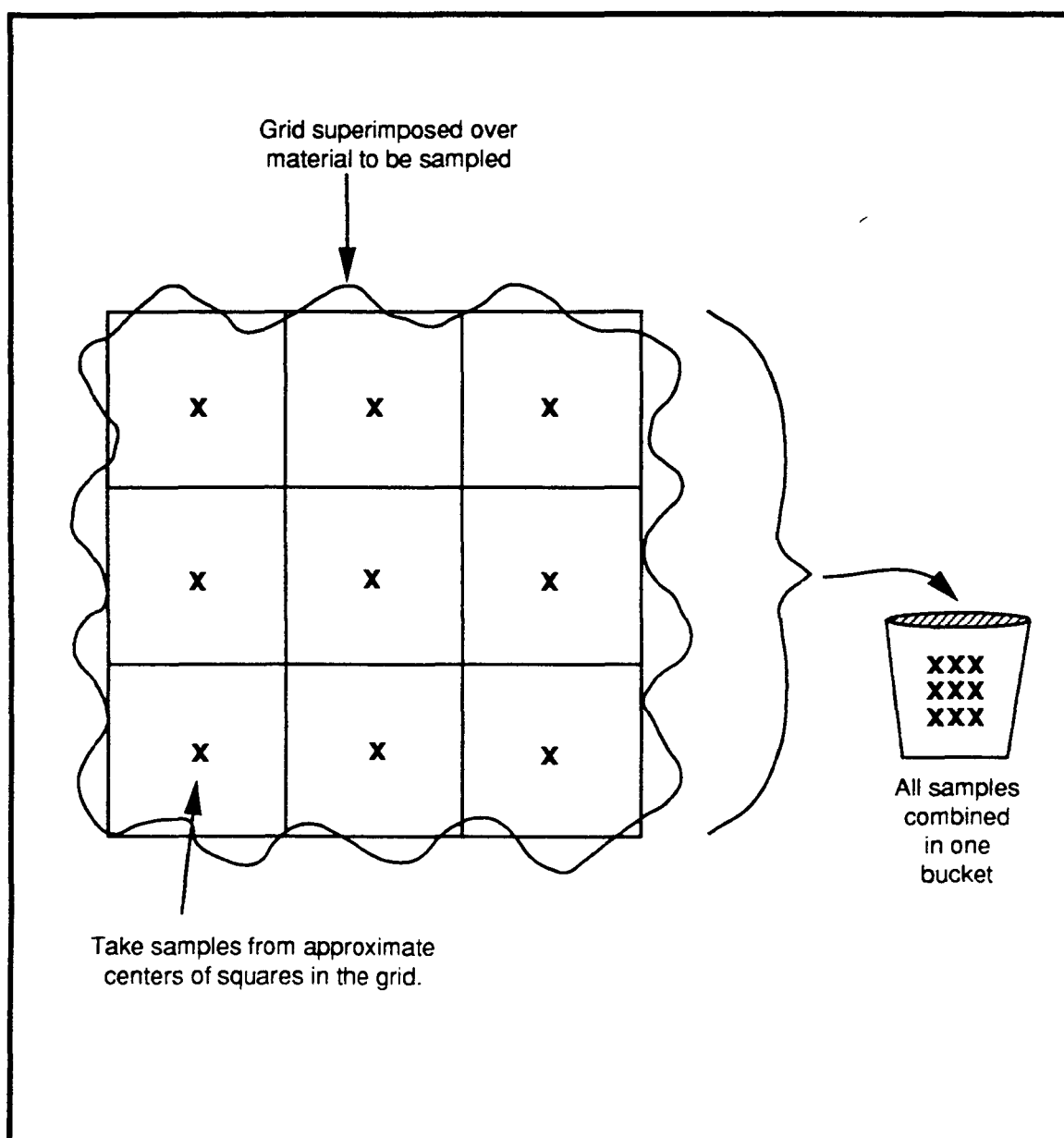


Office of Prevention, Pesticides, and Toxic Substances



SAMPLING GUIDANCE FOR SCRAP METAL SHREDDERS

Field Manual



**SAMPLING GUIDANCE FOR SCRAP
METAL SHREDDERS**

Field Manual

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1. INTRODUCTION

Purpose of this Document. The purpose of this document is to provide basic instructions for collecting and statistically analyzing samples of materials that are produced as a result of shredding automobiles, refrigerators, washing machines, and other metal objects. Shredders constitute an important component of this country's environmental management program, annually recycling 6-9 million cars, 19 million appliances, and 10 million tons of scrap metal. Unfortunately, the by-products of these recycling operations may, in some cases, contain significant concentrations of polychlorinated biphenyl's (PCBs) or other toxic substances, notably lead and cadmium. As a result, communities, environmental agencies, and shredder operators have expressed concern over the possibility of contamination in waste products generated at shredder sites and have indicated a need for guidance in assessing the presence of toxic substances in these materials.

Previous Studies. Several States have done exploratory studies of shredder sites. Analysis of approximately 200 samples of waste materials collected at shredder sites have revealed concentrations of PCBs ranging from 0 to 1,242 parts per million (ppm).

Based on concerns raised by these studies, the U.S. Environmental Protection Agency (USEPA) has gathered samples of various waste materials at seven shredder sites distributed across the United States.¹ In this study, analysis of samples of PCBs revealed concentrations ranging as high as 870 ppm. The same study found concentrations of lead and cadmium ranging as high as 43,000 ppm and 200 ppm, respectively. Information from these prior studies, particularly the one done by the USEPA, has been used in developing the sampling methods discussed in this document.

Shredder Output Streams. Shredders are very large machines that convert autos, truck bodies and other light gauge metal objects into fist size or smaller pieces of scrap metal.² A typical shredder operation is depicted schematically in Figure 1. The actual "shredding"

¹ *PCB, Lead, and Cadmium Levels in Shredder Waste Materials: A Pilot Study.* USEPA, Office of Toxic Substances. EPA 560/5-90-008B. 1991.

² The technical background for this section is based on material taken from *PCB, Lead, and Cadmium Levels in Shredder Waste Materials: A Pilot Study*, *ibid.*; on Chapters 1 and 2 of *Analytical Chemistry of PCBs*, by Mitchell D. Erickson, Butterworth Publishers, 1986; and on conversations with shredder operators and environmental consultants specializing in scrap metal recycling.

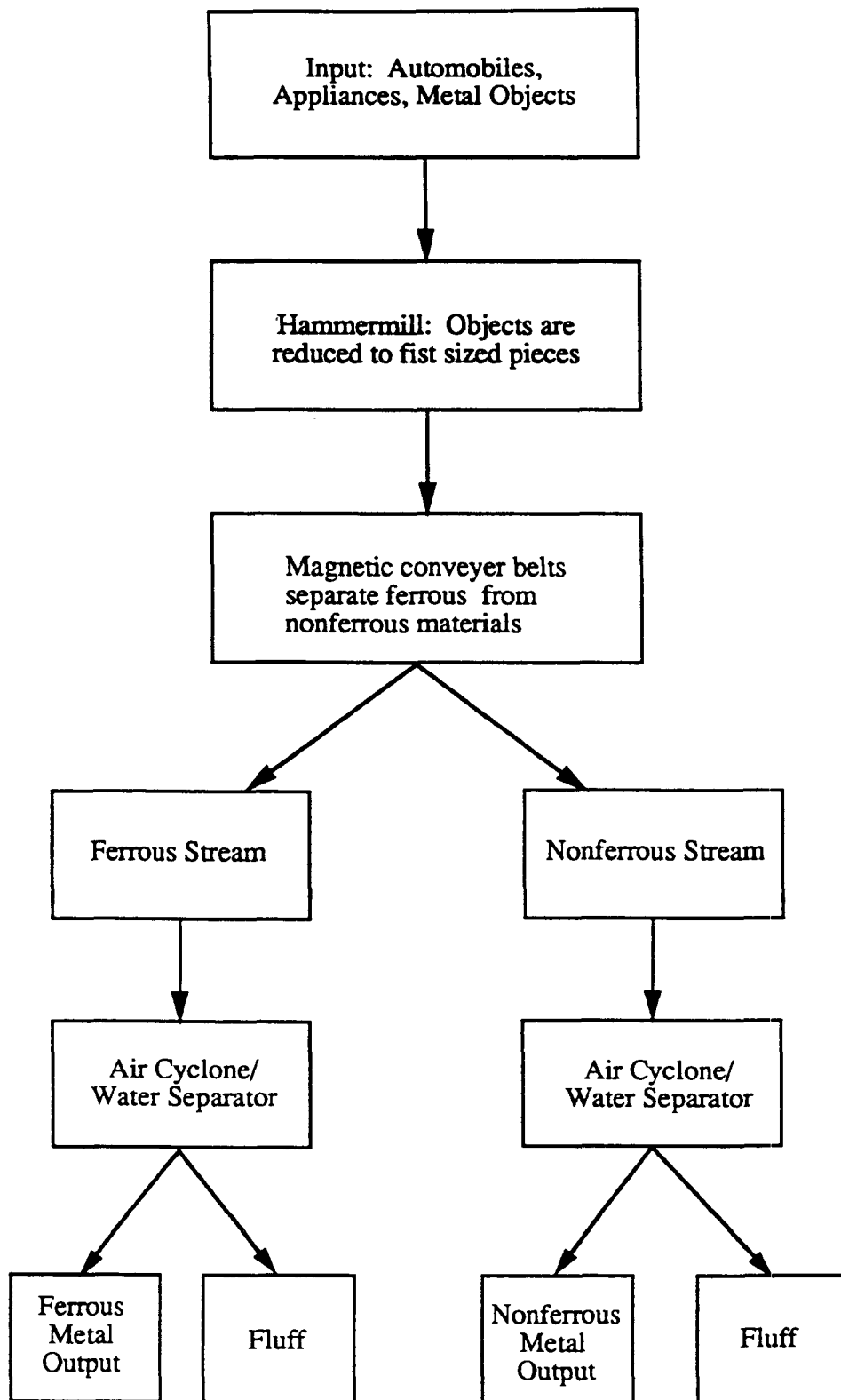


Figure 1. Schematic illustration of shredder process

is accomplished by a large hammer mill, after which the resulting output is sorted into three main output streams:

- Ferrous metals,
- Nonferrous metals, and
- Fluff.

Fluff is extremely heterogeneous. While it consists largely of plastic and foam, it may also contain pieces of metal, rubber, fabric, wire, and other materials. In general, it has a fibrous, "fluffy" appearance, at least when viewed from a distance. The initial separation into ferrous and nonferrous materials is carried out using magnetic devices. After this step, metal and fluff are separated using either air cyclone or water separation processes. In addition, nonferrous metals are often subjected to some hand-sorting as well. Both ferrous and nonferrous metals are recycled, while fluff is typically deposited in landfills.

It should be noted that this is a description of a "typical" shredder, but there are many types of shredders and the instructions in this document may have to be adapted for special circumstances at a given location.

How PCBs Enter Output Streams. PCBs enter output streams when materials containing PCB-bearing fluids are shredded. PCB-bearing fluids have been used in the construction of capacitors, transformers, electric motors, air conditioners, and hydraulic devices. PCBs have also been used as additives in pesticides, paints, sealants, and plastics.

The materials processed at shredder sites may be roughly categorized as follows:

- Motor vehicles, including passenger cars, light trucks, vans and small school buses: In such vehicles, PCBs may be found in paint, hydraulic fluids, oil capacitors, plastic materials, and in oily dust accumulated from roads.
- Appliances, including refrigerators, washers, dryers, dishwashers, freezers, ranges, air-conditioners, microwaves, and hot water heaters: These materials are generally called "white goods." In white goods, PCBs may be found in capacitors and electric motors.
- Other materials, such as scrap metals, or industrial or office equipment: PCBs might be found in oil-filled capacitors, plastics, paints, and adhesives.

When objects containing PCB-bearing fluids are shredded, the fluids are dispersed and may be absorbed by the fluff, or the fluids may coat metal and plastic objects. Similarly, when plastics or painted objects are shredded, PCBs in particulate form may enter the fluff output stream. In any case, the concentration of PCBs in (or on) materials produced at shredder sites may pose an unreasonable risk to health or the environment.

PCBs have been regulated by the Toxic Substances Control Act (TSCA) since 1976. According to these regulations, materials that contain PCBs in a concentration of 50 ppm or more must be disposed of in a chemical waste landfill, boiler or incinerator approved under TSCA. EPA has determined that fluff is regulated under TSCA, 40 C.F.R, Part 761. The U.S. Shredding Industry produces approximately three million tons of fluff a year. If widespread contamination were found and the materials were deposited in TSCA landfills, the demand for these landfills could exceed their capacity due to the volume of fluff.

Where to Look for PCBs and Other Toxic Substances. Very little is known about the volume and distribution of PCBs at shredder sites. It is generally suspected that PCBs are much more likely to enter output streams when processing white goods than motor vehicles because of the higher prevalence of electric motors in the former. Because of this, many operators refuse to process white goods, while others accept them only if the motors have been removed. Those operators that do process white goods typically "mix" them with motor vehicles, usually at a rate of about 10% or less white goods (by weight).

When PCBs are present at a given site, it is generally expected that they would be found in fluff because of its absorbent nature. While metal output may be coated with PCB-bearing fluids, it seems unlikely that the coating would contain enough PCBs to constitute a health hazard. PCBs may be present in the soil at shredder sites, particularly in locations where fluff accumulates or is moved for storage. However, it must be stressed that very little is known about levels of PCBs at shredder sites and the possible contamination of materials produced by shredders.

Even less is known about other toxic substances that may be present at shredder sites. Lead and cadmium may enter output streams from paint and metal plating on component parts in motor vehicles. Unlike PCBs, lead and cadmium are not typically suspended in fluids, but they might adhere to particles of fluff as materials are shredded.

Sampling Objectives. There are several possible objectives in sampling for PCBs. At the time of this writing, no one knows very much about the presence of PCBs at shredder sites. Large concentrations of PCBs have been identified in some samples that have been collected; some of these findings have been questioned, based on data collection procedures and/or analytical methods. Thus, agencies may wish to collect data at shredder sites in order to study the situation in their locality. In such studies, the objective is simply to gather data and make a preliminary assessment of possible contamination, as measured by the overall concentration of PCBs, without any preconceived ideas about whether such contamination exists.

Another objective is to monitor the output of one or more shredder sites. In this situation, the monitoring agency – which may be the shredder operator or an outside agency – develops a program of regular sampling and analysis of materials to assure that shredder output meets specified standards.

In the event that a shredder site or output from a site is established as being contaminated with PCBs – if large piles of stored fluff or the soil around the site are known to contain high concentrations of PCBs, for example – then it may become necessary for the site to undergo some form of clean-up or change in operating procedures. Thus, a third objective of sampling might be to collect data to verify that a site is free of PCBs.

The sampling procedures described in this document are intended to produce representative samples of fluff that will give reasonably accurate estimates of the overall concentration of PCBs in the material being sampled. The sampling methods are suitable for any of the objectives described above. The document primarily addresses analytical methods for exploratory studies; an appendix discusses analytical methods for monitoring and clean-up verification.

Contents of This Document. The document consists of three main parts. In Chapter 2, we will discuss procedures for selecting samples of fluff and other media at shredder sites. Next, in Chapter 3, we will discuss subsampling and other issues in laboratory testing. Finally, in Chapter 4, we will discuss statistical procedures for deriving conclusions after the data have been analyzed at the laboratory. The methods discussed in Chapter 4 are intended for exploratory studies undertaken to assess the extent of PCB contamination, if any, at one or more shredder sites. Analytical methods for regulatory procedures are discussed in an appendix.

This document is intended for users of all backgrounds and no special statistical knowledge is required. The statistical background and technical justification for the material presented here is given in a companion volume.¹

Cautions about Using This Document. This document consists of directions for collecting and analyzing samples of materials at shredder sites. The sampling plans, estimated sample size requirements, and the accuracy of statistical tests that are discussed in this document are based on data from samples collected at seven different shredder sites located throughout the United States. Although it is not likely, the data that you encounter at your shredder (or the site you are investigating) may differ substantially from the data used to develop the guidelines in this document. If this occurs, the sample sizes shown in tables in this document may yield results that are somewhat more or less precise than you would expect based on the parameters discussed in Section 4 and in the appendix.

¹*Sampling Guidance for Scrap Metal Shredders: Technical Background.* USEPA, Office of Pollution Prevention and Toxics. EPA/560/5-9 i-002.

2. SAMPLING PROCEDURES

2.1 Basic Sampling Guidelines

Overview. The purpose of the field sampling procedures described in this section is to estimate the overall concentration of PCBs, rather than to identify “hot spots” with high concentrations. Thus the sampling methods described here are intended to produce representative samples of fluff, since this material is generally considered to be the most likely to contain PCBs, if they are present at all.

Fluff is often stored in piles on the shredder site before being shipped to a landfill for disposal. We will differentiate between *stored fluff*, which is stored in piles at the shredder site, and *fresh fluff*, which is produced at the site while sampling is being done. In particular, we will describe different sampling procedures for stored and fresh fluff. The former may consist of very large piles which are difficult to access, while the latter is being continuously produced and is generally easier to sample.

In collecting samples, care should be taken to minimize the disruption of the normal operations of the shredder. This is important not only from the standpoint of maintaining good relations with the shredder operator, but also because the samples collected should, to the greatest extent possible, reflect the normal output of the shredder. If shredding procedures are altered in order to collect samples, the data collected may not reflect the usual PCB content (if any) of the shredder output streams.

How Large Should Samples Be? The materials present in fluff are very heterogeneous, and samples must be relatively large in volume to get a good cross-section of the types of materials present. In most cases, we suggest taking individual samples of about one gallon in size. Many of the sampling procedures we recommend require combining several samples of which each is one-half to one gallon in size. In any case, we recommend that the total volume of fluff collected at a site be at least five gallons.¹

Duration of the Sampling Period. When sampling from the stream of fresh fluff as it is being produced, the duration of the sampling period is an important consideration. Samples

¹ This recommendation is based on techniques for sampling heterogeneous materials presented in a seminar titled “Sampling Methodologies for Monitoring the Environment” by Pierre Gy and Francis Pitard Sampling Consultants.

may be collected only once during a visit, once each half-hour for several hours, or once each half-hour for an entire day. The longer the duration of the sampling period, the greater the likelihood of obtaining a representative sample of shredder output, since it is more likely that the materials shredded will be representative over a longer period. It is difficult to give fixed guidelines on how long to collect samples, but, in general, we suggest collecting samples of fresh shredder output each half-hour for a period of at least eight hours, or one working day. In any case, the general operating procedures followed at the shredder should be considered in deciding how long to make the sampling period and how frequently to collect samples. For example, if an operator runs white goods in the morning and automobiles in the afternoon, samples should be taken of each.

When different types of materials are recycled, the PCB content of the samples may vary considerably. Thus, regardless of the duration of the sampling period and the number of samples collected, the results of one day's sampling cannot be extrapolated to any other day unless the materials that are recycled on the two days are similar. Because of the variability in the materials shredded, high or low concentrations of PCBs may be found at one visit but not on a subsequent visit. Because of this fact, it is important that the samples collected at a site are as representative as possible of the usual activities of the shredding operation.

Collecting Representative Samples. The basic technique that we recommend for collecting samples requires two steps. First, a square, two-dimensional grid is superimposed over the material that is to be sampled, as shown in Figure 2. Stretching strings across the material is an efficient way of constructing the grid; the cells should be approximately equal in area. Next, samples should be taken from each cell in the grid and combined. This type of sampling is called *grid sampling*. It may be applied in sampling either fresh or stored fluff. The purpose of grid sampling is to obtain a sample that is spread throughout the material that is being sampled. Larger grids (e.g., four squares on each side) may be used, but a three-by-three grid is generally sufficient for this purpose.

When sampling material that is spread out in a grid, it is important to dig down into the material *to the bottom*. Finer particles will settle down and samples that are simply grabbed off the top will not be representative.

In order to collect more than one grid sample, use *replicated grid sampling*. Using this procedure, multiple samples are taken from each cell and combined in separate buckets, as illustrated in Figure 3. Each bucket is analyzed as an independent sample of material.

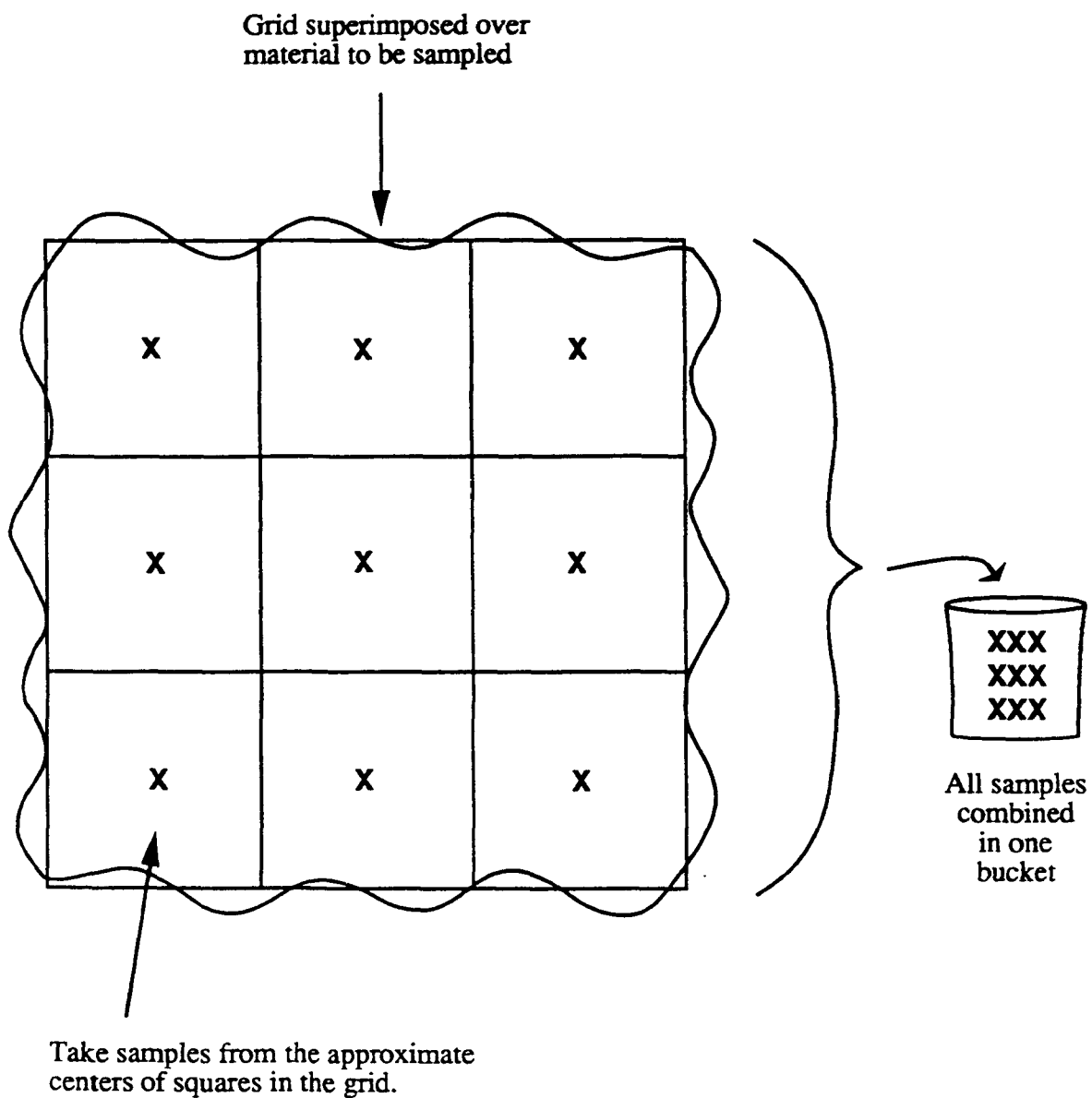


Figure 2. Illustration of grid sampling

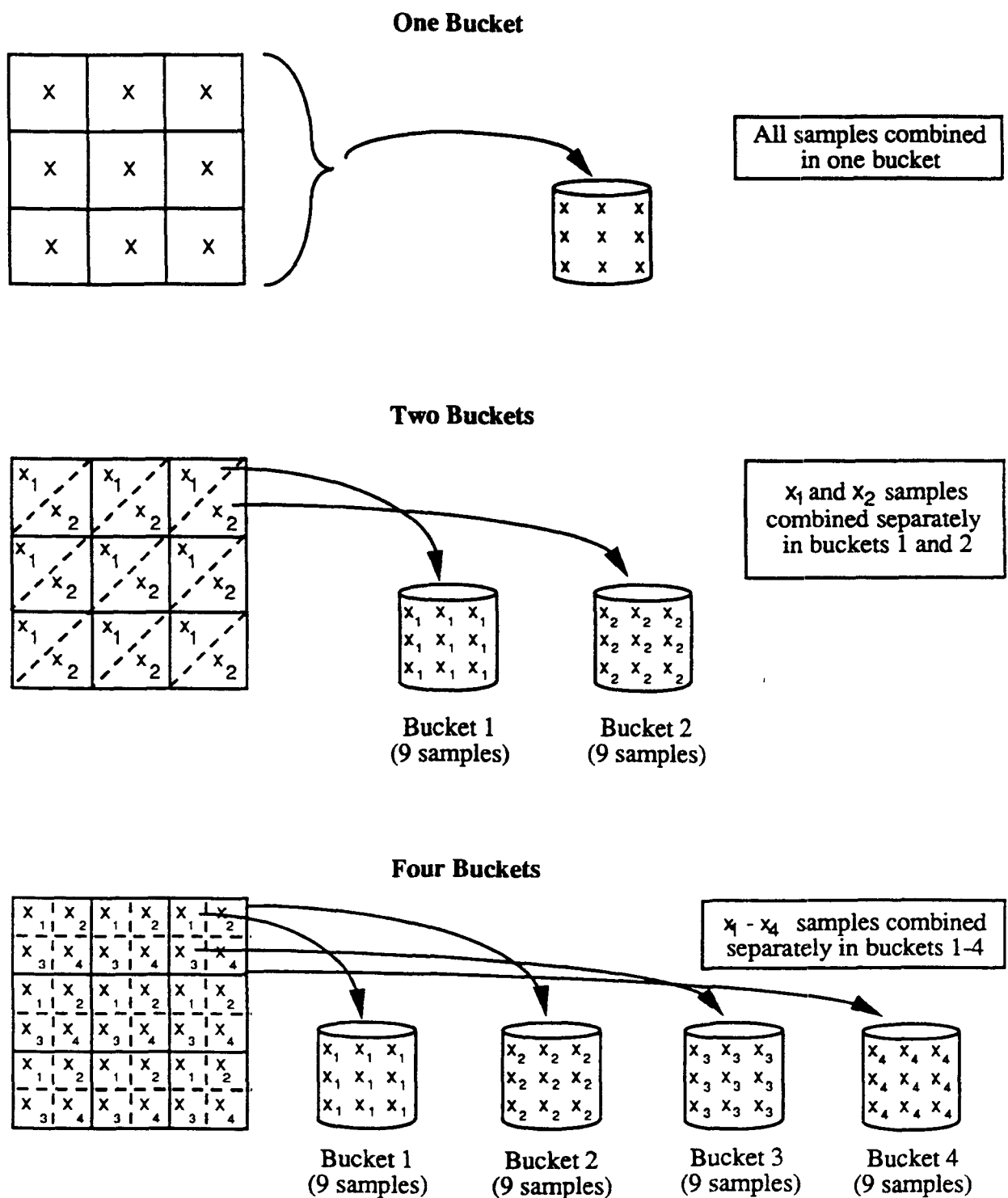


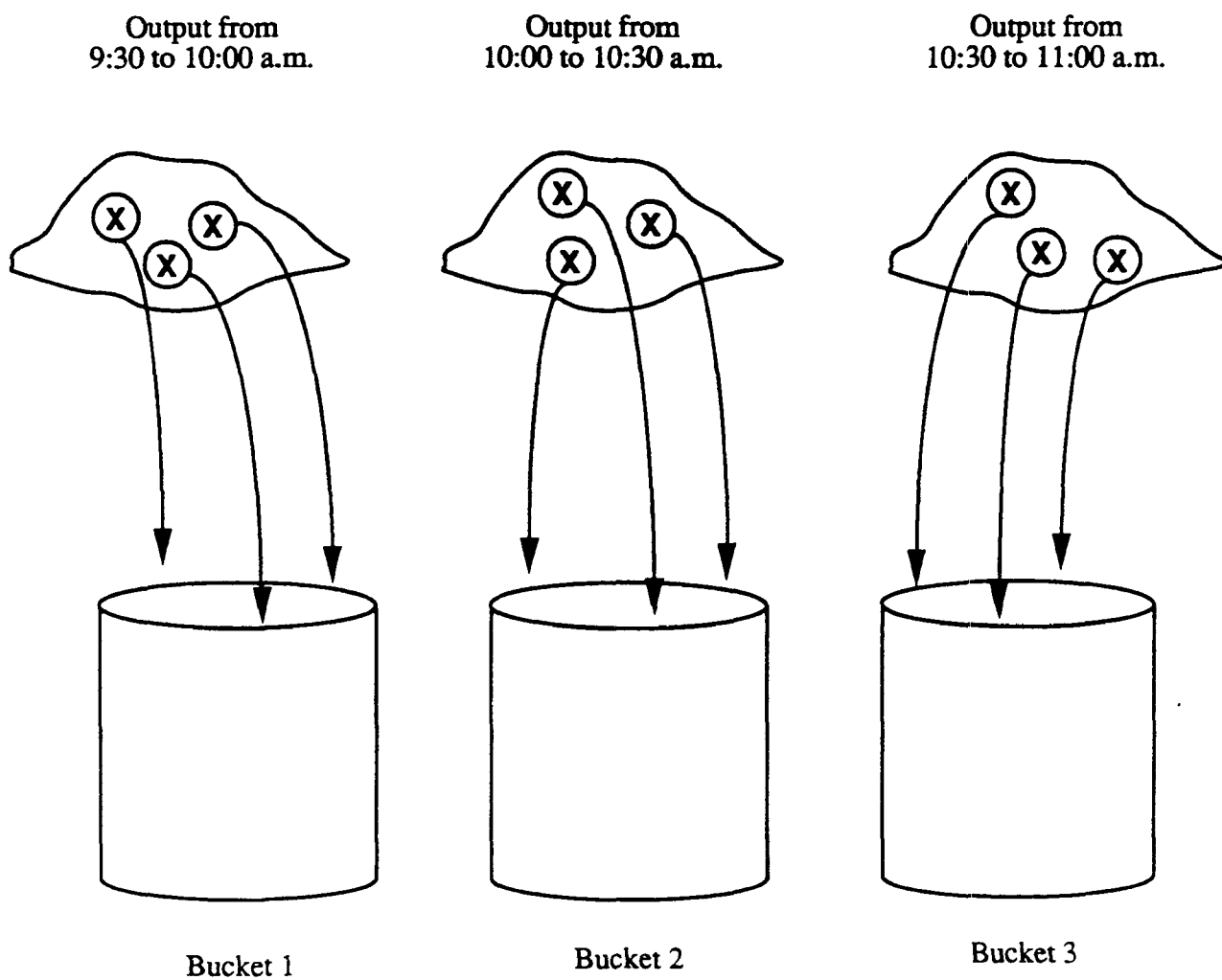
Figure 3. Replicated grid sampling

In some cases, grid sampling is not a practical option. For example, when sampling from large piles of fluff, it will be necessary to collect samples from various points in the pile without formally creating a grid. Detailed descriptions of how to sample stored fluff will be discussed below.

Sampling Over Time. When samples are collected from freshly produced fluff, samples must be collected at different times; for example, sampling might be done each half-hour over a 4- or 8-hour period. Figure 4 illustrates the basic technique for sampling over time. Here a separate grid sample is taken at each point in time, with each time period represented by a different bucket. Each bucket may consist of 1 gallon or more, but only one bucket per time period should be collected. If three samples are required, then samples should be collected at three different time periods (e.g., every 2 hours for a 6-hour period). If more samples are required, then either more time periods must be sampled (e.g., every hour for a 6-hour period) or samples must be collected for a longer duration (e.g., every 2 hours for a 12-hour period).

How Many Samples Should Be Collected? The number of samples that need to be collected depends on the accuracy required. As we will see in more detail later, about 10-20 samples should be sufficient for most purposes. For example, in sampling over time, 16 samples could be taken at half-hour intervals over the course of an 8-hour work day. These samples can be combined, using the technique of *compositing* which will be discussed later in Section 3.2, to reduce laboratory costs. Of course, fewer samples can be taken but at the risk of greater error. In Section 4, we will discuss the trade-offs between sample sizes and the reliability of conclusions.

What Equipment Should be Used? Because of the size and heterogeneity of materials that are produced at shredder sites, conventional core-sampling tools are usually of little use. Front-end loaders and backhoes may be useful for transporting and arranging materials, particularly if large amounts of fluff are involved. Similarly, trowels, rakes and shovels may be useful for smaller amounts of fluff. Because of the difficulty in manipulating fluff, it may be necessary to pick it up by hand and place "grab samples" manually in gallon containers. If available, a rotating gravity tumbler drum (RGTD) may be useful for mixing samples.



Samples Composited into Three Buckets

Figure 4: Sampling over time

Cleaning Equipment and Handling Samples. Whatever equipment is used, it must be clean in order to avoid contaminating the samples that are collected. Furthermore, equipment should be cleaned regularly, preferably after each sample is taken. To clean shovels, hoes, buckets, containers, and other equipment, soak them in dilute (20%) nitric acid and then rinse them three times, first with deionized water, then acetone, and finally hexane. Alternatively, steam cleaning can be used; if the steam condensate is free of PCBs, it can be disposed of easily. By comparison, disposal of solvents is always expensive.

If equipment is not cleaned, samples can become cross-contaminated. Cross-contamination occurs when PCBs from a sample that *is* contaminated are transmitted to a second sample which was *not previously* contaminated. This problem can occur when materials are not handled carefully and one sample leaks into another, or when equipment is not cleaned and a residue of PCBs builds up and is transmitted to multiple samples.

Besides keeping equipment clean, it is important to handle samples carefully. All samples should be clearly labelled, indicating the time, date and location. Samples should be stored in clean, sturdy containers. If samples are handled manually, gloves should be changed after collecting each sample.

Clearly, the cleaning of equipment can be cumbersome; moreover, it will be impractical in most circumstances to clean large equipment, such as backhoes. However, small equipment and containers should be cleaned as often as possible. While the risk may be small, it is in the best interests of both the shredder and environmental agencies that samples be as free as possible from cross-contamination. Cross-contamination can lead to erroneous conclusions about the level of toxic substances in the media. For example, stored fluff may be contaminated by fresh output, leading to the erroneous belief that the stored material may not be deposited in a sanitary landfill. Cross-contamination is especially serious when it occurs with samples from different sites, since questions of liability may be involved.

2.2 Sampling Fluff

General Guidelines. As described earlier, fluff is generated as a waste product which is separated from recyclable metals after the shredding operation. First, ferrous and nonferrous materials are separated using magnetic devices, and then fluff is separated from the metals either by using cyclone blowers or by washing with water, most commonly the former.

Fluff may either pile up below the cyclone separator or it may be removed to storage piles using conveyor belts.

There are generally three sources of fluff at a shredder site. First, fresh fluff is continuously being produced during the shredder operation. Second, there may be piles of stored fluff, although most shredder operators regularly ship fluff to avoid wasting storage space. Third, some fluff, which we will call *spillover*, is likely to have piled up around conveyor belts and other equipment. Although the basic sampling procedures are similar, we will give directions for sampling each form of fluff separately.

Fresh Fluff: Front-End Loader Assisted. We will describe two methods for sampling fresh fluff, the first of which involves the use of a front-end loader. This method is preferred for reasons of safety, sampling consistency, and minimal facility interruption.

Briefly, the front-end loader method involves (1) collecting the fluff in the front-end loader bucket as it is produced, (2) spreading the collected fluff out on the ground, and (3) taking samples from the fluff after it has been spread out on the ground. In order to use this method, you will need a front-end loader, which should have a safety cab and should be used only by an experienced operator. You will also need a clean space of ground on which to spread out the fluff. In some cases, it may be necessary to arrange with the operator to start and stop the shredder at appropriate intervals.

First, the front-end loader bucket should be positioned under the mouth of the cyclone (or the end of the conveyor belt, depending on which is used) during shredding to collect the fluff. The shredder should run until the bucket is full, typically about 3 minutes, or the equivalent of about two automobiles. (Note: If large objects are being shredded, it is preferable to process the entire object, rather than part of it.) After the shredder has stopped, move the front-end loader to an open, clean area for spreading the fluff. This area should be about 10 feet square, or large enough that the contents of the front-end loader can be spread evenly to a depth of about 1 foot.

Second, have the front-end loader operator spread the collected fluff on the ground in a square area to an even depth of about 1 foot, using the back of the bucket. Divide the square into nine roughly equal subsections, as shown in Figure 2. Take one-half gallon of material from the approximate center of each subsection, using a shovel and digging down into the material; combine the samples in the 5-gallon bucket. Smaller samples may be collected on a tarpaulin

placed under the cyclone or conveyor, moved to a clear area and then spread with a rake. For small samples, four roughly equal subsections may be used, with a half-gallon being selected from the center of each one.

At some sites, the fluff stream is fed continuously into rolloff boxes which can contain up to 20 cubic yards of material. In order to collect samples of fluff at these sites, the boxes must be pulled away from the output stream, which can then be collected using a front-end loader as described above.

Fresh Fluff Sampling Without a Front-End Loader. Arrange for the operator to shut down the line after shredding material for about 3 minutes. Take five one-gallon samples as follows. First, take four one-gallon samples by systematically sampling at four equidistant points around the perimeter of the pile, approximately 1 foot above the ground. Dig about 18 inches into the pile horizontally, or, depending on the size of the pile, far enough to obtain layers of fluff deposited at different times. Take the fifth sample from the center of the pile, digging down about a foot into the pile.

Stored Fluff. It is much more difficult to obtain representative samples from stored piles of fluff, but such samples are potentially more useful because they may be more representative of the normal output of the shredder. (We will assume that the stored pile to be sampled is large; small piles can be raked into a square shape, divided into nine roughly equal subsections, and sampled as described above for fresh fluff.) In collecting samples from stored piles of fluff, the objective is to obtain samples of the *oldest* fluff, the *deepest* fluff, and two samples of *surface* fluff. If a large pile of new fluff has been stored next to a smaller pile of old fluff, then the deepest fluff may not be the oldest. However, if the oldest fluff is also the deepest, take a sample half-way between the bottom and the surface in place of the deepest fluff. The procedures described below, which are illustrated in Figure 5, will provide a total of 20 one-gallon samples. To prevent cross-contamination between samples, collect one five-gallon bucket at a time.

First, take five one-gallon samples of surface fluff from the edge of the pile, at equal distances around the pile, one foot off the ground. Dig straight into the surface, including the actual surface material in the sample.

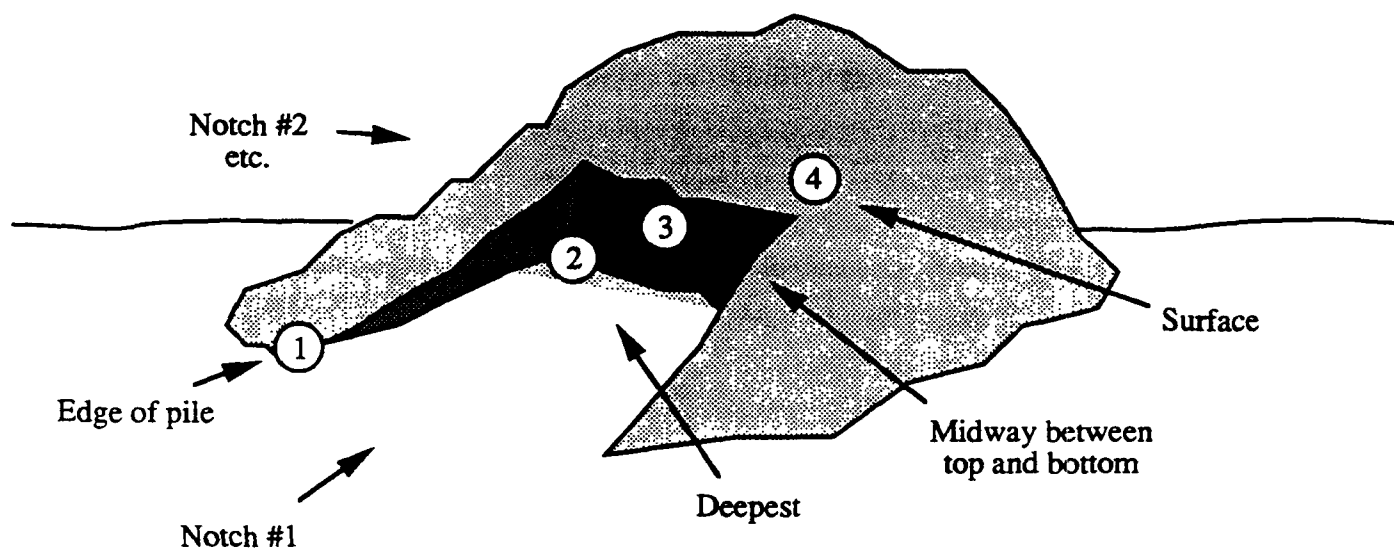


Figure 5. How to sample stored fluff

1. Take five one-gallon samples of fluff at equal distances around the edge of the pile.
2. Cut five notches at equal distances around the pile and take a one-gallon sample from the deepest fluff in each notch.
3. Take five one-gallon samples of the oldest fluff.
4. Take five one-gallon samples of fluff from the surface of the pile.

Second, use heavy moving equipment (such as a front-end loader) to cut five notches in the pile for the other samples, as shown in Figure 4. These notches should be located at equal distances along the perimeter of the pile, if possible. From each notch, take a one-gallon sample from the fluff that is deepest down in the pile. Some care may be required to get a sample of the deepest fluff in the notch, since fluff from the surface may fall down into the notch. One approach would be to have the operator remove upper layers of the pile before cutting the notch; it might also help to take the sample from the center of the notch, rather than the sides where material is more likely to fall into the notch. In making notches and collecting samples, remember that safety is a paramount consideration. Do not cut notches deeper than five feet in height. Proceed with caution at all times.

Third, collect five one-gallon samples of the oldest fluff. You will have to ask the shredder operator which fluff is the oldest. It may be a particular area of the fluff pile, or it may be the deepest layer. If it is not known which fluff is the oldest, then take a one-gallon sample from a point mid-way between the bottom of the pile and the surface in each of the notches.

Finally, collect five one-gallon samples of fluff from the surface of the pile at points near the center of the pile. The notches may provide easy access to points near the center of the pile.

As noted above, this procedure will result in 20 samples. After reviewing Section 4, which discusses analyzing the samples, you may decide that more samples are needed. The number of samples may be increased by taking more samples at each of the steps described above. For example, if *six* samples are taken from the perimeter, *six* notches are cut, etc., *six* samples of the deepest fluff are taken, and so forth, there will be 24 samples.

Spillover. During normal shredding operations, fluff will pile up along conveyor belts and cyclone separators. We will refer to this fluff as spillover. Spillover tends to consist of smaller particles, sometimes called "fines". Because these "fines" are suspected of being more susceptible to PCB contamination, you may want to take some samples of this material.

Inspect the area along the conveyor belt for spillover. Take five one-gallon samples of any spillover material along the conveyor belt at approximately equal distances. Mix these five one-gallon samples into one five-gallon bucket. If desired, repeat this procedure to fill additional buckets. In some cases, the pattern of spillover may not be regular enough to use this strategy. If necessary, identify the areas where spillover exists and take a one-gallon sample (or more) from

each location to achieve one five-gallon sample (or more) that is representative of the spillover material.

2.3 Quality Assurance

The Necessity for Quality Assurance. There are many sources of error in evaluating contamination by PCBs or other substances. First, since we are selecting samples of material to analyze, there is *sampling error*, which is due to the fact that not all of the material is being analyzed and thus there is variability in the results from one sample to another. (Please note that sampling “error” is a statistical term which reflects the natural variation that exists from one sample to another. This term does *not* imply any “error” on the part of those collecting the samples!) Second, there is *analytical error*, which results from the difficulty of accurately identifying and quantifying the substances present in a given sample of material. Third, there is the possibility of errors through *cross-contamination*, which results from PCBs (or other substances) being introduced into a sample during the collection process. For example, PCBs might be present in the buckets used for data collection and then transferred to the fluff during the process of collecting samples.

Below we describe two quality control procedures. The first, the use of field blanks, will help to detect the presence of cross-contamination. The second, the analysis of duplicate samples, will help to quantify analytical error.

More extensive treatment of quality control issues can be found in the following publications:

OTS Guidance Document for the Preparation of Quality Assurance Project Plans. USEPA, Office of Toxic Substances.

Test Methods for Evaluation Solid Waste. USEPA, Office of Solid Waste and Emergency Response. SW-846, Third Edition. 1986

Analytical Chemistry of PCBs, Mitchell D. Erickson. Butterworth Publishers, Stoneham, Massachusetts. 1986.

Field Blanks. Field blanks are materials that are known *not* to contain PCBs, but which are handled using the procedures specified for collecting fluff, soil or other materials which are suspected of being contaminated. When the field blanks are analyzed, they should not contain

any PCBs. Empty containers, such as buckets, should be taken to the site, opened for the duration of the time that sampling is done, and then closed and taken to the laboratory, where wipe samples can be taken and analyzed. This procedure will indicate whether containers were contaminated either before data collection or through improper handling. The use of field blanks helps protect the operator by indicating when samples are being collected improperly and possibly giving incorrect findings.

Duplicate Analyses. As a general practice, at least 10% of the samples selected should be analyzed in duplicate, meaning that the same sample (or parts of it) should be analyzed twice. In particular, if one sample has an extremely high concentration of PCBs relative to other samples, replicates should be analyzed for verification; Section 3 will discuss how replicates are formed. Preliminary studies suggest that laboratory or analytical error for the procedures described in this manual are, on average, about 30% of the estimated PCB level, ranging from 5% to 80%. If the results for replicates vary by more than this, it may be due to inadequate laboratory procedures.

3. PREPARATION FOR ANALYSIS

3.1 Preparing Fluff Samples for Laboratory Analysis

Overview. After samples are collected in the field, they must be prepared for laboratory analysis. Because of the extreme heterogeneity in some of these materials, one part of the sample can give an estimate which is not representative of the whole. In this section we will discuss procedures for splitting the collected samples into several replicates so that each replicate is representative of the original sample, containing the same components in approximately the same proportions. One or more of these replicates can then be analyzed to test for PCB contamination. The reason for creating such replicates is, first, to reduce the amount of material that is actually subjected to laboratory analysis, and, second, to create backup replicates for retesting if this becomes necessary. Altogether, at least five gallons of material should be prepared for analysis, with about 400-500 grams of this material actually undergoing analysis. In Section 3.2, we will discuss compositing, a technique for combining samples to reduce laboratory costs.

Step 1: Weigh the Fluff Sample. Determine the weight of the entire fluff sample. Since 400-500 grams of fluff are required for each replicate, weighing will indicate what fraction of each bucket of material will comprise a replicate. Generally, a five-gallon bucket of material will produce about eight replicates. However, if the weight of your fluff sample is substantially smaller than 3,200 grams or larger than 4,000 grams, then divide the weight of the sample by 450 to determine the number of replicates.

Step 2: Sort Out Large Pieces of Material. Pour the contents of the bucket onto a 9.5 mm screen above a laboratory tray or table with a nonabsorbent surface. Pieces that do not pass through the screen should be cut into pieces or milled until they are small enough to pass through the screen and then mixed into the sample. Larger pieces of material (metal, atypical wire, hard plastics) that cannot be cut with shears should be segregated. Smaller pieces of wire or other solid material that are distributed uniformly throughout the sample should remain with the sample.

Step 3: Divide Material into Replicates. Uniformly distribute the fluff which remains over the tray or table. This material will vary in composition, and dense granular materials (e.g., dirt, pulverized metal, plastics, glass, ceramics, etc.) will tend to settle below lighter material, such as shredded fabric and foam rubber. Care must be taken to ensure that these components of the fluff are uniformly distributed throughout the tray.

Using the information on the total weight of each sample, divide the fluff on the table into approximately equal parts, with the number of parts being equal to the number of replicates to be obtained. In most cases, you will divide the material on the table into eight roughly equal parts to form eight replicates.

Step 4: Cut Large Pieces and Distribute Among Replicates. In Step 2, large pieces that could not be easily cut were removed and set aside. Now cut these pieces with either tin snips or a hack saw, assuming that the materials can be cut using one of these tools, and distribute the pieces of the material equally among the replicates. If both cutting methods fail, the material should be analyzed separately, and any detected PCB levels should be prorated based on the number of replicates, the weight of the replicate, and the weight of the material. For example, suppose that eight replicates are produced, each weighing about 450 grams, and a large piece of material, weighing about 50 grams, cannot be cut. If the piece of material is analyzed and shown to have a PCB level of 30 ppm, then the revised PCB level for any replicate that is analyzed should be calculated as

$$\text{Revised PCB Level} = \frac{\frac{(30)(50)}{8} + (\text{Replicate PCBs})(450)}{\frac{(50)}{8} + (450)}.$$

Step 5: Place Replicates in Containers. Place each replicate in a container. Seal, label and number the container so that both the replicate number and original bucket number are included (e.g., Replicate #2 of 4 from Bucket #12).

3.2 Compositing

Because of the expense of analyzing samples at the laboratory, equal sized parts of two or more different samples are sometimes mixed together and sent to the laboratory for analysis as if the mixture were only one sample. Samples can also be composited after the preparatory steps described in Section 3.1; this method is preferable to compositing in the field, although it may be less cost effective. We will refer to the mixed sample as a *composite* sample (or simply a *composite*) and to the parts that were mixed together as *subsamples*. This procedure is illustrated in Figure 6. Because the subsamples have been mixed, the concentration of PCBs or other toxic substances in the composite sample should be roughly equal to the average of the concentrations

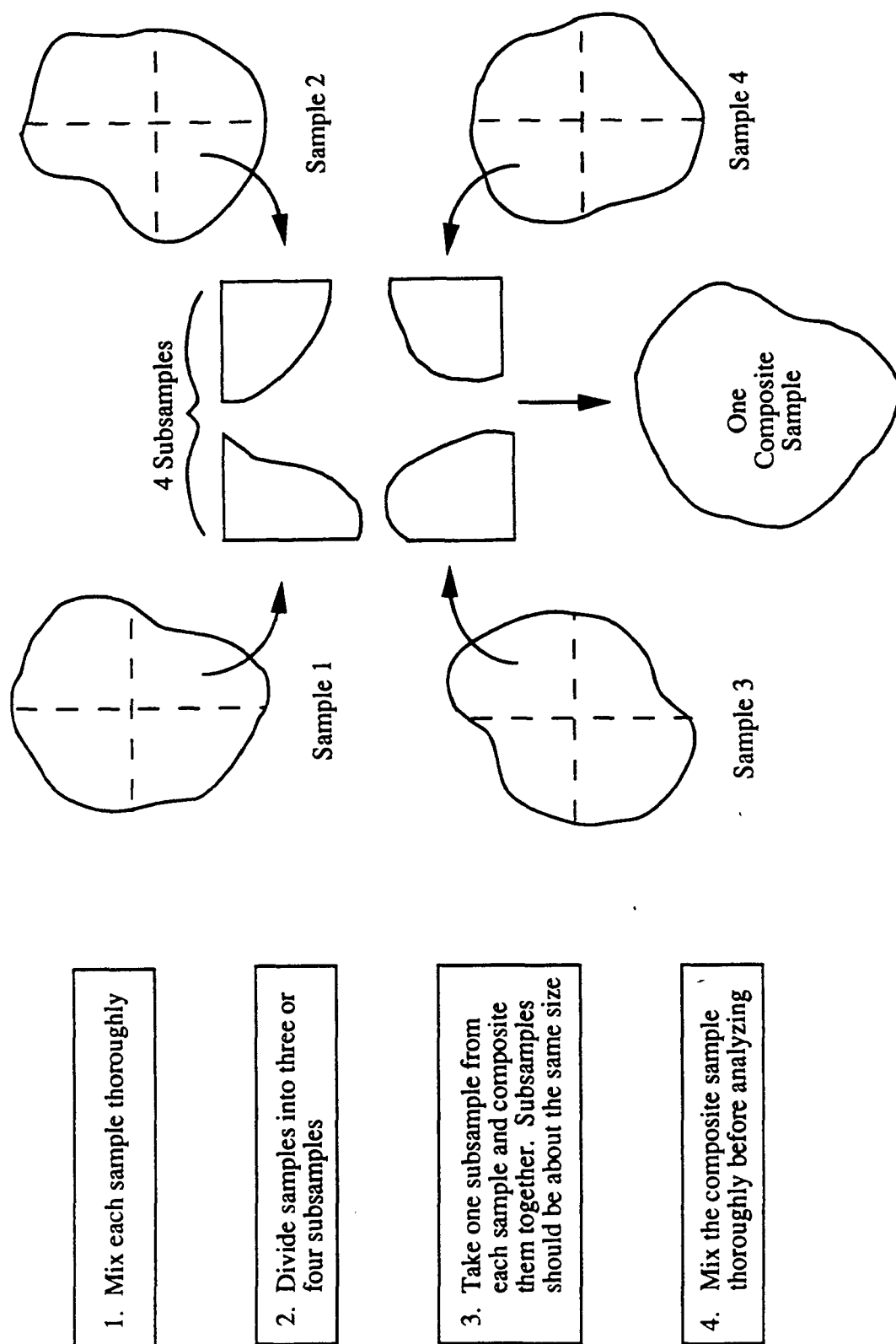


Figure 6. Guidelines for composing samples

that would have been obtained by analyzing the subsamples individually, even though the concentrations in the subsamples may vary substantially due to the heterogeneous nature of fluff. Assuming that laboratory errors are not large compared with sampling error – which is almost always the case when analyzing samples of fluff – compositing effectively reduces the cost of laboratory analysis while maintaining about the same level of accuracy as if the samples had been analyzed individually.

When forming composite samples, several general rules should be followed. First, mix each sample thoroughly *before* compositing. Second, divide each sample into three or four parts, or subsamples. All the subsamples must be of roughly equal size. One simple method for dividing the sample is to spread the sample out on a clean area and split it into two, then four, equal parts. Another method is to take scoops of the material and put the first scoop in the first subsample, the second scoop in the second subsample, the third in the third subsample, and so on, repeating the process until the material is exhausted. Finally, take one subsample from each of the samples and combine them to make up the composite sample. Mix the composite sample thoroughly.

If the samples are from different sites or different parts of a single shredder (e.g., stored and fresh fluff), then use only one subsample – not the entire sample – for compositing. If large concentrations of toxic substances are found, it may be desirable to analyze part of each sample separately.

Throughout the next section we will discuss the effects of compositing on various analytical procedures. While compositing is normally considered to involve *two* or more subsamples, it is preferable for simplicity in presenting tables to speak of composite samples which consist of *one* or more subsamples. For example, if four samples of fresh fluff are taken over a period of 4 hours (as described in Section 2.2), these samples might be analyzed as one composite of four subsamples, two composites of two subsamples each, or as four “composites” of one subsample each.

4. EVALUATING SAMPLE RESULTS

4.1 Possible Sources of Error

In Section 3.2 we noted that there are several possible sources of error in assessing contamination by PCBs or other toxic substances. Specifically, we discussed errors due to sampling, laboratory analysis, or cross-contamination when the samples are collected. Cross-contamination creates bias and can be avoided only by careful handling of materials. However, the first two types of errors can be taken into account by using the statistical methods described in this section. For example, if the laboratory analysis of five samples of fluff at a given site shows an average PCB concentration of 60 ppm, does this conclusively indicate that the entire output of fluff from that site actually contains more than 50 ppm? Is it possible that the actual concentration is 45 ppm and the difference (i.e., 60 ppm instead of 45 ppm) is due to sampling error and/or laboratory error? In this section we discuss a statistical procedure, called a confidence interval, for answering such questions.

Because of the errors associated with the selection and analysis of samples, we cannot be sure that the numerical value (e.g., an average PCB concentration of 60 ppm) resulting from a series of laboratory tests is *exactly* accurate. Instead we must use statistical analysis to obtain an interval (e.g., 50 to 70 ppm) which we are relatively sure is accurate. This interval is called a *confidence interval* and our degree of certainty is called the *level of confidence*. For example, based on the results of our statistical calculations, we may be 95% confident that the actual average concentration is somewhere between 50 and 70 ppm. In Section 4.2 we discuss the calculations necessary for making statements like this one.

4.2 Confidence Intervals

Overview. The objective of an exploratory study is to estimate the concentrations of PCBs or other toxic substances present in the output streams, soil, or other material at a given shredder site. Because of the sampling error and laboratory error, it is not possible to determine exactly the concentration of toxic substances. However, by using the methods in this section, you will be able to make statements such as, "As a result of our study, we are 95% certain that the concentration of PCBs in this pile of stored fluff is between 40 and 100 ppm." In this statement, the interval "between 40 and 100 ppm" is called a *confidence interval*. Because of sampling and

measurement errors, we are never sure of the exact concentration of a given substance in the material we are studying. By calculating confidence intervals, we obtain a range that is *likely* to contain the actual concentration. In this manual, all confidence intervals are calculated to have a 95% chance of being correct – i.e., of including the actual PCB concentration – and are thus called *95% confidence intervals*.

Preliminary Calculations. The first step is to make two basic calculations, the average and standard deviation of the samples. These calculations are illustrated in Worksheet 1. In the example given in Worksheet 1, 6 samples are analyzed and found to have measured PCB concentrations of 5, 15, 65, 11, 33, and 27 ppm, respectively. For these data, the average and standard deviation are 26 and 21.72 ppm.

Confidence Intervals for Concentrations. To find estimates of the actual concentration of PCBs or other substances, follow the calculations shown in Worksheet 2. For the example data shown in Worksheets 1 and 2, the lower and upper limits are 3.21 and 48.79 ppm, respectively, so that we are 95% certain that the estimated PCB level is between 3.21 ppm and 48.79 ppm.

Interpretation of Estimated Concentrations. What conclusions can be made based on the estimates that you have made? There are several ways to answer this first question, but the overriding concern should be whether estimated levels of PCBs and/or other toxic substances are considered to be too high. Suppose, for example, we regard 50 ppm to be an acceptable level of PCBs in shredder output. There are three possible cases:

- **Case 1:** The *upper limit* of the interval falls *below* 50 ppm. In this case, we are 95% certain that the level of PCBs is acceptable.
- **Case 2:** The *lower limit* of the interval falls *above* 50 ppm. In this case, we are 95% certain that the level of PCBs is *not* acceptable.
- **Case 3:** The *interval contains* 50 ppm. In this case we are unsure as to whether the level of PCBs is acceptable. If the interval is not too wide (e.g., 45 to 51 ppm) then we might be willing to assume that the level of PCBs is acceptable; otherwise, the study is inconclusive.

With regard to Case 3, it should be noted that most of the time it can be avoided by specifying a large enough sample size when planning the study; this problem will be discussed shortly. Furthermore, whenever it is necessary to make an absolute judgment about the safety of shredder

WORKSHEET 1: Calculation of Average and Standard Deviation

Example Data. Assume that 6 composite samples are analyzed and are estimated to have these PCB levels:

PCBs (ppm)	Squared PCBs
5.0	25.0
15.0	225.0
65.0	4,225.0
11.0	121.0
33.0	1,089.0
27.0	729.0

Step 1: Find the sum (Σ):

$$\Sigma x = 5 + 15 + \dots + 27 = 156.0.$$

Step 2: Find the sum of the squares:

$$\Sigma x^2 = 25 + 225 + \dots + 729 = 6,414.0.$$

Step 3: Find the average:

$$\text{Average} = \frac{\Sigma x}{\text{Sample Size}} = \frac{156.0}{6} = 26.0.$$

Step 4: Find the Standard Deviation:

$$\begin{aligned}\text{Variance} &= \frac{\Sigma x^2 - \frac{(\Sigma x)^2}{\text{Sample Size}}}{\text{Sample Size} - 1} \\ &= \frac{414.0 - \frac{(156.0)^2}{6}}{5} \\ &= 471.9.\end{aligned}$$

$$\text{Standard Deviation} = \sqrt{\text{Variance}} = 21.72.$$

WORKSHEET 2: Calculation of Confidence Intervals

Example Data. As in Worksheet 1, the example data consists of laboratory measurements from 6 composite samples, showing the following PCB levels:

PCBs (ppm)	Squared PCBs
5.0	25.0
15.0	225.0
65.0	4,225.0
11.0	121.0
33.0	1,089.0
27.0	729.0

Step 1: Find the average and standard deviation. Follow the directions in Worksheet 1. For the data shown above:

Average of Samples = 26.0

Standard Deviation = 21.72

Step 2: Estimation of Confidence Intervals. In Table 1, find the *t*-value for a sample size of 6, which is 2.57. Now make the following calculations:

$$\text{Average of Samples} - t\text{-value} \frac{\text{Standard Deviation}}{\sqrt{\text{Sample Size}}} = 26.0 - 2.57 \frac{21.72}{\sqrt{6}} = 3.21$$

and

$$\text{Average of Samples} + t\text{-value} \frac{\text{Standard Deviation}}{\sqrt{\text{Sample Size}}} = 26.0 + 2.57 \frac{21.72}{\sqrt{6}} = 48.79.$$

Step 3: Interpretation of Confidence Intervals. We are 95% certain that the actual PCB level is between 3.21 and 48.79.

output, then the hypothesis testing procedures described in the appendix should be used instead of the exploratory procedures discussed here.

In each of the preceding scenarios, we have used the expression “95% certain.” As we discussed earlier, there will always be some uncertainty as to the actual concentration of PCBs because of sampling and laboratory error. When we say that we are 95% certain that the level of PCBs is within a given range, we simply mean that there is a 5% chance that we are wrong. Put another way, this means that if we checked PCB levels at 20 sites (or at the same site at 20 different times) using the procedures described here, we could expect, on average, that our estimate for one of the sites would be wrong.

4.3 Sample Sizes

Sample Sizes and Relative Error for PCB Levels. Because of sampling and laboratory measurement error, we can never be certain of the exact concentration of PCBs. However, by increasing the number of samples analyzed, we can reduce the degree of error in our estimates. How many samples need to be taken? There is no universal answer to this question, but based on data from preliminary studies, we can make rough estimates of the level of error that can be expected from samples sizes ranging from 1 to 25.¹

When we select a sample and average the measured PCBs, there is always some difference between our *sample average* and the *true concentration* of PCBs in the sampled material. This difference represents error that is due to both sampling and laboratory analysis. The *relative error* is the absolute difference between the *sample* and *true* concentrations divided by the *true* value:

$$\text{Relative Error} = \left| \frac{\text{Sample Average} - \text{True Concentration}}{\text{True Concentration}} \right|.$$

Since the sample average is subject to random fluctuations, the relative error will vary also, and we will never know the relative error for any given sample. However, as the sample size increases,

¹ The estimates for standard errors, sample sizes and precision presented here are based on preliminary data from an EPA-supported study of 85 samples collected at seven shredder sites throughout the country and on a dataset of 200 samples collected and analyzed by various state and local agencies.

Table 1: t-values for confidence intervals

Number of composite samples	t-values
2	12.71
3	4.30
4	3.18
5	2.77
6	2.57
7	2.45
8	2.36
9	2.31
10	2.26
11	2.23
12	2.20
13	2.18
14	2.16
15	2.15
16	2.13
17	2.12
18	2.11
19	2.10
20	2.09
21	2.09
22	2.08
23	2.07
24	2.07
25	2.06
30	2.05
50	2.01
75	1.99
100	1.98
>100	1.96

*The values shown in the table are taken from Student's t distribution. This distribution is often used as a measure of uncertainty due to sampling and other sources of error

the relative errors decrease and, although the relative error may change from one sample to another, we can give a value, the *maximum relative error*, that it will generally not exceed.

Table 2 shows the maximum relative error for estimating PCB levels with sample sizes of 1 to 25. Unfortunately, even to get 50% maximum relative error may require a large number of samples. For example, if 10% white goods are processed (with 90% automobiles or other materials), approximately 25 samples are required to obtain 50% maximum relative error when no compositing is used. Notice that when compositing is used, the number of samples that must be analyzed to achieve a desired maximum relative error is reduced. For example, 64% maximum relative error can be expected when 16 samples are analyzed without compositing. If 18 samples are composited into 9 groups of 2 samples each, however, then 68% maximum relative error can be obtained by analyzing the 9 composited samples. There is a slight increase in maximum relative error (since 68% is greater than 64%), but the laboratory costs are reduced almost by half (i.e., 9 samples analyzed instead of 16). Finally, notice that to obtain maximum relative error of less than 25% requires very large sample sizes, even when compositing is used.

In discussing sampling over time in Section 2, we recommended taking samples every half-hour for at least 8 hours, which would result in 16 samples. From Table 2, we see that the resulting maximum relative error would be about 64%, if no compositing is used. This will be adequate when the level of PCBs found is low (e.g., 10 to 20 ppm), but may be unacceptable if a high level of PCBs is found. If the 16 samples are composited into 8 composite samples of 2 subsamples each, the maximum relative error would be about 70% (i.e., slightly higher than that shown for 9 composites of 2 subsamples each). If the 16 samples are composited into 4 composites of 4 subsamples each, the maximum relative error increases to 106%. Again, this is probably acceptable when the level of PCBs is low, but will not be acceptable when the PCB level is, say, 20 or 30 ppm. The sampling procedures described in Section 2 for stored fluff will produce 20 samples; the maximum relative error for 20 samples would be similar to those for 16 samples, although slightly lower.

The key factor in deciding how many samples to take is the maximum relative error desired. In deciding the maximum relative error, the concentration of PCBs must also be taken into account. Suppose, for example, that the actual PCB concentration is 10 ppm and that we estimate the level of PCBs as being between 0 and 20 ppm. Then the maximum relative error is 100%, but since the estimated PCB concentration is well below the 50 ppm standard, this level of error is acceptable. However, if the actual PCB concentration is 50 ppm and we estimate that the level of PCBs is between 0 and 100 ppm, the maximum relative error is again 100%, but it is

Table 2: Relative error for estimating PCB levels with sample sizes of 2 to 25

Total samples collected	Number of composites analyzed	Subsamples in each composite	Maximum relative error*
2	2	1	1084%
4	4		192%
9	9		93%
16	16		64%
25	25		50%
4	2	2	793%
8	4		140%
18	9		68%
32	16		47%
50	25		36%
8	2	4	597%
16	4		106%
36	9		51%
64	16		35%
100	25		27%
16	2	8	468%
32	4		83%
72	9		40%
128	16		28%
200	25		21%

*A relative error of 50% means that with 95% certainty, the estimated average concentration will be within 50% of the actual average concentration. A relative concentration of more than 100% (e.g., 150%) has the same interpretation (e.g., the estimated concentration will be between 0% and 1.5 times the actual concentration).

clearly not acceptable. In exploratory studies, high relative errors can generally be tolerated, since more data can be collected to investigate the situation more closely if high levels of PCBs are suspected.

Sample Sizes and Relative Error for Lead and Cadmium. In general, the samples sizes required for estimating PCB levels should be more than adequate for estimating levels of lead and cadmium. Analysis of preliminary data indicates that both sampling and measurement errors are smaller for these substances than for PCBs. Comparable data for other toxic substances is not available.

4.4 Analytical Methods for Other Objectives

Exploratory studies are only one possible objective of sampling for PCBs at shredder sites. Another objective would be monitoring shredder output to make sure that PCB levels do not exceed a given level. In practice, monitoring programs are often put in place by shredder operators to verify to landfill operators that fluff from the site meets TSCA landfill regulations. A third objective would be “clean-up” verification, which might be required if a site – or the fluff produced at a site – were found to be extensively contaminated with PCBs. In both cases, the statistical method of *hypothesis testing* would be used in place of confidence intervals. These topics are discussed in an appendix.

4.5 Additional Reading

For more details on statistical procedures for use in environmental sciences, see

Statistical Methods for Environmental Pollution Monitoring, Richard O. Gilbert.
Van Nostrand Reinhold Company Inc. 1987.

APPENDIX

ANALYTICAL METHODS FOR REGULATORY PROCEDURES

A.1. Introduction

A.1.1 Objectives of Regulatory Procedures

As discussed in the Section 1, there are several possible objectives in sampling for PCB's. Analytical methods for exploratory studies were discussed in Section 4 of the *Sampling Guidance*. The two objectives of regulatory functions are monitoring and clean-up verification. This appendix discusses statistical methods for these applications.

When monitoring the output of a shredder site, the monitoring agency – which may be the shredder operator or an outside agency – develops a program of regular sampling and analysis of materials to assure that shredder output meets specified standards. In this situation, the output is *assumed not to be contaminated* until the samples collected for the monitoring program demonstrate otherwise.

In the event that a shredder site or output from a site is established as being contaminated with PCB's – if large piles of stored fluff or the soil around the site are known to contain high concentrations of PCB's, for example – then it may become necessary for the site to undergo some form of clean-up or change in operating procedures. In this case, the site (or output from it) is *assumed to be contaminated* until the samples collected during the clean-up verification demonstrate otherwise.

The statistical methods for these two applications appear to be very similar. In each case, the average PCB concentration is found and compared with a known value to make conclusions about the PCB level. Although the procedures differ slightly in the methods of calculation, the important difference is in the decision-making process indicated by the italics shown above. While the procedures discussed in Sections A.2 and A.3 may appear redundant, purpose of the analysis and the conclusions that would be reached are different.

A.1.2 Sampling Issues

A number of sampling issues arise in planning monitoring and clean-up verification programs. These issues are mainly related to the frequency and duration of visits to the shredder site to collect samples. This is more of an issue for monitoring programs, where regular visits are more likely to be required.

Should samples be collected once a week? Once a month? Four times a year? In deciding how often to collect samples, it must be remembered that the material output from a shredder is the direct product of the input to the shredder. The primary objective in sampling is to obtain a representative sample of the material that is output during the normal operation of the shredder. It is possible for the shredder operator to run only "clean" materials – for example, materials that have had all electric motors, air conditioning units, etc., removed – while the samples are being collected. If this is done, the samples may not reflect the materials that are normally output at the shredder.

Ultimately, the question of "how often" is really less important than whether the samples collected are representative of the normal output of the shredder. Obviously, samples taken four times a year may not be representative of the output being produced during the rest of the year. However, sampling even once a week may not be sufficient if the samples selected are not representative.

When monitoring programs are in place, sampling usually takes place at regular intervals, ranging anywhere from four times a year to once a week. Within this context, samples may be collected once a visit, once each half-hour for several hours, or once each half-hour for an entire day. As part of either a monitoring or a clean-up program, we suggest collecting samples of fresh shredder output each half-hour for a period of 8 hours, or one work day. As noted in the *Sampling Guidance*, the longer the duration of the sampling period, the greater the likelihood of obtaining a representative sample of shredder output. Sampling for an entire working day is likely to provide good representation of the shredder's normal operations, at least for that day, and also will provide a minimum number of samples for statistical analysis.

A.1.3 Hypothesis Testing

As we have noted, there are several possible sources of error in assessing contamination by PCB's or other toxic substances. For exploratory studies, we used confidence intervals as a statistical procedure for analyzing data in the presence of error. For monitoring and clean-up programs, hypothesis tests are the primary analytical tool.

In hypothesis testing, an assumption is made – for example, that the normal fluff output of a given shredder site has a PCB concentration that is 50 ppm or less – and then evaluated in relation to the results of a laboratory test. For example, suppose that laboratory tests indicate that the average concentration in samples collected is 60 ppm. We know that because of sampling and measurement errors, the *actual* concentration is not *exactly* 60 ppm. In an hypothesis test, we do a set of calculations which provide a numerical cut-off against which our sample value is compared. This cut-off depends on the number of samples analyzed and some other considerations. For example, suppose that the cut-off is 75 ppm. Comparing the sample estimate of 60 to the cut-off value of 75, we would conclude that the laboratory results are within the range of sampling and laboratory error and that we do not have sufficient evidence to conclude that the output of the shredder is more than 50.

A.2. Monitoring

A.2.1 Considerations in Monitoring Programs

As we discussed earlier, the objective of a monitoring program is to make sure that the output of a shredding operation meets some specified standard. Frequently this standard is taken to be 50 ppm, since this is the requirement for TSCA landfills, but other standards might be considered as well. In this manual, we will use three possible standards – 25, 50 and 100 ppm – as illustrations. Monitoring programs may also vary with respect to the frequency and duration of sampling. Samples of output materials may be taken weekly, monthly, or quarterly, with samples collecting over several hours or an entire day. In most cases, the sample sizes discussed for monitoring are intended for a single visit.

There are two major difficulties in monitoring shredder sites. First, because of the time delay in having samples analyzed, the actual shredder output that is sampled will probably be in a landfill by the time the analysis is done to determine whether it is contaminated or not. Second, the amount of PCB's can be loosely controlled by processing different materials, since, for example, automobiles appear to be less likely to produce PCB contaminated output than white goods. Thus, shredder operators being monitored by outside agencies could deliberately process materials with low PCB levels during the monitoring period. If the materials processed during the monitoring period are not representative of the normal output of the shredder, then the results of the monitoring program will not be valid.

Clearly, monitoring programs, which depend on statistical principles and random inspections, cannot detect all violations. The best strategy for keeping contaminated output out of landfills is to develop monitoring programs that are *likely* to detect *most* violations, so that appropriate enforcement actions can be taken. One of the key steps in developing an effective monitoring program is to collect representative samples. We suggest three steps. First, regulatory agencies can make unannounced visits to the shredder site at randomly chosen times to help assure obtaining representative samples. Similarly, shredder operators can collect samples at irregular intervals to help assure representative sampling. Second, the longer the duration of the data collection period, the more likely that shredder input will be representative; we recommend that the monitoring period last 8 hours or for the normal duration of operating hours. Finally, samples of stored fluff and spillover should be collected, in addition to fresh fluff, since these materials are likely to reflect the output during normal operation even when fresh fluff may not.

A.2.2 Hypothesis Testing for Monitoring Programs

When monitoring the output of a shredder site, it is first assumed that the output streams are *not* contaminated. Samples are collected and chemically analyzed at intervals to monitor the shredder output, and, based on a statistical analysis of these samples, the monitoring agency determines whether this assumption – i.e., that the shredder output is in compliance with safety standards – is reasonable. The process used to make this determination is called a *hypothesis test*. The basic steps are simple: the average and standard deviation are calculated, a cut-off value is determined and the average

is compared to the cut-off value. If the average is larger than the cut-off value, then the output is declared in violation, otherwise it is assumed to be in compliance. In the following sections we will discuss how to determine the cut-off value and the sample sizes necessary for making hypothesis tests.

As we discussed earlier, the presence of sampling error and analytical error make it difficult to determine whether shredder output is in compliance with regulations. The fact that chemically analyzed samples are above the safety standard is not sufficient evidence that the entire output from which the samples were taken is in violation. A more careful evaluation must be done to account for sampling and analytical error. The procedure that must be followed is illustrated in an example in Worksheet A-1.

The first step is to find the average and standard deviation using the procedures given in Worksheet 1 in Section 4. Next, the cut-off value must be determined. This value can be found by following the calculations in Worksheet A-1. Finally, to evaluate whether or not shredder output violates the relevant standard, simply compare the average of the analyzed samples to the cut-off value and follow these rules:

- If the average is *larger* than the cut-off, conclude that the output violates the standard
- If the average is *smaller* than the cut-off, assume that the output is in compliance with the standard.

A.2.3 Effects of Sampling and Analytical Error

Like all decisions that are based on statistical methods, hypothesis testing procedures are subject to error. For example, in a pile of fluff that is relatively free of PCB's, we may pick a sample simply by chance that has an unusually dense concentration of PCB's, leading us to conclude that the entire pile of fluff is contaminated. In this case we would *incorrectly conclude that the output was in violation*. On the other hand, in a pile of fluff that is heavily contaminated, we might happen to pick a sample that has a relatively low level of PCB's, leading us to *incorrectly conclude that the output is in compliance*. These two errors have many names in the statistical literature, but they are most commonly called "Type 1" and "Type 2" errors, respectively.

Worksheet A-1: Hypothesis Testing for Monitoring PCB Levels

Example Data. Assume that 4 composite samples are analyzed and have these PCB levels:

PCB's (ppm)	Squared PCB's
70.0	4,900.0
121.0	14,641.0
48.0	2,304.0
51.0	2,601.0

Step 1: Find the average and standard deviation. Use the directions in Worksheet 1. For the example data given above:

$$\text{Average of Samples} = 72.50$$

$$\text{Standard Deviation} = 33.77$$

Step 2: Determine the Cut-Off Value. Make the following calculations:

- **Short-Cut Method.** In Table A-1, select the appropriate safety standard and then find the cut-off which corresponds to the standard deviation and sample size that are closest to the yours. For the example data, the standard deviation and sample size are 33.77 (which is close to 35) and 4. Assuming the safety standard is 50, the cut-off is 91.1.
- **Exact Method.** This method is slightly more complicated. First, in Table A-2, find the *t*-value for a sample size of 4, which is 2.35. Now make the following calculation:

$$\text{Cut-Off Value} = \text{Standard} + t\text{-value} \frac{\text{Standard Deviation}}{\sqrt{\text{Sample Size}}}$$

If the standard is 50 ppm, then

$$\text{Cut-Off Value} = 50 + 2.35 \frac{33.77}{\sqrt{4}} = 89.7.$$

Step 3: Interpretation. Since the average, 72.5, is smaller than the cut-off, 91.1 (using Method 1, or 89.7, using Method 2) we do not have sufficient evidence to conclude that the output exceeds the 50 ppm safety standard.

Table A-1: Cut-off values for monitoring*

Safety Standard	Standard Deviation	Number of Composite Samples Analyzed				
		2	4	9	16	25
25	20	114.2	48.5	37.4	33.8	31.8
	35	181.2	66.1	46.7	40.3	37.0
	50	248.1	83.8	56.0	46.9	42.1
	75	359.6	113.1	71.5	57.8	50.7
	100	471.2	142.5	87.0	68.8	59.2
	150	694.3	201.3	118.0	90.6	76.3
	250	1,140.5	318.8	180.0	134.4	110.5
50	20	139.2	73.5	62.4	58.8	56.8
	35	206.2	91.1	71.7	65.3	62.0
	50	273.1	108.8	81.0	71.9	67.1
	75	384.6	138.1	96.5	82.8	75.7
	100	496.2	167.5	112.0	93.8	84.2
	150	719.3	226.3	143.0	115.6	101.3
	250	1,165.5	343.8	205.0	159.4	135.5
100	20	189.2	123.5	112.4	108.8	106.8
	35	256.2	141.1	121.7	115.3	112.0
	50	323.1	158.8	131.0	121.9	117.1
	75	434.6	188.1	146.5	132.8	125.7
	100	546.2	217.5	162.0	143.8	134.2
	150	769.3	276.3	193.0	165.6	151.3
	250	1,215.5	393.8	255.0	209.4	185.5

*If the average of the analyzed samples is larger than the cut-off value in the table, then conclude that the shredder output violates the given standard. Otherwise, assume that the output meets the standard. The chance of incorrectly finding a violation is 5%.

Table A-2: t-values for hypothesis tests*

Number of composite samples	t-values
2	6.31
3	2.90
4	2.35
5	2.13
6	2.02
7	1.94
8	1.89
9	1.86
10	1.83
11	1.81
12	1.80
13	1.78
14	1.77
15	1.76
16	1.75
17	1.75
18	1.74
19	1.73
20	1.73
21	1.73
22	1.72
23	1.72
24	1.71
25	1.71
30	1.70
50	1.68
75	1.67
100	1.66
>100	1.65

*The values shown in the table are taken from Student's t distribution. This distribution is often used as a measure of uncertainty due to sampling and other sources of error.

Using the procedure described in Worksheet A-1, you will have a 5% chance of making a Type 1 error – that is, of concluding that output is in violation when in fact it is not. The chance of this type of error is 5% regardless of the sample size. The chance of a Type 2 error – the chance of missing violations when they actually exist – does depend on the sample size. Because characteristics of fluff vary from place to place, it is difficult to determine the exact probability of making a Type 2 error, but based on preliminary studies we have made some approximate calculations that are shown in Tables A-3 through A-5. These tables give the chance of correctly identifying violations (i.e., *not* making a Type 2 error) for a range of sample sizes and hypothetical PCB levels for safety standards of 25, 50, and 100 ppm.

For example, in Worksheet A-1, the hypothesis test based on four samples concluded that the output met the 50 ppm safety standard. In Table A-4 (which covers the 50 ppm standard) we see that with 4 composite samples, assuming each consists of 1 subsample, the chance of detecting a violation of even 125 ppm is only 11%. Thus, we should not feel too confident that the material is actually in compliance with the standard. As might be expected, the larger the sample size the greater the chance of detecting violations. This is true if the sample size is increased by analyzing more composite samples or by compositing more subsamples together. Thus, when 9 composites of one subsample each are analyzed, the chance of detecting a violation of 125 ppm is 44%, meaning that 44% of the time a violation of 125 would be detected using procedures like this, while 56% of the time a PCB level of 125 would remain undetected. Notice that the situation improves substantially if 9 composites are used with 4 subsamples each, in which case the chance of detecting a violation of 125 ppm increases to 88%.

A.3. Clean-up Verification

A.3.1 Considerations in Clean-up Verification

In exploratory studies, there is little if any prior knowledge about contamination by PCB's or other substances at a site. In monitoring programs, it is assumed that shredder output streams are in compliance with PCB standards unless the data indicate otherwise. However, when a statistical evaluation is undertaken to verify a site

Table A-3: Chance of finding violations in monitoring with a 25 ppm standard

Total samples collected	Number of composites analyzed	Subsamples in each composite	Chance of detecting violation*				
			Actual PCB concentration				
			30	35	40	50	60
2	2	1	0.00	0.00	0.00	0.00	0.00
4	4		0.02	0.04	0.05	0.08	0.11
9	9		0.08	0.15	0.22	0.33	0.42
16	16		0.13	0.25	0.37	0.56	0.68
25	25		0.18	0.36	0.53	0.75	0.86
4	2	2	0.00	0.00	0.00	0.00	0.00
8	4		0.03	0.05	0.08	0.14	0.20
18	9		0.11	0.22	0.34	0.53	0.65
32	16		0.19	0.39	0.57	0.79	0.89
50	25		0.26	0.55	0.76	0.93	0.98
8	2	4	0.00	0.00	0.00	0.00	0.00
16	4		0.04	0.08	0.14	0.25	0.35
36	9		0.15	0.34	0.51	0.75	0.86
64	16		0.26	0.57	0.78	0.95	0.99
100	25		0.38	0.76	0.93	0.99	1.00
16	2	8	0.00	0.00	0.00	0.00	0.00
32	4		0.05	0.12	0.22	0.40	0.54
72	9		0.21	0.48	0.69	0.90	0.96
128	16		0.36	0.74	0.92	0.99	1.00
200	25		0.51	0.90	0.99	1.00	1.00

*Power calculations assume a 5% chance of incorrectly finding a violation.

Table A-4: Chance of finding violations in monitoring with a 50 ppm standard

Total samples collected	Number of composites analyzed	Subsamples in each composite	Chance of detecting violation*				
			Actual PCB concentration				
			60	70	85	100	125
2	2	1	0.00	0.00	0.00	0.00	0.00
4	4		0.02	0.04	0.06	0.08	0.11
9	9		0.08	0.15	0.25	0.33	0.44
16	16		0.13	0.25	0.43	0.56	0.70
25	25		0.18	0.36	0.60	0.75	0.87
4	2	2	0.00	0.00	0.00	0.00	0.00
8	4		0.03	0.05	0.10	0.14	0.21
18	9		0.11	0.22	0.39	0.53	0.68
32	16		0.19	0.39	0.64	0.79	0.91
50	25		0.26	0.55	0.83	0.93	0.98
8	2	4	0.00	0.00	0.00	0.00	0.00
16	4		0.04	0.08	0.17	0.25	0.37
36	9		0.15	0.34	0.59	0.75	0.88
64	16		0.26	0.57	0.85	0.95	0.99
100	25		0.38	0.76	0.96	0.99	1.00
16	2	8	0.00	0.00	0.00	0.00	0.00
32	4		0.05	0.12	0.27	0.40	0.56
72	9		0.21	0.48	0.77	0.90	0.97
128	16		0.36	0.74	0.96	0.99	1.00
200	25		0.51	0.90	1.00	1.00	1.00

*Power calculations assume a 5% chance of incorrectly finding a violation.

Table A-5: Chance of finding violations in monitoring with a 100 ppm standard

Total samples collected	Number of composites analyzed	Subsamples in each composite	Chance of detecting violation*				
			Actual PCB concentration				
			125	150	175	200	250
2	2	1	0.00	0.00	0.00	0.00	0.00
4	4		0.02	0.04	0.06	0.08	0.11
9	9		0.10	0.18	0.26	0.33	0.44
16	16		0.16	0.31	0.45	0.56	0.70
25	25		0.22	0.45	0.63	0.75	0.87
4	2	2	0.00	0.00	0.00	0.00	0.00
8	4		0.03	0.07	0.11	0.14	0.21
18	9		0.14	0.28	0.42	0.53	0.68
32	16		0.24	0.49	0.68	0.79	0.91
50	25		0.34	0.67	0.85	0.93	0.98
8	2	4	0.00	0.00	0.00	0.00	0.00
16	4		0.05	0.11	0.18	0.25	0.37
36	9		0.20	0.43	0.62	0.75	0.88
64	16		0.34	0.69	0.87	0.95	0.99
100	25		0.49	0.86	0.97	0.99	1.00
16	2	8	0.00	0.00	0.00	0.00	0.00
32	4		0.06	0.17	0.29	0.40	0.56
72	9		0.27	0.59	0.80	0.90	0.97
128	16		0.47	0.85	0.97	0.99	1.00
200	25		0.65	0.96	1.00	1.00	1.00

*Power calculations assume a 5% chance of incorrectly finding a violation.

clean-up, it must be assumed that the site (or the output stored on a site) is contaminated until the data demonstrate that an effective clean-up has been carried out. Except for this important distinction, the procedures for clean-up verification are nearly identical to those described in Section A.2.

A.3.2 Hypothesis Testing for Clean-up Verification

The procedure for determining cut-off values in clean-up evaluation is illustrated in an example in Worksheet A-2. As before, the first step is to find the average and standard deviation using the procedures given in Worksheet 1. Next, the cut-off value is determined, either by following the calculations in Worksheet A-2 or from Table A-6. Finally, to evaluate whether or not the output attains the safety standard, simply compare the average of the analyzed samples to the cut-off value as follows:

- If the average is *smaller* than the cut-off, conclude that the site has attained the safety standard; and
- If the average is *larger* than the cut-off, assume that the site is still in violation and requires further clean-up.

A.3.3 Effects of Sampling and Analytical Error

Because of sampling and analytical error, these procedures are subject to Type 1 and Type 2 errors, just like the methods described in Section 2. Here the possible errors are (1) concluding that the site has attained the safety standard when the actual concentration of PCB's still exceeds it, and (2) concluding that additional clean-up is required when in fact the site has attained the safety standard.

For the methods described above, the chance of incorrectly concluding that the site has attained the safety standard is at most 5%. (It is exactly 5% when the actual level of PCB's meets the standard and it decreases sharply as the level of PCB's increases.) Tables A-7 through A-9 show the chance of requiring additional clean-up for standards of 25, 50, and 100 ppm when the concentration of PCB's at the site actually meet the standard. This probability becomes larger when either the level of PCB's approaches the standard, or when the sample size is small. It should be noted that because clean-up will

Worksheet A-2: Hypothesis Testing for Verifying Clean-Up of PCB's

Example Data. Assume that 4 composite soil samples from the cleaned site are analyzed and have the following PCB levels:

PCB's (ppm)	Squared PCB's
11.0	121.0
5.0	25.0
52.0	2,704.0
10.0	100.0

Step 1: Find the average and standard deviation. Use the directions in Worksheet 1. For the example data given above:

Average of Samples = 19.50

Standard Deviation = 21.83

Step 2: Determine the Cut-Off Value. Make the following calculations:

- **Short-Cut Method.** In Table A-6, select the appropriate standard and find the cut-off which corresponds to the standard deviation and sample size which are closest to yours. Assume the standard is 50 ppm. For the example data, the standard deviation and sample size are 21.83 (which is close to 20) and 4, indicating a cut-off of 26.5.
- **Exact Method.** This method is slightly more complicated. First, in Table A-2, find the *t*-value for a sample size of 4, which is 2.35. Now make the following calculation:

$$\text{Cut-Off Value} = \text{Standard} - t\text{-value} \frac{\text{Standard Deviation}}{\sqrt{\text{Sample Size}}}$$

For the example data,

$$\text{Cut-Off Value} = 50 - 2.35 \frac{21.83}{\sqrt{4}} = 24.3.$$

Step 3: Interpretation. Since the average, 19.5, is smaller than the cut-off, 26.5 (using Method 1, or 24.3, using Method 2), we can conclude that the site meets the 50 ppm standard.

Table A-6: Cut-off values for clean-up verification

Standard	Standard deviation	Number of composite samples analyzed					
		2	4	9	16	25	
25	10	—	13.3	18.8	20.6	21.6	
	15	—	7.4	15.7	18.4	19.9	
	20	—	1.5	12.6	16.3	18.2	
	25	—	—	9.5	14.1	16.5	
	35	—	—	3.3	9.7	13.0	
	50	—	—	—	3.1	7.9	
50	65	—	—	—	—	2.8	
	10	5.4	38.3	43.8	45.6	46.6	
	20	—	26.5	37.6	41.3	43.2	
	30	—	14.8	31.4	36.9	39.7	
	50	—	—	19.0	28.1	32.9	
	60	—	—	12.8	23.8	29.5	
100	75	—	—	3.5	17.2	24.4	
	125	—	—	—	—	7.3	
	15	33.1	82.4	90.7	93.4	94.9	
	25	—	70.6	84.5	89.1	91.5	
	50	—	41.3	69.0	78.1	82.9	
	75	—	11.9	53.5	67.2	74.4	
	100	—	—	38.0	56.3	65.8	
	150	—	—	7.0	34.4	48.7	
	250	—	—	—	—	14.5	

*A dash (—) indicates that the standard deviation is too large to establish that the site is clean.

Table A-7: Chance of requiring additional clean-up with a 25 ppm standard

Total samples collected	Number of composites analyzed	Subsamples in each composite	Chance of requiring more clean-up*				
			Actual PCB concentration				
			1	5	10	15	20
2	2	1	-	0.82	1.00	1.00	1.00
4	4		-	-	0.31	0.86	0.97
9	9		-	-	0.01	0.48	0.87
16	16		-	-	-	0.22	0.79
25	25		-	-	-	0.07	0.70
4	2	2	-	0.16	1.00	1.00	1.00
8	4		-	-	0.07	0.74	0.96
18	9		-	-	-	0.24	0.81
32	16		-	-	-	0.05	0.68
50	25		-	-	-	-	0.54
8	2	4	-	-	1.00	1.00	1.00
16	4		-	-	-	0.54	0.93
36	9		-	-	-	0.07	0.72
64	16		-	-	-	-	0.53
100	25		-	-	-	-	0.35
16	2	8	-	-	0.97	1.00	1.00
32	4		-	-	-	0.33	0.90
72	9		-	-	-	0.01	0.61
128	16		-	-	-	-	0.37
200	25		-	-	-	-	0.18

*These calculations assume a 95% (or greater) chance of requiring additional clean-up when the concentration of PCB's is 25 ppm or greater. A dash (-) indicates that the chance is less than .005.

Table A-8: Chance of requiring additional clean-up with a 50 ppm standard

Total samples collected	Number of composites analyzed	Subsamples in each composite	Chance of requiring more clean-up*					
			Actual PCB concentration					
			10	15	20	30	40	
2	2	1	0.82	1.00	1.00	1.00	1.00	1.00
4	4		-	0.02	0.31	0.86	0.97	
9	9		-	-	0.01	0.48	0.87	
16	16		-	-	-	0.22	0.79	
25	25		-	-	-	0.07	0.70	
4	2	2	-	0.16	1.00	1.00	1.00	1.00
8	4		-	-	0.07	0.74	0.96	
18	9		-	-	-	0.24	0.81	
32	16		-	-	-	0.05	0.68	
50	25		-	-	-	-	0.54	
8	2	4	-	0.77	1.00	1.00	1.00	1.00
16	4		-	-	-	0.54	0.93	
36	9		-	-	-	0.07	0.72	
64	16		-	-	-	-	0.53	
100	25		-	-	-	-	0.35	
16	2	8	-	0.27	0.97	1.00	1.00	1.00
32	4		-	-	-	0.33	0.90	
72	9		-	-	-	0.01	0.61	
128	16		-	-	-	-	0.37	
200	25		-	-	-	-	0.18	

*These calculations assume a 95% (or greater) chance of requiring additional clean-up when the concentration of PCB's is 50 ppm or greater. A dash (-) indicates that the chance is less than .005.

Table A-9: Chance of requiring additional clean-up with a 100 ppm standard

Total samples collected <i>m</i>	Number of composites analyzed <i>m/c</i>	Subsamples in each composite <i>c</i>	Chance of requiring more clean-up*				
			Actual PCB concentration				
			20	30	40	60	80
2	2	1	0.82	1.00	1.00	1.00	1.00
4	4		—	0.02	0.31	0.86	0.97
9	9		—	—	0.01	0.48	0.87
16	16		—	—	—	0.22	0.79
25	25		—	—	—	0.07	0.70
4	2	2	0.16	0.98	1.00	1.00	1.00
8	4		—	—	0.07	0.74	0.96
18	9		—	—	—	0.24	0.81
32	16		—	—	—	0.05	0.68
50	25		—	—	—	—	0.54
8	2	4	—	0.77	1.00	1.00	1.00
16	4		—	—	—	0.54	0.93
36	9		—	—	—	0.07	0.72
64	16		—	—	—	—	0.53
100	25		—	—	—	—	0.35
16	2	8	—	0.27	0.97	1.00	1.00
32	4		—	—	—	0.33	0.90
72	9		—	—	—	0.01	0.61
128	16		—	—	—	—	0.37
200	25		—	—	—	—	0.18

*These calculations assume a 95% (or greater) chance of requiring additional clean-up when the concentration of PCB's is 100 ppm or greater. A dash (—) indicates that the chance is less than .005.

remove PCB's from the contaminated area, the homogeneity of samples taken after clean-up may be greater; that is, the standard deviations *after* clean-up may be smaller than the standard deviations before clean-up. In this case, the chance of requiring additional clean-up would be decreased from the values shown in Tables A-7 through A-9.

Notice that the probability of being required to do additional clean-up is related to both the PCB level remaining after clean-up – and thus to the intensity of the clean-up effort – and to the amount of data collected for verification. For example, suppose that the standard is 50 ppm. If the clean-up effort is less rigorous, resulting in residual PCB levels of about 30 ppm, say, then it will require more data to verify the clean-up than if the clean-up had been more intensive and the residual PCB level were only 20 ppm. This point has implications for allocating funds between the clean-up and verification efforts.

Clean-Up Verification for Lead and Cadmium. Because of smaller sampling and measurement errors, it is easier to detect whether lead and/or cadmium have been cleaned up with the amount of data required for detecting clean-up of PCB's.

A.3.4 What to Do When Clean-Up Is *Not* Verified

When the sample results indicate that the site has not been cleaned up thoroughly, it is very important to realize that it is *not sufficient* to simply clean and re-inspect the parts of the site that are in the sample. The reason for this is that the samples collected are representative of the entire site; if the collected samples have not been thoroughly cleaned up, then it must be assumed that the rest of the site has not been satisfactorily cleaned up, either.

Therefore, where clean-up does not pass verification, the *entire site* must be cleaned again! Then, after the site has been cleaned, all the verification steps must be repeated using a second, *independent* collection of samples.

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16. Abstract (Limit: 200 words) The purpose of this document is to provide basic instructions for collecting and statistically analyzing samples of materials that are produced as a result of shredding automobiles and other metal objects, since the by-products of these recycling operations may contain concentrations of polychlorinated biphenyl's (PCBs). Shredders are large machines that convert light metal objects into fist size or smaller pieces of scrap metal. PCBs enter the shredder output when materials containing PCB-bearing fluids are shredded. Large concentrations of PCBs have been identified in some samples that have been collected at some recycling sites. Thus agencies may wish to collect data at shredder sites in order to study the situation in their locality. The sampling procedures described in this document are intended to produce representative samples of fluff that will give reasonably accurate estimates of the overall concentration of PCBs in the material being sampled. The document discusses sample selection, laboratory testing, and statistical procedures for analyzing the data.			
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