiving laboratory must legibly appear on each package. When sent by mail, register the package with return receipt requested. When sent by common carrier, obtain a copy of the bill of lading.

Post office receipts and bill of lading copies may be used as part of the COC documentation. The shipper of the samples may wish to sign the shipping paper; (Name of IHW Investigator) for TNRCC.

Sources:

TNRCC Field Operations Division, TNRCC Solid Waste Inspection Manual, September 1997.

TNRCC RCRA Quality Assurance Project Plan, dated August 1998.

TNRCC IOM, Summary & Recommendations of Shipment Methods of Samples Known or Suspected to be Hazardous, dated 12/5/97.

Ray Murray, TNRCC, personal interview, January 1999.

TNRCC Field Investigator's Laboratory Training Manual, Terry Mills, Rev.03, dated 01/01/97. Barry Kalda, TNRCC, personal interview on 2/4/99.

DOT Information Center (1-800-467-4922), Arthur Pollock, personal interview on 2/5/99. Chris Barry, Maxim Technologies Inc., personal interview, 2/5/99.

Table 6-1SAMPLING EQUIPMENT FOR VARIOUS WASTE MEDIA

OPEN DRUMS:	Glass Tubing, Drum Pump, Small Sample Jar
TANKS:	Oil Thief, Bucket, Glass Tubing, Bailer
WASTE PILE:	Plastic Scoop, Trier, Shovel
SOILS:	Bucket Auger, Shovel, Plastic Scoop
IMPOUNDMENTS:	Pond Sampler, Bucket
AIR:	Organic Vapor Analyzer, Personal Air Sampler, 0_2 Meter, Explosimeter
WELL:	Hydrocarbon Bailer, PVC Bailer, Stainless Steel Bailer, Small Sample Jar, Submersible Sampling Pump

TABLE 6-2 SAMPLING VOLUMES

		RECOMME	NDED
		RECOMMENDED	MIN. VOLUME
PARAMETER	MEDIA TYPE	CONTAINER	or MASS
WET CHEMI	STRY		
Cyanide (a)	Liquid	1 liter cube container	1 liter
Cyanide (a)	Soil or Solid	1 liter glass jar (b)	1 kg(c)
Phenol	Liquid	1 liter glass jar (b)	1 liter
Phenol	Soil or Solid	1 liter glass jar (b)	1 kg(c)
COD (d)	Liquid	1 liter cube container	1 liter
COD (d)	Soil or Solid	1 liter glass jar (b)	1 kg(c)
Total Metals	Liquid	1 liter cube container	1 liter
Total Metals	Soil or Solid	1 liter glass jar (b)	1 kg(c)
Leachates	Liquid	1 liter glass jar (b)	1 liter
Leachates	Soil or Solid	1 liter glass jar (b)	1 kg(c)
Flash Point Flash Point	Liquid Solid or Soil	1 liter glass jar (b) Do not analyze flash p	300-500ml oint on solids
Corrosivity	Liquid	1 liter glass jar (b)	1 liter
Corrosivity	Solid or Soil	Do not analyze corrosi	
	Solid of Soli	Do not anaryze conosi	vity on solids
ORGANICS			
GC/MS (e)	Liquid (f)	1 liter glass jar (b)	1 liter
GC/MS (e)	Liquid (g)	1 liter glass jar (b) or VOA vial	40-200ml
GC/MS (e)	Soil or Solid (f)	1 liter glass jar (b)	1 kg(c)
GC/MS (e)	Soil or Solid (g)	1 liter glass jar (b)	100-300 gram
VOA (h)	Liquid	VOA vial	40 ml (i)
VOA (h)	Solid	1 liter glass jar (b) or VOA vial	50 grams (j)

(a) Warn the laboratory.

(b) The lids for the glass jars should be teflon lined.

(c) One kilogram is approximately one-half liter solid or soil.

(d) Chemical Oxygen Demand.

(e) Gas chromatography or Gas Chromatography/Mass Spectrophotometry.

- (f) Low concentrations of organics.
- (g) High concentrations of organics.
- (h) Volatile Organics Analysis.
- (i) Leave no headspace when sealing vials (no air bubbles). Collect duplicate vials.
- (j) More difficult than liquid, can also use VOA vial.

Table 6-2a

Sample Volumes Collected, Containers, Preservatives, & Holding Times

*HDPE = High Density Polyethylene LDPE = Low density Polyethylene CWM = Clear wide mouth *IF PRESERVATIVES ARE ADDED TO CONTAINERS PRIOR TO SAMPLE COLLECTION, ONLY HDPE OR GLASS CONTAINERS MAY BE USED.

PARAMETER	SOLID	SAMPLES	LIQU	ID (AQUEOUS) SA	AMPLES	MAXIMUM
EPA Method Number Standard Methods / SW-846	SAM PLE WT	CONTAI NER TYPE	SAMPLE VOLUME*	CONTAINER TYPE	PRESERVATI VE	HOLDING TIME
ORGANICS						
SEMI-VOLATILES - 6410/8270C	50 G	8 OZ CWM	1 Lx2	2-1 L/1-80 OZ	COOL 4º C	14/7 DAYS
VOA (VOLATILES) - 6210/8260B	20 G	4 OZ CWM	40 MLx2	2-40 ML	pH<2 NaHSO ₄ ; COOL 4º C	14 DAYS
METALS (DO NOT US	E BROW	N GLASS)				
METALS Part 3000 Methods / 7000 series	20 G	8 OZ CWM	1L	1 L HDPE/LDPE	pH<2 HNO ₃	6 MONTHS/28 DAYS
MERCURY - 3500-Hg / 7471	20 G	8 OZ CWM	1L	1 L HDPE/LDPE	pH<2 HNO ₃	28 DAYS
CHROMIUM VI - 3500-Cr/7195-7199	20 G	8 OZ CWM	1L	1 L HDPE/LDPE	COOL 4º C	24 HOURS/ 28 DAYS (soils)
TCLP 1311	200 G	16 OZ CWM	-	-	COOL 4º C	Depends on parameters
IGNITABILITY - 1010	10 G	8 OZ CWM	1L	8 OZ CWM	NONE	NONE
INORGANICS (ALL CO	DOLED 1	CO 4º C)				
ALKALINITY - 310.1 2320			1L	1L HDPE/LDPE	COOL 4º C	14 DAYS
AMMONIA - 350.14500-NH ₃			1L	1 L HDPE/LDPE	pH<2 H ₂ SO ₄	28 DAYS
BOD/CBOD - 405.1 5210/5220			1 GAL	1 GAL HDPE/LDPE	COOL 4º C	48 HOURS

COD - (Hach)	1L	1L HDPE/LDPE	pH<2 H ₂ SO ₄	28 DAYS
CHLORIDE - 325.2 4500-Ct	1L	1L HDPE/LDPE	NONE REQ'D	28 DAYS
CYANIDE - 335.2 4500-CN ⁻	(2) 1L	(2)1 L HDPE/LDPE	pH>12 NaOH (Ascorbic Acid if Chlorinated)	14 DAYS
FLUORIDE - 340.2 4500-F	1L	1L HDPE/LDPE	NONE REQ'D	28 DAYS
HARDNESS -130.2 2340	1L	1L HDPE/LDPE	pH<2 HNO ₃ OR H ₂ SO ₄	6 MONTHS
KJELDAHL NITROGEN - 351.2 4500-N _{org}	1L	1 L HDPE/LDPE	pH<2 H ₂ SO ₄	28 DAYS
NITRATE - 353.2 4500-NO ₃ G	1L	1L HDPE/LDPE	COOL 4º C	48 HOURS
NITRATE-NITRITE - 353.2 4500-NO	1L	1L HDPE/LDPE	pH<2 H ₂ SO ₄	28 DAYS
NITRITE - 353.2 4500-NO ₂ G	1L	1L HDPE/LDPE	COOL 4° C	48 HOURS
OIL & GREASE - 1664	1 L	1 L GLASS	pH<2 H ₂ SO ₄	28 DAYS
PHENOLS - 420.1 6420/8041,8270	1L	1 L BROWN GLASS OR CLEAR IF STORED IN DARK	pH<2 H ₂ SO ₄	28 DAYS
PHOSPHATE,ORTH O - 365.1 4500-P	1L	1L HDPE/LDPE	COOL 4º C	48 HOURS
PHOSPHATE, TOTAL- 365.44500-P	1L	1L HDPE/LDPE	pH<2 H ₂ SO ₄	28 DAYS
SULFATE - (Hach 8051)	1L	1L HDPE/LDPE	COOL 4º C	28 DAYS
TDS - 160.1 2540	1L	1L HDPE/LDPE	COOL 4º C	7 DAYS

TPH - TNRCC 1005	10 G	4 OZ CWM	30 ML	40 ML	pH < 2, HCL, COOL 4º C	14/7 DAYS
TOC - 415.2 5310	10 G	4 OZ CWM	1L	1L GLASS	pH<2 H ₂ SO ₄	28 DAYS
TSS - 160.2 2540			1L	1L HDPE/LDPE	COOL 4º C	7 DAYS

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Table 6-3 POSSIBLE CHEMICAL PARAMETERS TO REQUEST

1. ARE THE COMPONENTS OF THE WASTE KNOWN OR SUSPECTED? YES - PROCEED TO 3 BELOW. NO - PROCEED TO 2 BELOW.

2. IS THE SITE STABLE? (Will the material be there in the near future?)

YES - In a stable situation, time is on our side. It is possible to collect a composite sample for a full spectrum of organic or metal analyses or to collect a minimal number of samples for basic organic parameters. Later, when specific problem areas are discovered, more samples can be collected for advanced organic parameters or for the suspected metals. Proceed to 3 below.

NO - In this situation (unknown components at an unstable site) all points of interest will need to be sampled at once. Since the contaminants are not known, a balance between the number of samples and the number of parameters must be developed by the investigator. Consider both organic and inorganic parameters in 3 below.

3. ARE THE CONTAMINANTS ORGANIC, INORGANIC, OR BOTH?

ORGANIC - Basic organic parameters include, COD, phenol, and flash. point. Advanced organic parameters include GC, GC/MS, volatile organic analysis (VOA). Virtually all samples can be analyzed for the basic organic parameters. Request those parameters which will meet the investigation objectives and give information on the general type of organic compounds with a molecular weight with less than 105 AMU. Styrene is one of the heaviest organic compounds which is best analyzed by VOA. GC is requested for suspected contaminants which include toxaphene, PCBs, pentachlorophenol, or samples which few organic components suspected. GC/MS is best utilized in analyzing complex mixtures of organic compounds (e.g., resins, esters, polymers, monomers) which are of interest to investigators but which cannot be analyzed by gas chromatography. Call the Quality Assurance Office if you have any questions.

INORGANIC - This will include pH, COD, cyanide, and metals analysis. Aqueous samples are preserved with approved chemicals but soil and solid waste samples are not.



CHAIN OF CUSTOD Y Field Designation

FIGURE 6-1

FIGURE 6-2 CHAIN OF CUSTODY RECORD

TEXAS MATURAL FESSIONACE CONNESTION	A TALL		ain o	f Cus	Chain of Custody Record	Heco	brd	0123456
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Region: Organization #	₩Q ₩	PCA Code:	ode: 93677	Progr	Program	·	Sampler telephone number: (13-7) // c/- 729 O	729.0
E-Mail ID: TDE	Sampter:	(signatu	te	$ \sim$			Samplar: (please print clearly)	
Latitute Sample Number Sample	Date	Time ^{c.}	# of Graty Bolizas Comp.	Malrix D. L.3.M.O.Y	C12 9H	- O I I I	Anatyses Requested	AEMARKS
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FIGURE 6-3 CHAIN OF CUSTODY LABELS

TNRCC Sample ID: 0123456-01 Preservative: _HCl __(H2SD4 __NaOH __HNO3 _ascorbic ácic __zinc acetate _FAS __th/osulfate None Other: Ide in Cooler Analysis/Comments: Volatiks TPR RERA METALS (Total) TNRCC TNRCC Sample ID: 0123456-02 Preservative: Preservative: _ HCJ _____H2SO4 ____NaOH ____HNO3 ____zscorbic actio _____zinc acetare ___FAS ____thiosolitate X None Other Ice in Couler

Analysis/Comments: Velztiles TPH RERA METALS (Totals)

\$

Sample ID: 0123456-05 HCI _ H2SO4 _ NaCH _ HNO3 ascortic acid _ zinc ace:ate FAS _ thiosulfate XNone Other: ILE in Cooler Analysis/Comments: Volatiles TPH. RERA METALS (Total)

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FIGURE 6-4 REQUEST FOR ANALYSIS

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Request for Analysis

COC Number: 0123456

	<u>.</u>			···· 1	<u>SAMP</u>	LE 11	<u> </u>		-	
ANAL YSIS	<u>-01</u>	-02	<u>03</u>	-04	-05	-06	<u>±07</u>	-0.5	[▶] -09	-10
8015B: Nonhelogenated Organics Using GC/FID						4				
8021B: Aromatic & Halogerated Velatiles by GC Using PID / ECD					Y.m.					
BTEX						Ÿ		N.		
8031A: Organochloring Pesticides Using GC				X		À				
8032: PCE: Using GC					Ň		à.		-	
8100: Polynuclear Aromatic Hydrocarbans (PAHs)										
\$121: Chlorinated Hydrocarbons by GC (Capillary Column)			13			S *				
8141A: Organophosphorae Compound: by GC (Capillary)		i k			Trea					
8131A: Chlorinated Herbicides () GC				Ø.						
\$250B: VESDOREN/S		\checkmark	1							
8260B: VCA by GC (0.5. Library Search for Tentatively for the Compound: (1108)										
TCLP y Sotiles	۶									\vdash
82709: Semivoletile Organiz										
2200 Semivalstile Orbitic Semicords by CCMS + TICs								· ·		
9020B: Total Organic Halides (TOX)										
TX1005: TPH	\checkmark	\checkmark								
413.1: TPH										

FIGURE 6-4a REQUEST FOR ANALYSIS