

## **CHAPTER 6 - ISSUES RELATED TO INCINERATOR ASH SAMPLING**

### **6.0 INTRODUCTION**

Assessing the appropriate management options for MSW incinerator residues requires a knowledge of their physical and chemical characteristics and an understanding of how these characteristics contribute to their behaviour in the field. This type of information is generally derived from laboratory analyses of samples which are extremely small in size compared to the volume of material actually generated.

“The initial ... and perhaps most critical ... element in a program designed to evaluate the physical and chemical properties of a solid waste is the plan for sampling.” (U.S. EPA SW-846)

Thus sampling can be an error generating process, (Gy, 1982; Pitard, 1986). Furthermore, the laboratory procedures have the potential to generate highly variable data if the inherent heterogeneity of the residues is not taken into account during the sample preparation stages. Consequently, the selection of an appropriate strategy for sample collection and preparation should be based on techniques which ensure an adequate level of representativeness is achieved.

The information presented in this chapter places these issues into context with the overall objectives of a sampling program, the factors which can influence sample variability, and recommended approach for sample collection of the different ash streams. These discussions will draw on the information provided in Chapters 2, 3 and 4.

### **6.1 THE CONCEPT OF THE REPRESENTATIVE SAMPLE**

A continuous theme throughout the U.S. legislation (US EPA SW 846 - 40 CFR, Part 261, Appendix II) is the concept of representativeness. The legislation also defines variability as the change of properties with time, whereas the variance is defined as the variability between samples which is attributable to laboratory procedures. Therefore, the overall error of any characterisation process is the sum of the sampling error and the analytical error. If results are to be considered representative, not only must the laboratory accuracy be high (the closeness of an analytical result to its true value), but also the sampling precision must be within reasonable limits (the closeness of repeated sample values). Irrespective of the accuracy of the laboratory analyses, data will still be of limited usefulness if the sample collection procedure is inadequate or inappropriate. Since repeating the analytical portion of a study is much easier than re-conducting the sampling program, the importance of a sound sampling program cannot be overemphasised.

Although it would be ideal to collect and analyse the total of anyone residue stream from an incinerator, this is generally highly impractical, if not impossible. Moreover, the ash streams tend to exhibit a relatively high degree of heterogeneity. In view of these limitations, it is unlikely a truly representative sample can be taken, and therefore the definition of a “representative” ash sample becomes a sample collected based on an approach which minimises sample variability.

Many factors will influence the “representative” nature of a sample of ash from a specific facility including:

- the origin/type of waste
- the type of incinerator/APC system and the operating conditions
- residue type and the means by which the residue stream is transported and stored

### 6.1.1 Waste Type

One of the first considerations regarding ash sampling is to identify some general characteristics of the waste being fed to the incinerator. MSW characteristics will vary on a load-to-load, day-to-day and season-to-season basis. Unless the incinerator burns RDF, it is unlikely the waste will be adequately mixed in the storage pit to maintain uniform feed quality, which invariably means the chemical and physical properties of the residues generated from burning the waste will also vary. Since it is difficult to identify the origin of a load of waste fed to an incinerator and attribute its characteristics to that of a corresponding ash sample, it will suffice to note the general characteristics of the waste. For example:

- is the waste pre-processed into RDF or burned “as-received”?
- is the waste largely commercial or residential waste?
- is there a source separation or curbside recycling scheme in place?
- what day of the week and what time of year is the sampling being conducted?

This type of information will not only assist with development of a sampling strategy, but also help with data interpretation and allow other researchers to make better comparisons with data from other facilities. For example, since the processing of RDF acts to “homogenise” the waste to a much greater extent than mixing “as-received” waste in a storage pit, the sampling frequency may not need to be as great as when sampling a mass-burn design incinerator. However, the carry-over of particles in the flue gas stream of the RDF system will increase the volumes of fly ash and APC residue generated and thereby may increase the number of grab samples required to generate a composite sample. Furthermore, commercial waste may be more homogenous than residential waste, but contain a greater proportion of larger items which will influence the size of the sample which needs to be collected. In addition, if

a source separation scheme is in place to remove recyclable materials, a much lower frequency of ferrous and glass in the ash may result in fewer preparation steps to generate samples for analysis. Overall, this type of assessment will assist with setting some of the initial parameters for sampling ash.

### **6.1.2 Type of Incinerator/APC System**

In addition to waste feed variability, a sampling program developed for one type of incinerator system may not be suitable for another system because of differences in the operating regime of the two facilities. All of the different processes or systems should be factored into the sampling plan and each facility should be considered as a unique set of circumstances. Issues such as the ash discharge cycle must be taken into consideration. For instance, conventional large mass-burn units with moving grates discharge ash on a relatively continuous basis, but smaller controlled air systems, such as the Consumat technology, discharge residue to the quench tank on a periodic basis. This means the frequency of grab samples taken from a Consumat system should be geared toward collecting grab samples at intervals covering the period that ash is being removed from the quench tank.

Another example relates to the manner in which samples can be taken from the different incinerator systems. Extracting a sample of dry grate ash prior to quenching from a mass-burn design can be accomplished by accessing the chute between the discharge end of the grate and the quench tank. However, obtaining dry ash directly from the discharge end of a two-stage system prior to quenching is complicated. The "starved-air" conditions in the primary chamber means the system is under pressure and backdrafts can occur when accessing the last discharge ram, thereby not only causing upsets in the operating conditions, but making this type of sample collection a dangerous task as well.

The heat recovery and APC systems typically generate residues on a cyclical basis. Boiler soot blowers may operate on a timed cycle, i.e., only once every 6 to 8 hours, whereas the cleaning cycles for the fabric filter systems are generally initiated when the pressure differential across the bags is sufficiently high. While the differences in times are not a problem when sampling these streams in isolation of each other, the actual cleaning cycle's events must be considered when sampling combined ash streams, especially if they are entering the quench tank. Since these residues constitute a portion of the total ash being generated, the sampling events must be timed to ensure the appropriate proportions of these residues are included in the combined sample. Without this type of consideration, any wide variations in ash properties may be unaccountable.

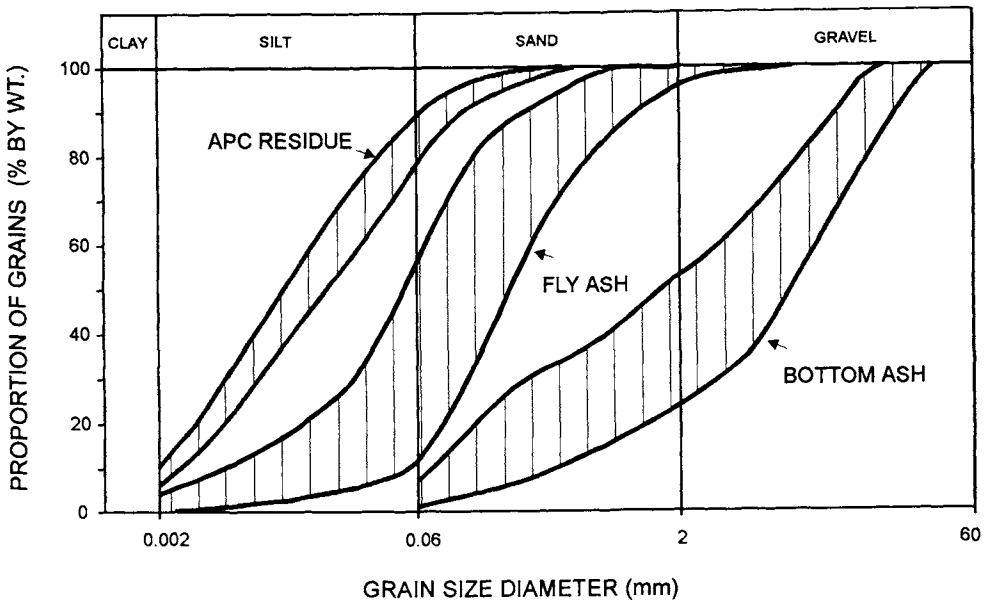
Finally, the operation conditions during the sampling must be recorded so any deviations from "typical" operations which might affect the ash characteristics, can be identified. For example, residues created during transition periods such as start-up and

shutdown will not be representative of normal operation. Similarly, excursions in furnace temperatures below or well above the "normal" operating temperatures (due to either wet waste or an unusually high proportion of highly flammable materials) should be noted to assist with data interpretation.

### 6.1.3 Residue Streams

MSW incinerator residue streams consist of particles of varied sizes and shapes, each of which can have different physical and chemical properties. Typical particle size distribution data for various residue streams are shown in Figure 6.1, although it should be noted that bottom ash data is based on samples screened to remove <50 mm material (e.g., metal cans, small appliances, large auto-parts, and large clinkers). Normally, metal cans and other materials larger than 150 mm would be anticipated to represent between 5 and 20% of the total bottom ash stream. The sampling of large clinkers requires special consideration particularly considering the weight, size and temperature of this material as it exits the system. Such materials should be sampled and analysed separately from the bulk of the bottom ash.

Figure 6.1 Particle Size Distribution of Various Residues



Hartlén and Elander, 1986

Sampling waste material with wide particle size distributions requires recognition of the physical processes that can affect the distribution of materials in a storage pile. If both fine and coarse particles are discharged to a storage pile, the coarse material tends to remain on the outside of the pile whereas the fines sift to the interior (Boomer, et al, 1988), see Figure 6.2. Furthermore, materials with different angles of repose will not intermix in a pile and surface friction effects can cause a separation of different materials as they are discharged from a chute (e.g. the high friction angle material falls nearer the chute). These problems can generally be addressed by employing pile sampling strategies which account for the differences in particle size, shape and density.

## 6.2 OBJECTIVES OF MATERIAL SAMPLING PROGRAMS

Generally there are two principal objectives for sampling the MSW incinerator residues:

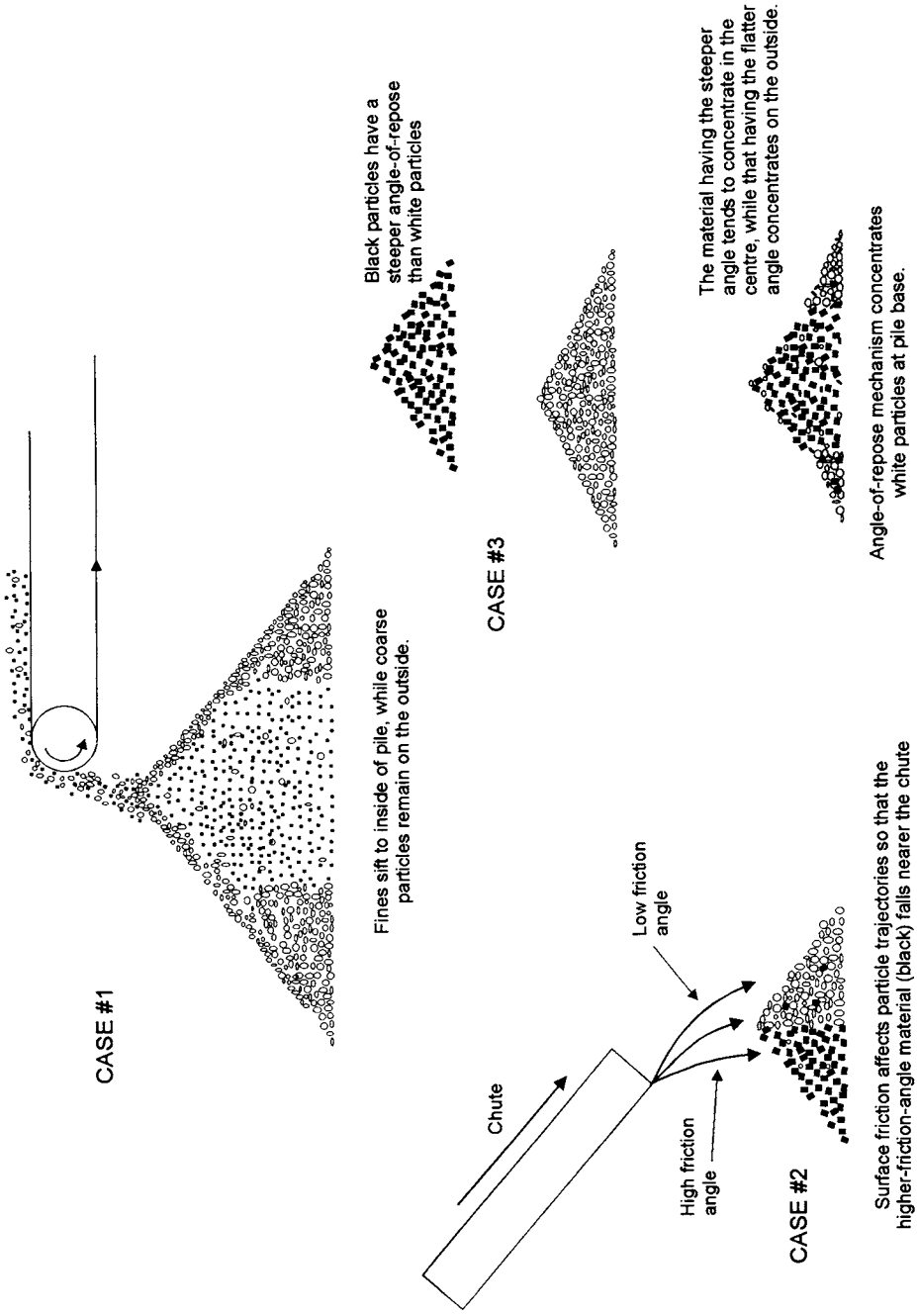
- quality control/regulatory monitoring - to confirm that the variability of the ash stream characteristics is within an acceptable range for a number of parameters
- research - to develop a better understanding of the mechanisms responsible for the physical and chemical properties of the streams, and to determine which treatment procedure can be used to render the streams acceptable for the chosen management method

For each objective there is likely an ideal strategy that involves both the quantity of material collected and the frequency of the collection, but tradeoffs are likely to be made between the ideal sampling strategy and the practical or optimal strategy for a given case. If, for instance, the purpose of the sampling is quality control related to utilisation of the material, it is likely the optimal sampling strategy would involve rather large increments taken on a frequent basis from a pile, whereas regulatory sampling may only require small volume samples taken on a low frequency. Regardless of the strategy, if true statistics are to be developed, the frequency of sampling should always be greater than the rate of change of the characteristics.

Although a sampling approach should be conducted within well defined limits, a certain degree of flexibility in strategy is required. For example, if significant fluctuations in the material quality are found during low frequency sampling, the sampling frequency could be increased to achieve a better understanding of the average nature of the material. A statistically valid sampling plan attempts to sort out the inherent noise and overlapping cycles so a true estimate or value of a time-dependent characteristic can be obtained.

Equations 6.1 - 6.6 provide a means by which the representative nature of any sample can be evaluated (SW-846). These equations define the mean,  $\bar{X}$ ; standard deviation,

Figure 6.2 Examples of Material Heterogeneity



S; standard error,  $S_{\bar{x}}$ ; a confidence interval based upon the standard error, (CI), and the appropriate number of samples,  $n$ , that need to be collected. The sampling protocol defines that a minimum of 4 samples must be collected regardless of the results of calculations based upon the equations:

$$\text{Mean} = \bar{X} = \frac{\sum_{i=1}^n x_i}{n} \quad (6.1)$$

where  $n$  = the number of sample points

$$\text{Variance} = S^2 = \frac{\sum_{i=1}^n x_i^2 - \frac{(\sum_{i=1}^n x_i)^2}{n}}{n-1} \quad (6.2)$$

$$\text{Standard Deviation} = S = \sqrt{S^2} \quad (6.3)$$

$$\text{Standard Deviation of Mean} = S_{\bar{X}} = \frac{S}{\sqrt{n}} \quad (6.4)$$

$$\text{Confidence Interval} = CI = \bar{X} \pm t_{.20} S_{\bar{X}} \quad (6.5)$$

$$n_R = \frac{t_{.20}^2 S^2}{(RT - \bar{X})^2} \quad (6.6)$$

where:

RT = the selected acceptance level or Regulatory Threshold

$n_R$  = the number of samples required

$t_{\alpha}$  = student's  $t$  for a suitable confidence limit (suggest 80% for residue sampling)

Since these equations are based upon the assumption that the parameters are normally distributed and caution should be used when applying these statistics. Most residue samples generate data which are log normally distributed rather than normally distributed. Therefore, it is necessary to assess the distribution of any data set. A simple test of comparison between the variance and the mean of the data should indicate a normal distribution. If the variance is larger than the mean, the data is

unlikely to be normally distributed and must be transformed before these equations can be used. The most common transformations are:

**Square Root Transformation -**

the transformed value is the square root of the original value with or without an offset parameter;

**Arcsine Transformation -**

the transformed value is the arc sine of the square root of the original value divided by an offset parameter and expressed as a percentage.

All statistical calculations and tests of acceptability should be carried out on a transformed data set.

### 6.3 AVAILABLE SAMPLING PROTOCOLS

Although few full sampling protocols for incinerator residues have been published, the Swedish Geotechnical Institute has compiled a comprehensive discussion of the issue (SGI, 1993) and an earlier protocol was proposed by Fiesinger (1988). Moreover, many reports on ash testing generally contain a basic description of the sampling approach, however, these descriptions usually do not contain the rationale for the selection of a particular method. Consequently, the framework for developing an experimental design for residue sampling is generally adapted from published standards for sampling of other materials.

Besides the SGI report, there are several other documents which can provide relevant guidance with respect to ash sampling and statistical interpretation:

- Test Methods for Evaluating Solid Waste (EPA SW-846) from the U.S. EPA which covers sampling and analytical methods for a wide range of solid waste materials
- ASTM D346-78: The Collection and Preparation of Coke Samples for Laboratory Analysis. Documents sampling for Crushed or Powdered Materials.
- ASTM D1452-80: Soil Investigation and Sampling by Auger Borings. Documents sampling for Soil-like Materials.
- ASTM D2234-86: Collection of a Gross Sample of Coal. Documents sampling of materials that are similar to bottom ash materials.
- ASTM E 122 - Choice of Sample Size to Estimate the Average Quality of a Lot or Process; and,

- ASTM E 177 - Practice for Use of the Term Precision and Bias in ASTM Test Methods

Another important issue is handling the sample to obtain the necessary testing samples. This issue is covered in detail in:

- ISO 1988-1975 (E) Standard for Sampling Hard Coal.
- ASTM C702-87, Standard Practice for Reducing field Samples of Aggregate to Testing Size
- CEN/TC 154/TG 5 Aggregates: Sampling and Precision

Consideration of various aspects of sampling aggregates is also addressed in the ZWL (1992) document that supports the CEN Aggregate Standard.

Many of these sampling protocols utilise similar approaches and terminology when referring to collecting portions of the stream. Although the following general outline of the issues provides a common framework for discussing examples of sampling programs suitable for various residue streams and processes, it is recommended that further details be obtained from a direct review of the above-mentioned documents.

## 6.4 SAMPLING CONSIDERATIONS

To obtain a representative sample, it is normal practice to collect a definite number of portions, known as increments, from the whole mass of the material being sampled. Depending upon the purpose of the sampling, the increments can be stored for either individual analyses or they can be combined in the field to produce a gross (or composite) sample over a certain time period, e.g. day, week, month, or year. The method of collecting the increment usually influences the representative nature of the sample.

### 6.4.1 Increment Collection Classification

The conditions under which individual increments are collected, and the methods of spacing the increments (both spatially and temporally) from the whole stream are classified according to the descriptions given below. These designations can be used for sampling specifications and for descriptions of sampling programs and sampling equipment.

**Conditions of Increment Collection** - These conditions relate the main body of the residue relative to the portion withdrawn. Five conditions are recognised:

- Condition A (Stopped-Belt Cut) - A loaded conveyor belt is stopped and a full cross-section cut with parallel sides is removed from the residue stream.
- Condition B (Full-Stream Cut) - A full cross-section cut is removed from a moving stream of residue.
- Condition C (Part-Stream Cut) - A portion, not a full cross-section, is removed from a moving stream of residue.
- Condition D (Falling Stream) - Material is removed by inserting the sampler into a falling stream of the residue being sampled.
- Condition E (Stationary Sampling) - A portion of residue is collected from a pile, container, truck trailer, rail car, barge, or ship's hold.

Condition A is the one that is most likely to produce a representative sample. However, owing to the disruptions that can be caused by stopping a conveyor belt, Condition A is mainly used as a reference method to calibrate other methods. Theoretically, Conditions B and C should give samples with the same representative nature as Condition A, however, in practice it is difficult to remove material from a moving stream without introducing some disruptions that might bias results. Condition D is often referred to as the second best method, however its use requires care since the material being sampled must be relatively uniform and equipment should be robust enough to withstand the forces involved. Achieving a representative sample with a stationary sampling program (Condition E) is energy intensive and usually requires heavy equipment.

**Spacing of Increments** - The spacing of increments pertains to the distances between sample points, and the time interval between increments. Three methods of spacing the increments have been proposed (ISO 1988-1975(E)):

- systematic sampling - the increments are spaced evenly in time or position over the unit
- random sampling - the increments are spaced at random in time or in position over the unit
- stratified random sampling - the unit is divided by time or quantity into a number of equal strata and one or more increments are taken at random from each stratum.

From a statistical point of view (to ensure the highest precision and thus the most representative samples), random sampling or stratified random sampling should be used. The position of sampling points or the intervals used for sampling are

determined by using random numbers. Stratified random sampling should be used only if the stratification can be determined accurately enough, i.e. if the distribution of chemical contaminants in a waste is sufficiently known to allow an intelligent identification of strata and enough samples can be taken from each stratum. If this condition is not fulfilled, stratified random sampling is likely to give lower precision than simple random sampling. Many times, however, a proper random sampling can be difficult to carry out because of difficulties such as accessing the individual stream or too short a time interval within the process. Therefore, many random sampling plans turn out to be systematic random sampling in which the position and/or the point of time for the first sample is selected randomly and all subsequent samples are taken at a fixed space or time interval.

Systematic sampling is usually chosen as it is easier to operate on a routine manner than random sampling. Systematic sampling can give samples of less precision, especially if the samples are collected in such a manner that cyclic variations have a great influence on the quality of the residue. Thus, it is essential that the influence of cyclical operations on the proportions of different residues be considered when sampling combined ash streams.

**Types of increments** - The types of increments are based on whether or not there is human discretion in the selection of the residue portions sampled.

- Type I - Specific pieces or portions are not subject to selection on a discretionary basis. This includes increments collected in precise accord with previously assigned rules on timing or location that are free of any bias. Type I selection increments generally yield more accurate results.
- Type II - Some measure of human discretion is exercised in the selection of specific pieces of residue of specific portions of the stream, pile, or shipment.

#### 6.4.2 Bias

Bias is defined as a tendency to obtain results which are consistently too high or too low. This may happen very easily during sampling. These conditions are often difficult to detect and care should be taken to prevent this occurrence during sampling. The two major causes of such bias are:

- selecting an unrepresentative part of the stream for sampling; or
- removing an unrepresentative portion of the stream when sampling.

As noted earlier, sampling a pile consisting of particles with a wide size distribution is difficult because the material tends to segregate as a function of size and angle of repose. Therefore, it is necessary to collect increments from both the outside of the pile and the internal part of the pile.

The size of the device being used to collect the sample can influence the nature of the sample collected as well. For instance, using too small a sampler will automatically reject particles larger than the diameter of the sampler from the collected mass. Typically, bottom ash contains some ferrous material that is larger than 150 mm in size, and by using a 150 mm wide sample thief to collect bottom ash samples will result in selectively rejecting all the larger materials discharged from the hearth. In most cases, this larger material is composed of ferrous metals and relatively inert materials which are typically recorded and discarded from further analysis anyway. Consequently, this limitation would not be considered a severe detriment to the accuracy of the data.

To eliminate the potential for bias, both the size of the sampling equipment and the mass of the increment should be in accordance with the maximum dimensions of the particles in the stream being tested. This means different criteria can be applied to bottom ash, heat recovery ash and APC residue streams. Bias can be improved by changing the sampling equipment or moving the sampling location, however, precision and accuracy of the sampling cannot be improved by increasing the size of the increment (ISO 1988-1975(E)). Similarly, while precision may be altered by adjusting the number of increments, this will have little, if any, effect on bias. The level of bias should be checked regularly, or at least when a new sampling program or new equipment is employed. A check procedure is provided in ISO 1088-1975(E).

### **6.4.3 Precision**

Equation 6.6 illustrates a relationship between the variability of sampling results and the number of samples required to ensure satisfactory results. Although a standard for sampling aggregates and coals (ISO 1988-1975(E)) suggests the precision should be within 10% for low values and 2% for higher values of ash or moisture, these values are derived by having a large base of data upon which to develop the requirement. Since only limited data are available for a wide variety of tests, it is not possible to develop precision guidelines for each residue type or each test. Rather, it is necessary to rely upon the guidance provided by various standards and an intuitive assessment of what is required to determine the number of increments to collect.

#### **Number of Increments in Composite Sample**

ASTM D 2234 defines the objective of incremental sampling as the formation of a representative sample from the lot for analyses. This representative sample would have been within 1/10 of the average value of all the determinations for the parameter of interest for 95 out of 100 such samples. The standard also notes the number of composite samples that are collected defines the potential accuracy of the results. To reduce the error by a factor of two requires squaring the number of composite samples. Similarly to triple the accuracy takes 9 times as many composite samples. The ultimate is to analyse each increment, a situation that should provide one fifth of the error that would arise from analysing 1 sample composed from 24 increments.

ISO 1988-1975 (E) suggests the number of increments for sampling hard coal can range from 16 to 64 depending upon where the sample is taken and the type of coal. Conveyors provide a greater chance of sampling the full cross-section of the material and require fewer increments than stockpiles. Similarly, the variability in the material, i.e. washed versus unwashed coal, is reflected by the need to double the number of increments for the unwashed material.

With bottom ash from MSW incinerators, experience suggests it is satisfactory to collect 24 increments although a minimum of 10 samples has been deemed to be more practical. Statistical relations should be used in all cases to verify that the number of increments taken is appropriate. If there is prior knowledge of the characteristics of interest in the residue, statistical relationships can be used to estimate the number of samples required. These estimates should then be confirmed using the results of the testing.

#### 6.4.4 Size of Increments

The number and weight of the increments required for a given degree of precision depends upon the variability of the residue, and the parameters being tested. While the number of increments is a function of the precision required, the size of the increment is more a function of the size of the particles being collected. Several standards suggest some form of Equation 6.7 be used to determine the appropriate increment size for a given size of materials. Regardless of the result of applying the equation, most standards also specify minimum increment size requirements. Typically 0.5 kg is used as a minimum criterion. ISO 1988-1975(E) recommends a minimum increment size of 10 kg for materials >150 mm in size. This would be considered appropriate for an increment of wet bottom ash, which in practice may be 5 to 7 litres in size.

$$P \text{ (kg)} = 0.06 D \text{ (mm)} \quad (6.7)$$

where      P = the mass of the increment  
               D = the nominal upper size of the material, i.e. the square mesh screen size such that no more than 5% of the material in oversize.

The ISO standard offers further guidance on sampling large size material. It suggests that the proportion of >150 mm material be determined for several increments and then the number of increments be adjusted to ensure that the >150 mm fraction is sampled in proportion to its mass in the overall stream. These large sized increments, which will weigh well in excess of the nominal increment weight, should be size reduced to <80 mm and then mixed and subdivided to reduce the mass to the required increment size before the increment is added to the composite sample. The standard also cautions that collecting larger than necessary increments can lead to problems with sample handling and reduction to the appropriate analytical sample size. Conversely, it notes

that under no circumstances should the number of increments be decreased if the mass is too large.

#### **6.4.5 Collection Procedures**

In order to obtain complete representation of all size fractions within an ash stream, it is preferable to have the sample increments withdrawn from the full cross section of the stream. This is accomplished using Condition A and D outlined earlier. The increment must be distributed throughout the lot to be sampled. This distribution is related to the entire volume of the lot, not merely its surface or any linear direction through it or over it.

The spacing of the increments should be varied to ensure the sampling does not correspond to one single part of any cyclical operation which occurs in the facility. For instance, sampling should not occur every time fines are discharged to the bottom ash system from the boiler soot-blows or vice versa. This would bias not only the chemical composition of the samples, but also the particle size range for these samples. Similarly, if the boiler and bottom ash from the facility are mixed before the sampling point, ensure that within the range of samples collected, the number of increments includes an appropriate amount of the fine material that comes from the soot blow cycle.

When sampling from moving streams, the sampling device should be designed to minimise disturbance of the residue, thereby avoiding separation of various residue densities and sizes, or both. Again the ISO 1988-1975(E) standard helps to define some important factors. When sampling from any stream, it recommends the minimum opening of the sampling instrument should be 2.5 times the upper size of the materials. This implies the minimum width of a stopped belt cut, moving stream cutter, and sampling scoop for trucks or stockpiles should be 2.5 times the material's upper dimension. As a note of caution, it does not recommend sampling material larger than 80 mm from a moving stream, therefore the largest opening size would be 200 mm.

Several cautions have been noted previously on the need to ensure the size distribution of the samples is not biased. This is important when sampling the bottom ash from a vibratory conveyor. Vansant (1991) notes there is an increasing preference in North America for these devices on the bottom ash discharge since they are rugged and easy to service. The vibratory nature of the conveyor tends to cause both the segregation of certain size materials and the agglomeration of fines across the conveyor. This segregation continues into the ash bunker. Sampling the material off the conveyor is tedious and interrupts the plant operating cycle since the whole width of the conveyor would need to be carefully cleaned. Sampling from the bunker requires careful consideration of the potential for biased distributions.

All sampling equipment should be designed and used in a manner to prevent contamination of the samples so no biases are introduced into the results. Every sample must be collected carefully following the established procedure. As noted earlier, the errors introduced by poor sampling cannot always be corrected by repeating the work.

In selecting the appropriate sampling procedure, local knowledge and input are also imperative. These provide an understanding of the operational variability of the furnace so compensating steps can be taken in the design of the program.

#### **6.4.6 Sampling Streams Other Than Bottom Ash**

The nature of residues from other parts of the system (ie. heat recovery and air pollution control residues) are sufficiently different that many of the concerns outlined above are not as relevant. The materials are more homogeneous and consist of a much narrower size distribution based mainly in the sand or clay range (i.e. <2 mm). This reduces the variability and allows reduction of the size and number of both increments and composite samples required to obtain representative samples.

##### **Grate Siftings and Heat Recovery Ash**

It should be recognised there is a potential for boiler ash to significantly change the characteristics of relatively large amounts of bottom ash. Clearly, the proportion of boiler ash in the total quantity of residue generated from a facility is <5% for mass burn systems, and much less for two-stage systems. For example, a recent study determined a 250 tpd mass burn incinerator generated approximately 150 kg of boiler ash per hour (WASTE Program, 1993), whereas only about 1 kg of boiler ash was generated over 12 hours in a two-stage combustion unit (Environment Canada, 1985).

An issue that must be considered is the potential for the residue from these sources to accumulate in various sections of the incinerator/boiler/economiser during the operating cycle. The staff at the Burnaby EFW facility reported that during shutdown it is necessary to clean the grate siftings chutes to remove solidified materials from the inside surfaces of the hoppers. Similar experiences were observed during testing of the Connecticut Resource Recovery Facility where aluminum solidified in the screws used to remove siftings from under the grate. Boilers and economisers need to be cleaned on a periodic basis to remove solidified material from tubes and surfaces inside the units when heat transfer capabilities drop. In essence, the quantity of residues which are retained within these separate systems is highly dependent on the type of incinerator and the type of waste being burned. A thorough review of the maintenance problems and operating experiences will assist with making decisions regarding how to sample these residue streams. Therefore, boiler ash streams should be segregated from the bottom ash streams to ease the sampling requirements. In many cases, the addition of boiler ash to the bottom ash is not a continuous operation and the cyclical nature is difficult to compensate for in the sampling strategy.

### **APC Residues**

The quantity of APC residues from most mass burn systems represents about 10% of the total residue stream. Generally, given the fine and more homogeneous nature of this material, fewer samples collected by systematic sampling on a fixed time basis are adequate. However, modern plants tend to use enclosed transport systems that operate on a timed cycle and thus selecting the location and timing of sampling requires some preliminary investigation. For example, some dry lime injection systems recycle a portion of the spent residue to save on lime consumption. During operation the recycled lime is mixed with fresh reagent in the reactor and as it proceeds through the system, is collected in the fabric filter area. The material collected from the fabric filter is returned to the recycled lime storage bin where it is stored for up to 24 hours before it is re-injected into the reactor. Lime particles can go through the circuit up to 3 times before they are removed from the system and sent to the APC residue storage silo. The material that is bled from the circuit is generally removed from the base of the reactor. A possibility exists under this operating scenario for samples to be tainted by operational upsets that occur up to 24 hours prior to the sampling. In considering a sampling program, it may be appropriate to collect samples from this stream over longer periods of time to ensure all possible factors are assessed.

### **Storage Piles**

In some instances, residue piles may need to be sampled and these piles present a unique problem for sampling. In forming the pile, segregation of the different size fractions can occur either due to density, size, friction angle or the angle of repose of the material. In considering conical piles, it is important to recognise that 70% of the mass is in the lower third of the pile, 26% in the middle third and only 4% in the top third. For tent-shaped piles, these proportions become 56%, 33% and 11%. When sampling such piles the samples should be taken in a proportional manner. If, on the other hand, the material in the pile has segregated, care should be taken to sample from each of the segregated areas in proportion to its quantity. Practically however, representative samples will be difficult to obtain.

As noted elsewhere, sampling of piles should extend through the whole mass. During sampling, care should be exercised to avoid potential segregation of the material. The draft CEN/TC 154/TG 5 guidelines suggest driving a plank of wood or a steel plate into the pile above where the sampling is to be carried out and taking the sample immediately below the plate. The guideline also suggests the sample should be taken from a depth of at least twice the upper material size to ensure a representative sample, whereas the Swedish recommendation is at least 30 cm depth (SGI, 1993). Consideration of these issues relies heavily upon experience, however, scientific evaluation of the issues before initial sampling, and modification of the process after analysing the initial samples will allow collection of relatively representative samples from storage piles.

### **Sampling from Trucks or Containers**

In the case where an ash conveyor discharges directly to containers or trucks, it may be necessary to establish a systematic pattern for sampling the container. ASTM D346 provides guidance for sampling coke from rail cars. It suggests that, because there is a potential to segregate material by size in this situation, the surface is not the appropriate location to sample. A sample should be taken from 300 mm below the surface, and the hole to reach this location should be at least 3 times the diameter of the largest material in the shipment. In Sweden, it is generally accepted that the hole should be 5 times the diameter of the largest material. Given the practicality of these issues, it is recommended that a nominal diameter of 300 mm should be suitable for most bottom ash streams.

Sample locations are not selected at random but rather at fixed locations based upon given measurements of the container. Divide the container length into 10 equal segments and divide the width into 6 equal segments. The intersection of the lines denoting  $1/6$ th,  $1/2$  and  $5/6$ ths of the width and each length segment designate a potential sampling point. In a systematic fashion select sufficient sampling points to obtain the required number of increments.

### **6.4.7 Sample Preparation Concerns**

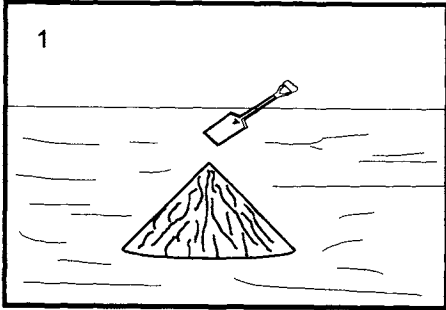
After samples are collected, it will be necessary to package them and ship them to the laboratory for processing. Care in sample handling is just as important as care in sample collection.

#### **Sample Size Reduction**

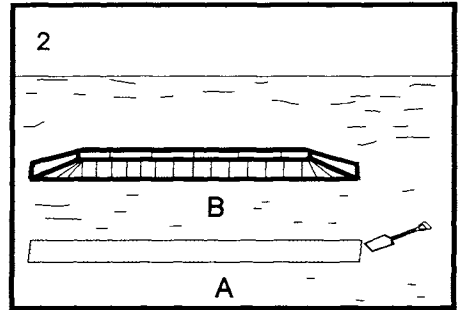
In most cases, the volume of material collected is far too large to send to laboratories for analysis. Field methods need to be used to reduce the volume of the sample to a workable size. This is the first step in sample preparation. The amount of sample required for laboratory analyses depends upon the tests that will be run and the quantity of material required for each test. In most cases, no more than 20 L of bottom ash is required to undertake the testing. Correspondingly smaller quantities of the finer residue streams are required. The exceptions to this rule of thumb are special tests that require large quantities of materials such as column or lysimeter leaching tests. In this case, the total composite sample volume might be required. Suitable adjustments can be made in the procedures to allow for larger sample requirements.

The samples collected in the field can be composites of the various increments or the increments themselves. Assuming the bottom ash sample is a composite formed of a number of increments, it will be necessary to reduce it in size to obtain the laboratory sample. It is recommended that the sample be divided by hand in the field. The procedure to be used follows that outlined in ASTM D346. (Figure 6.3)

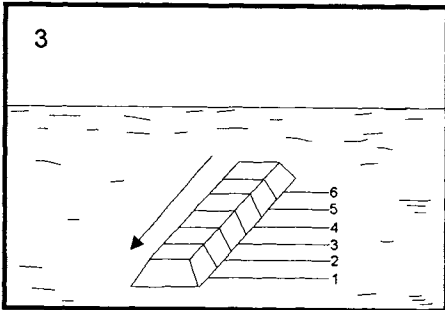
Figure 6.3 Example of a Manual Sample Dividing Technique



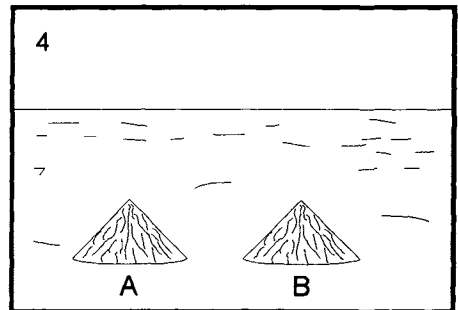
250 lbs. (113 kg)  
crushed to 3/4" (19 mm)  
and coned



Mix by forming long pile  
A- Spreading out first  
shovelful  
B- Long pile Completed



Halving by alternate shovel  
method. Shovelfuls 1,3,5 etc  
reserved as A; 2,4,6 etc  
rejected as B



Long pile divided  
into two parts;  
A- Reserve; B- Reject

Place the increment in a conical pile on a clean surface.  
Utilising a shovel, form a long pile as follows:

Take a shovelful of residue from the conical pile and spread it out in a straight pile having a width equal to the width of the shovel and a length of 1.5 to 3 m. Spread the next shovelful directly over the top of the first shovelful, but in the opposite direction, and so on back and forth, pile, occasionally flattening the pile until all the residue has been formed into one long pile.

This material can then be divided into two piles in the following manner:

Beginning on one side of the pile at one end, and shovelling from the bottom of the pile, take one shovelful and set it aside; advance along the side a distance equal to the width of the shovel and take a second shovelful and discard it. Continue the process, retaining alternate shovelfuls, and reducing the long pile in a uniform manner on one side and then the other. This should produce a sample pile approximately half the size of the original pile.

Continue the long pile and separation procedure until the sample is reduced to the appropriate volume.

### **Preservation of Samples**

The issue of sample preservation is important. All environmental samples require some type of preservation if measured values are to be truly representative of the material. This is most important for wet bottom ash which is known to change its characteristics when it is stored. Indeed, as mentioned in Chapter 5, this is one of the reasons for the bottom ash storage requirements in European protocols directed toward the utilisation of bottom ash. Drying the samples slows these transformation processes, but can only be satisfactorily accomplished in the laboratory. Samples, after they have been reduced in size, must be stored and shipped in appropriate conditions.

### **Sample Containers**

Containers can be either glass or plastic to limit the potential for interaction between the residue and the container, however, for residues that could potentially contain strong bases or HF, glass should not be used. Moreover, plastic containers are more durable. Irrespective of type, all sample containers should have tight fitting lids.

Previously used containers should at least be washed with 10% nitric acid solution and rinsed with distilled water prior to use to avoid contamination from the previous sample. The hygroscopic nature of the heat recovery and air pollution control residue streams require that these samples be placed in dry, air tight containers immediately after

collection. Care should be taken in handling these materials to minimise the sorption of moisture during processing.

### **Sample Storage**

Generally, the ideal storage temperature for ash samples is 4°C, since most reactions that will change residue characteristics are limited at this temperature. Should mercury analyses be required, it may be preferable to oxidise the sample prior to storage to prevent the mercury compounds from reducing and volatilising. One important note however, these samples should be stored in glass containers and the oxidation preservation should be carried out in an ice bath to cool the container since the reaction between the oxidising acid and the residue will be highly exothermic.

### **Laboratory Sample Preparation**

The normal analytical test series conducted on residue samples can be divided into three unique sets of data: physical, chemical and leaching. Preparatory to any of these analyses, the samples need to be processed to obtain a representative sample of the appropriate size for the specific analysis procedure. It is important to remember the sample size criteria related to particle size outlined earlier in this chapter should still be considered when attempting to reduce the volume of material to the laboratory sample. As a general rule, the finer the material, the smaller the sample required from the bulk sample.

### **Laboratory Sample Subdivision**

Since bottom ash is known to change its chemical and leaching properties if it is stored wet, a drying step is introduced early in the protocol to stabilise the material for testing. Assuming a 20 L sample weighing on the order of 25 - 30 kg is sent to the laboratory, a large drying facility would be required to achieve reasonable turnaround for analysis. It is likely preferable to extract a representative sub-sample for drying (@ 105°C) early in the process for chemical analysis (except Hg). Storing the balance of the sample in a refrigerated sealed container for several weeks before drying should not seriously affect the physical properties.

Although conventional sample splitting devices such as whole or divided stream rotary sample dividers or riffle boxes are suitable for dry residue streams, they are unsuitable for dividing the wet bottom ash samples. Conventional splitters should be employed following the provisions of ISO 1988-1975 (E) or other appropriate standards. For the wet samples alternatives such as coring or fractional shovelling following the procedure in CEN/TC 154/TG 5 N 106 E, should be used.

Coring should be accomplished by inserting a trier sampler or tube of at least 125 mm internal diameter at a slight angle to the vertical until it touches the bottom of the pail. The pail should then be tipped at an angle sufficient to allow

the probe to be withdrawn without loss of the sample. The sample extracted in this manner should be dried as described below.

The fractional shovelling method may prove to be simpler to accomplish. Given the maximum particle size of the material a sample of approximately 3 kg is required according to equation 7. Given a mass ( $m$ ) of approximately 30 kg in the laboratory sample, one tenth of the total would be required to provide the analytical sample. Assuming 10 sub-samples ( $n$ ) will be created from the laboratory sample, a scoop capable of holding  $m/(30n)$  of the material should be used. The scoop should thus hold approximately 100 g of sample. The scoop should have a width of 125 mm and could be fashioned from a half section of pipe. The length of the scoop should be at least 100 mm to retain the large sized material that might be present in the laboratory sample.

Take shovelfuls from the laboratory sample and add them to each of the  $n$  sub-samples in turn, until the whole of the laboratory sample has been used. Using random numbers select the sub-sample or sub-samples required for chemical and leaching analyses.

The balance of the sub-samples should be stored separately in sealed containers until such time as the laboratory has time to dry them. These samples may be required for physical testing purposes and storage time and transformations are not as critical for this testing.

### **Drying**

To facilitate easier manipulation of the ash samples for further size reduction and sub-sampling, it is recommended to dry the ash. The laboratory sample should be placed in an open pan and put in a vented drying chamber to allow it to dry at 60°C overnight. Dry the material to a constant weight at 60°C noting the final weight of the sample. Please note the 60°C temperature should not be substituted for determining the moisture content of the sample, or for preparing a dry sample for submission for chemical analysis. For analytical purposes, these samples should be dried at 105°C prior to submission.

### **Size Reduction**

Laboratory analyses are conducted on substantially smaller samples than that contained in the dried sub-sample discussed in the previous paragraph. Before a smaller sample can be created, it is necessary to complete size reduction on the dried sample. It is appropriate to remove ferrous material from the dried sample before size reduction using a magnet since milling this material could damage laboratory equipment. Weigh and note the amount of ferrous material removed.

The remaining ash should then be screened to <9.5 mm. Material trapped on the screen after shaking and stirring should be placed on a hard surface and subjected to three blows from a 3.5 kg. hammer dropped from approximately 0.3 m above the surface or alternatively passed through a standard jaw crusher. Material that will still not pass the 9.5 mm screen after attempting size reduction should be weighed, characterised and discarded. Typical sample preparation procedures are outlined in Figure 6.4.

### **Balance of Materials**

Although the previously mentioned steps have been used to obtain representative sub-samples from the mass of material collected, there may be questions about the results of the analyses. To ensure sufficient material is available to repeat any analyses if required, the bulk samples should be stored for a period of 3 months after the test results are made available. During field sampling procedures, it is worthwhile to consider collecting and storing additional sub-samples of the total increment in the event materials are lost or contaminated in transit to the laboratory.

## **6.5 SAMPLE COLLECTION RECOMMENDATIONS**

Based upon a consideration of the issues in the preceding sections, recommendations for sampling of the various residue streams from MSW incinerators have been developed. These recommendations represent an ideal sampling situation for each of the residue streams, however, it should be recognised that sampling in this manner may not be possible in every case. Limitations in the ability to apply these recommendations can lead to samples that are not truly representative and the user must make a judgement that weighs practicality and necessity to arrive at the best solution for each case. Examples of successful sampling programs are provided later in this chapter.

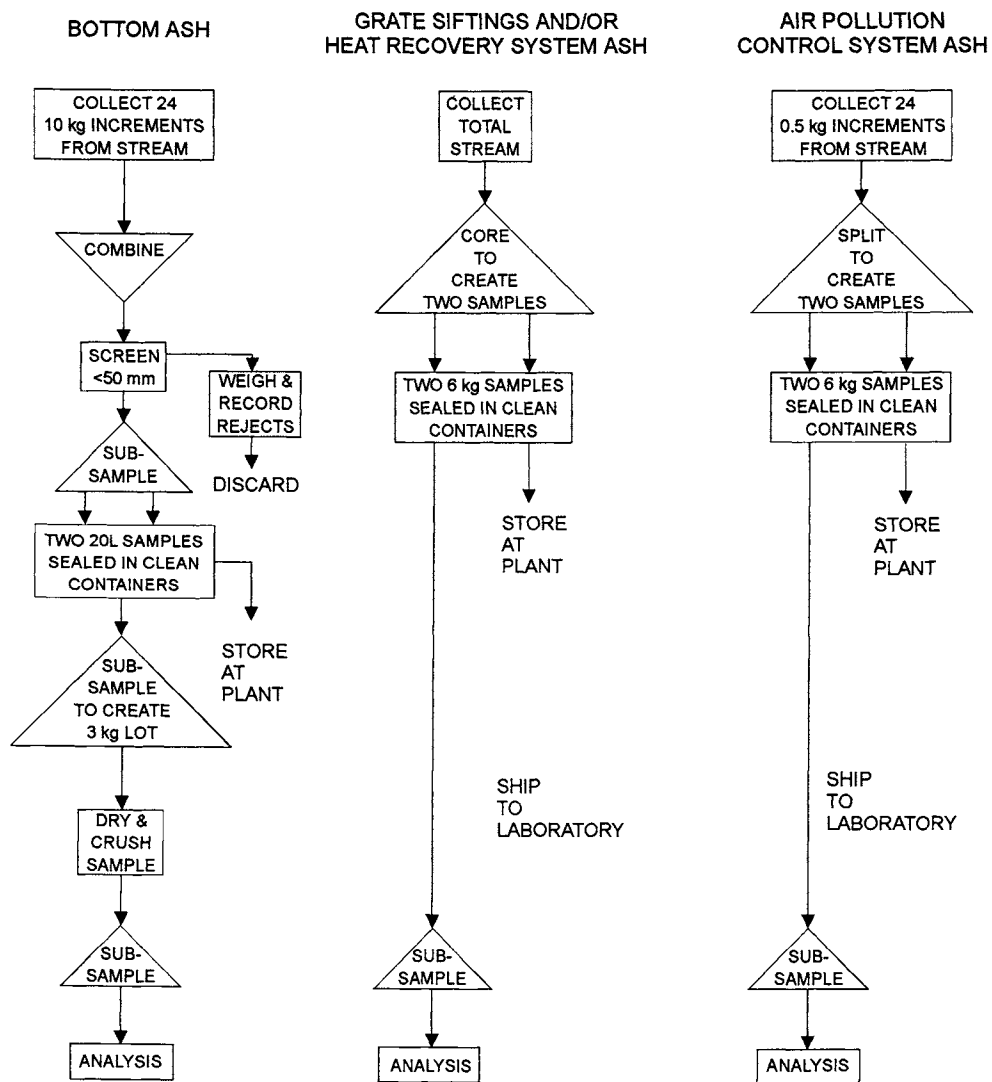
### **6.5.1 Generic Bottom Ash Testing Protocol**

Ideally, each composite sample of bottom ash should consist of 24 increments, however, 10 increments should suffice. ISO 1988 - 1975(E) suggests a minimum of 10 kg for materials whose maximum particle size exceeds 150 mm and this is deemed satisfactory for bottom ash. The procedure that follows will allow at least two samples to be collected in a working shift.

The sampling of bottom ash should follow these steps:

- Identify the safest and most accessible location for obtaining samples of bottom ash

Figure 6.4 Schematic of Residue Collection and Sample Preparation



- Construct a sampling device (trough, bucket, shovel, thief etc.) to be used to gather a grab sample of the entire width of the belt conveyor, drag chain flight, vibrating conveyor or entire depth of the hopper, pile or truck load.
- Gather 10 - 24 incremental samples weighing approximately 10 kg each. The sample should be collected from the entire width of the conveyor, etc., at the beginning of every ten-minute period for four hours. If trucks are to be sampled, then sample every other truck during the four-hour period, taking sufficient increments from each truck to achieve the desired number of increments.
- Screen the sample to minus 50 mm without employing any size reduction processes at this time. Examine the oversize material to characterise the type of components present (large pieces of glass, tin cans, steel etc.), then separate the oversize into two portions: metal and clinker. Weigh all three portions of the sample. This allows metal cans or other large material to be excluded from further examination.
- Mix the undersize fraction of the screened material thoroughly and sub-sample using procedures outlined in ASTM D346 or the previous section to obtain two 20 litre containers of ash. Discard the remaining material.
- Seal and label, both containers with the wet ash samples. Retain one at the facility and ship one to the laboratory.
- Place the oversize clinker material on a large clean hard surface and reduce it by dropping a 3.5 kg hammer on it from approximately 0.3 m height, up to four times. Screen the material through 50 mm mesh and place the minus 50 mm material in a separate labelled and closed container for shipment to the laboratory. Characterise and weigh the balance before storage at the plant.

Note: the intent of the size reduction step is not to reduce all material to the required size regardless of form, rather it is to establish which portions of the sample may be largely composed of metallic materials that cannot be size reduced.

- Store the samples in an appropriate manner until shipped to the laboratory.

This sampling procedure will result in the collection of between 100 and 240 kg of sample which will be reduced to a total mass of approximately 50 - 150 kg for sub-dividing and two sub-samples will be created. The generic procedure indicates that 4 hours are satisfactory for obtaining one composite sample, thus it is possible to collect

a second sample on each day. Not all sampling programs follow the generic plan and the 24 increments may in fact be collected over a different time frame. Where possible, it is recommended that a second four-hour composite be collected during the course of the work day, preferably during a different shift from the first composite.

Weekly samples may be desirable for some purposes. Such weekly samples can be collected by developing a predetermined schedule for randomly selecting a shift and a starting time within that shift so that two samples are collected on each day and a total of fourteen samples are collected during the week. Monthly samples would be collected by randomly selecting one day per week and one shift within that day to collect the appropriate samples.

### **6.5.2 Generic Boiler Ash Sampling Protocol**

As noted earlier, this material tends to be smaller than 2 mm in size, although on occasion large chunks break off the boiler tubes. In most facilities, this material is transferred from the hoppers under the boiler to either the fly ash disposal system or the bottom ash disposal system. In either case, it is likely possible to intercept the flow of material from the boiler and collect all of the ash discharged during the test period. The recommended protocol assumes this is possible, and provides a method for sub-sampling this material. Caution is advised in handling this material as it can be extremely hot as it leaves the boiler and the fine particle size potentially poses a problem with fugitive dust in the sampling area. However, the methods used for generating a suitable laboratory sample should minimise the potential for the finely divided material being released to the air.

The sampling of boiler ash should follow the steps below:

- Identify the most convenient location for obtaining samples of the ash to be evaluated
- Construct an appropriate diversion system to allow collection of all the material generated during the test period in a clean, sealed 210 litre barrel
- At the end of the test period, disconnect the barrel from the system, weigh, and move the barrel to a location suitable for sub-sampling
- Sub-sample the collected material by either the coring or the fractional shovel method discussed above. Each increment should contain approximately 0.5 kg of material. A total of 6 kg per composite sample should be created from 24 cores with alternate cores being used to create the two samples. Seal and label both containers with the boiler ash samples. Send one to the laboratory and retain the other at the facility

Coring should be accomplished by inserting a trier sampler or tube of at least 25 mm internal diameter at a slight angle to the vertical until it touches the bottom of the barrel. The barrel should then be tipped at an angle sufficient to allow the probe to be withdrawn without loss of the sample.

The fractional shovelling method may accomplish the same results in some cases, but can give rise to significant dust levels. A scoop capable of holding approximately 250 g of sample should be used. The scoop should have a width of 50 mm and could be fashioned from a half section of pipe. The length of the scoop should be at least double its diameter. Take shovelfuls from the bulk sample and add them to each of 30 sub-samples in turn, until the whole of the bulk sample has been used. Using random numbers select the two sub-samples required for analyses. The generic procedure indicates that 4 hours are satisfactory for obtaining one composite sample, thus, it is possible to collect a second sample on each day. Timing issues are similar to those considered for bottom ash.

### **6.5.3 Generic APC Residue Sampling Protocol**

APC residue volumes are considerably greater than those of boiler ash and it is not possible to collect the entire stream before sub-sampling. It is therefore necessary to collect increments over a fixed period of time to generate a composite sample for the test period. A minimum increment size of 0.5 kg is likely satisfactory for finely divided materials such as APC residues. It is recommended that 10 - 24 increments be collected. The resulting 6 - 12 kg of material can be divided into two samples, one for the laboratory, the other for storage at the plant. Caution is advised in handling this material as it is hot as it leaves the system and it is fine-grained. The methods used for dividing the sample into two parts should minimise the potential for releasing dust to the air.

The sampling of APC residues should follow the steps below:

- Identify the most convenient location for obtaining samples of APC residue samples. Most systems use some means of conveying these materials from the hoppers under the particulate collection device to storage silos. Tapping into these transport systems is generally the most appropriate location for sampling.
- Construct a sampling device (trough, bucket, shovel, thief etc.) to be used to gather a grab sample of the entire width of the belt conveyor, or conveyor discharge chute. In the case of sampling pneumatic transport lines, the introduction of a manually controlled valve into the transport system piping will allow the discharge of material into a suitable, filter equipped collection barrel.

- Gather a total of between 10 - 24 incremental samples at the start of every ten-minute period for four hours. Alternatively, if a pneumatic conveying system is to be sampled, open the valve for a fixed period of time every 10 minutes, and core the collected mass to obtain the composite sample.
- Divide the composite sample into two equal sized samples using coring, fractional shovelling or mechanical sample dividers. Seal and label both containers, one for the laboratory, the other for retention at the facility.

#### **6.5.4 Documentation of Sampling and Preparation Procedures**

The reporting of test results for residue samples should include:

- a complete description of the methods used to take the samples, including;
  - the type of increments
  - spacing of increments
  - number and weight of increments, and,
  - methods of creating composite samples.
- a description of the sample storage procedures, and,
- a description of laboratory preparation methods including any screening, separation or mixing procedures.

To assist in developing a more useful database on the relationship of physical and chemical properties of residues to the various factors which influence them, it is suggested that the following information should be compiled (where practical) during an ash sampling program:

- Composition of the waste materials being burned, including the types of waste processes at the facility on the dates on which the sample was generated, and any waste composition analyses that may be available.
- Designation and description of the incinerator units and any other specific equipment within the facility which contributed material to the sample, including the heat recovery, pollution control and quenching equipment.
- Description of the operating conditions of the specified equipment prior to and during collection of the sample, including material throughput rates, material discharge frequencies, quench water flow rates, and gas stream temperatures.

## 6.6 EXAMPLES OF SAMPLING STRATEGIES

To illustrate two methods of approaching the experimental design, this section provides examples of several protocols used for sampling residue streams. The examples cover all types of residues, except for wet scrubber systems, and are divided into regulatory testing protocols and research testing protocols to illustrate differences in the approaches. It is important to note that although it is possible to suggest general procedures for sampling, the exact protocol for any given facility and study cannot be outlined until issues of intent and safety are incorporated with the specific facility design and operating characteristics. As a general rule, consideration should be given to the potential influence of the various intermediate residue streams, siftings, boiler ash etc., to determine the best method for sampling. If the facility design permits segregation of the various residue streams, the sampling design will be simpler preventing the contamination of other streams. Moreover, regardless of the precise sampling protocol, good experimental design dictates all aspects of the operation be documented for the test period.

### 6.6.1 Bottom Ash

Bottom ash can be collected in two ways, either quenched or unquenched. The normal process regime is to quench the ash and this is the form the material would take before discharge to the environment. Hence, testing quenched ash is more likely to be specified for regulatory purposes. The unquenched stream may be tested to examine the chemical differences induced by process variations, however, this is generally limited to research. The bottom ash sampling protocols that follow illustrate sampling of both the quenched stream and the unquenched stream. The former was completed for regulatory compliance testing (PRRI, 1992), the latter for the special testing associated with the WASTE Program (WASTE, 1993).

#### Regulatory Testing

This particular sampling program (PRRI, 1992) was conducted for permit compliance purposes on a modular incinerator facility processing both raw MSW and a processed MSW fuel with the fines, glass and metals removed. The 100 tpd Consumat incinerators discharge bottom ash to a quench tank. The ash is removed via a drag chain system which conveys it up an incline allow free water to drain back into the quench tank prior to discharging the ash into a storage bunker. Ash stays in the bunker between 24 and 72 hours (depending upon the time of the week) before being removed with a front end loader and placed in a truck for haulage to a landfill site. The design of the facility precluded sampling the ash off the drag chain conveyor. Therefore, it was decided each bucket load from the front end loader would be sampled before the ash was placed into the truck. The following procedures were employed:

Each sample represented material from one segregated incinerator over the 24 hour operating period immediately preceding the sampling. As the ash was transferred from the storage areas to the haulage vehicle by the front-end loader, sampling personnel took two shovelfuls of ash from the centre of the front-end loader bucket and placed it into a 20 litre bucket. Ash samples were taken from each front-end loader lift. The average number of loader buckets needed to transfer the ash to the haulage vehicles was 12, thus 24 shovelfuls of ash were collected for each composite sample. This resulted in samples weighing between 110 and 200 kg. The use of this procedure excluded sampling large clinker type materials. However, one such mass was collected and several other samples of clinker that were more than 50 mm in diameter were isolated from the bulk samples and analysed. Sample preparation included screening the contents of each pail with a 50 mm screen composed of metal grating, separating the >50 mm material and metals into 2 different portions, and weighing all three portions. The <50 mm was then spread into a long pile and the pile was subdivided to provide two 30 kg samples.

Another example of this type of sampling illustrates an approach to random sampling to assist with the statistical validity of the sampling for regulatory purposes (Rigo & Rigo (1989)). The basic premise of this sampling plan was to collect samples over a year long period. One front-end loader lift of ash contained approximately 1.8 tonnes of ash and the ash was hauled six days a week. The samples from any given day were judged to adequately represent a week's production. Thus, there was a need to select the day of the week for sampling and the individual lifts that would be sampled.

Using a random number table, the day of the week was selected using the following procedure.

- Each shipping day was assigned a range of numbers as follows:

Monday	0-17	Wednesday	34-50	Friday	68-83
Tuesday	18-33	Thursday	51-67	Saturday	84-99

- A 2 digit random number was used to select the day of the week for each week of the sampling period. If the selected day fell on a holiday, the next number was used.

The lifts to be sampled were determined on the sample day by:

- estimating the amount of ash in the bunkers and the total number of bucket loads that would need to be handled that day.
- the buckets which were to be sampled were identified by taking the decimal equivalent of the random number and multiplying it by the total number of loads, then rounding the answer up to a whole number.

- 24 random samples were then taken from the total number of buckets. This resulted in time periods which decreased between samples taken on days when less ash was generated, whereas the duration of an interval increased on the days with more ash produced.
- two shovelfuls of ash were taken from the centre of each of the designated buckets, or as close to the centre of the bucket as possible if too large a piece of material obstructed the penetration of the shovel. It was assumed since the front-end loader was digging through the pile, the sample met the criteria of a grab sample from the pile. Furthermore, the randomness of sample bucket selection satisfied statistical requirements without leading to problems associated with limitations on mixing the sample.

This example could also be adapted to sampling combined bottom ash and APC system residues if necessary, as long as the cyclical operations of the heat recovery and APC system cleaning cycles are taken into consideration.

### **Research Testing**

One example of a research program designed to assess the quality of ash was the WASTE Program (WASTE, 1993), where simultaneous sampling of the waste feed and residue streams from a 250 tpd mass burn incinerator was conducted. Several issues were considered in developing the testing program including the desire to isolate the various streams, i.e., separate grate ash, grate siftings, heat recovery ash, and the residues from the different sections of the APC system. Safety issues and other difficulties made it impractical to sample quenched ash from the vibratory conveyor leading from the quench tank to the ash storage pit. Moreover, the grate ash, grate siftings and heat recovery system ash streams could not be isolated during the entire test period, and since all these streams were mixed in the quench tank, cross contamination of the samples was inevitable. Consequently, it was decided to sample unquenched grate ash as it fell off the end of the grate.

A 1600 cm x 1000 cm chute located at the end of the grate leading to the quench tank was chosen as the area to collect the grate ash. The inspection door on the side of the chute was modified by adding four access ports across the width of the door. These ports allowed a sampling device to be inserted across the width of the grate. Their positions provided access to all parts of the discharged ash stream. The variability in ash quantity and size distribution across the discharge plume was determined prior to start-up of the project tests. Preliminary tests indicated both the port closest to the end of the grate and the one furthest away collected very little material during a fixed sample period, whereas a sample thief inserted into the middle two ports gleaned nearly equal volumes of ash and the size distribution appeared to be similar.

The intent was to gather a sample from the full width of the chute at regular intervals. One grab sample was collected every 30 minutes during each 4 hour test period by inserting the sample thief (a section of 100 mm diameter pipe with the top half removed) across the entire width of the drop-off area and allowing the sample chamber to fill with ash. The thief typically filled within 2 to 3 minutes. The physical size limitations of the sampling ports precluded sampling large materials falling off the end of the grate. Chunks of material caught on the sample thief which were too large to be removed through the sampling port were pushed off prior to removing the sampler. A 25 cm wide by 2 metre long steel trough was constructed to aid sample collection. After the sample thief was extracted from the inspection door, the trough was placed underneath the sample thief to receive the collected ash. The trough was then used to transfer the sample into an appropriately labelled 4.5 L steel container, sealed, weighed and allowed to cool. The individual grab samples were then composited for each of the 4 hour test runs to generate a sample for use in the laboratory.

Another research program was aimed at evaluating the utilisation of ash in which a stratified random sampling approach was used for a continuous flow. The Long Island study (LIRPB, 1991) used the following method of obtaining samples from a continuous stream based upon modified ASTM protocols for sampling aggregate material:

- Conveyor sampling every 15 minutes, during one randomly selected hour in both the morning and afternoon. The samples were removed from the conveyor to form a composite sample. By assigning each 15 minute segment of the period a value, a random number table could be used to select the starting time.

However, various alternatives to these methods can be considered, including:

- random selection of flights from a conveyor based upon the total number of flights that must be sampled to obtain enough material, or,
- randomly pick times during the discharge cycle to sample.

In many cases it is sufficient to systematically sample the streams. For example, a constant discharge cycle can be sampled by collecting an increment every 10 minutes to provide the recommended 10 -24 samples for each composite sample in 4 hours.

### **6.6.2 Grate Siftings**

During the WASTE Program, the grate siftings were segregated to determine potential differences between these and the bulk of the bottom ash, and to determine how much was generated. The entire quantity of grate siftings generated during each test period was collected and sub-sampled by modifying the grate siftings hoppers underneath the

grates with diversion gates which directed the siftings into a duct leading to vented 200 L barrels. Since the grate siftings hoppers at this facility are purged at regular intervals under pressure, the vent on the barrel was fitted with a fabric filter bag to dissipate pressure and minimise fugitive dust.

Prior to hooking up the sample barrels for the morning test runs, the grate sifting hoppers were blown clear. The barrels were then attached to the diversion chutes, the diversion gates were swung into position and the blowers were set back to automatic. Just prior to the end of each test run, the hoppers were blown clear and the barrels removed and weighed. The quantity of grate siftings generated for each test period was monitored by measuring the volume collected and determining the density of the material.

Initial attempts to obtain composite core samples of grate siftings from the barrels proved to be very difficult and time consuming due to the density of the siftings. As a result, a 20 L composite sample for each test period was obtained by tilting the barrels on an angle and removing the contents down the side of the barrels with a shovel. The sub-samples from the two barrels from each test run were composited in a labelled 20 L plastic pail, sealed and weighed.

### **6.6.3 Boiler/Economiser Ash**

Depending on the configuration of the heat recovery system, boiler/economiser ash falls into hoppers under the equipment and can be periodically removed for disposal. The exact operating methods will dictate the appropriate sampling interval. It is important to collect samples of the material generated with and without operation of tube cleaning equipment, and careful documentation of these cyclical operations in relation to the sampling is essential.

Heat recovery ash is generally more homogeneous than grate ash, therefore, it is possible to collect smaller sample sizes without affecting the quality of the data generated. The total volume generated in any given period may be so small the sampler will decide to collect all of the available residue. Such was the case in both the regulatory and the research test programs outlined above. Since the mass of material collected in both cases was greater than that required for representative sampling, site methods were used to generate sub-samples for analyses.

### **Regulatory Sampling**

The boiler/economiser ashes from the Peel facility were collected at the end of every operating period marking the completion of a given set of operating conditions. Collected material included ash that settled in the hoppers during normal operation and the material dislodged during operation of the soot blowers. The entire quantity of the boiler and economiser ashes generated over the period was collected in separate 200

litre barrels for each boiler. Full depth tube cores were taken from the drum to produce approximately 5 litres of ash. Care was taken to ensure the samples do not absorb moisture during the cooling process since the material is slightly hygroscopic.

### **Research Sampling**

One of the objectives of the WASTE Program was to determine the partitioning of trace elements within the temperature regimes of the incinerator. Therefore, samples of ash were collected from hoppers #1, #2 and #3 via diversion chutes fitted to each of the boiler ash discharge hoppers. The entire quantity of ash generated in each hopper over each four hour test period was collected in 200 litre barrels. The soot blowing cycle for the boiler was initiated 30 minutes prior to attaching the barrels to the diversion chutes in the morning and again 30 minutes prior to removing the barrels after each test run. Typically, the diversion chutes needed to be cleaned out by hand prior to removing the sample barrels. This provided a means to monitor the quantity of boiler ash generated during each of the test periods, i.e., the volume of ash collected was monitored and the mass was calculating using the density of the ash. Sufficient full tube core samples for each four hour test period were taken to fill a 4.5 L steel container, sealed, weighed and allowed to cool.

### **6.6.4 Air Pollution Control System Residues**

This section is more relevant to residue from ESP's, and dry and semi-dry APC systems than to residues from wet scrubber systems. Typically, the quantities of ash generated are too voluminous to collect the whole stream, although attempts to monitor the total volume generated should be made to assist with later interpretation and calculations of mass balances. It may also be necessary to collect various samples from this stream to assess the potential variability in quality brought on by process changes in the system. Grab samples should be collected at fixed periodic intervals, however, the weight of each grab sample does not have to exceed 1 kg in weight. If it is possible to obtain samples from the various locations in the APC system, research sampling studies may attempt to identify speciation differences in the APC residues at different stages of the process.

### **Regulatory Testing**

The APC residue from the Peel facility was sampled from the pneumatic transport line that connects the bottom of the baghouse hoppers to the recycle and waste silos. The extraction pipe assembly, located after the last hopper of the second baghouse, was equipped with a ball valve to allow for periodic sampling. Samples were collected in a 20 litre bucket and transferred to a 200 litre drum for compositing.

The collection of fly ash samples from the APC was accomplished by opening the ball valve every half hour and filling the bucket with material. This procedure continued for

an 8 hour sampling period. Full depth tube cores were taken from the drum(s) to produce approximately 5 litres of ash.

### **Research Testing**

During the WASTE Program, grab samples from both the dry scrubber reactor and fabric filter were collected via a gate valve fitted to the Depac unit at the base of the respective hoppers. The Depac purge cycle was turned off during the test periods to allow residue to build up in the hoppers. Initially, attempts were made to collect separate dry scrubber reactor grab samples once every 30 minutes, however, since very little residue was captured, the sampling frequency was changed to once every hour. All the residue generated during each 1 hour period was purged and collected from the Depac. The fabric filter residue samples were collected from the Depac unit every half hour. Samples were subsequently collected by opening the valve and manually pressurising the Depac unit to fluidise the residue. The ash was collected in 4.5 L steel containers, then sealed, weighed and allowed to cool.

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