Sampling and Analysis of Solid Incinerator Refuse and Residue

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ABSTRACT

Physical and chemical analyses of solid refuse provide basic information for the design and evaluation of refuse reduction and disposal systems. Likewise, analyses of incinerator residue assist in evaluating the performance of incinerators.

The paper outlines a practical, interim method for obtaining physical and chemical data on refuse and residue. Sampling the refuse, fractionation and reduction of samples, drying, and preparation of laboratory samples are described. Chemical analyses and Btu determinations are by standard methods for coal and coke of the American Society for Testing and Materials (ASTM). Data obtained include physical composition, bulk density, percentages of moisture, carbon, hydrogen, oxygen, nitrogen, sulfur, chlorine, and inerts, as well as calorific value.

INTRODUCTION

Characterization of refuse by physical and chemical analysis, and by determination of the calorific value is fundamental to the engineering design of incinerator systems. Chemical analyses and Btu values of refuse and residue supplement furnace calorimetry data in arriving at valid mass and energy balances of incinerator tests. Although the precision of measurement for refuse does not need to equal that for purchased fuels, reliable methods are necessary for the testing of incinerators, the study of trends, and research purposes.

Sampling of complex refuse mixtures, such as solid industrial and municipal refuse, is a developing art that is basic to accurate analyses. Fortunately, the ASTM [1] methods of chemical analysis and Btu determination for coal and coke can be adopted for solid waste, but the special techniques for taking and preparing the refuse samples are still under development and have not been standardized. Additional tentative methods of analysis for organic matter are described by the APWA [2].

The purpose of this paper is to present and discuss the sampling procedures used by the authors on municipal refuse. These methods require a minimum of equipment but are time consuming. Undoubtedly the time and labor could be reduced by other mechanical aids, which would be justified as the number and frequency of samplings increase.

The present approach is a pragmatic one based on experience and practicality. It needs the statistical analysis that establishes the influences of the size and number of increments on the accuracy of the results. We know that the individual particles range in inorganic matter from almost 0 to almost 100 percent. The moisture content of particles can range from almost 0 to over 90 percent. The weights of individual particles range from almost zero to the upper limit set by the incinerator plant or test personnel. Obviously, for equal accuracy, the size of increments for a sample of sawdust would be much less than for a sample of a heterogeneous material like industrial or municipal refuse.
BULK DENSITY

Bulk density of refuse and residue are of interest in the sizing of storage bins, containers, trucks, crane buckets, and landfill volumes. Tonnages are sometimes estimated from bulk densities, either measured or assumed. Unless the weight and volume are carefully measured with full knowledge of the refuse composition, moisture content, and packing, the data are likely to be highly inaccurate.

Bulk density can be determined by filling a measured storage bin or pit at an incinerator plant by normal dumping of truckloads and levelling of the bin by a crane. The bin need not be filled level full inasmuch as the unfilled volume can be measured from the top. As reported elsewhere [3], in one instance household refuse was collected by packer trucks and dumped into a pit 29-to 30-ft. deep, with the results as shown in Table 1.

<table>
<thead>
<tr>
<th>Days, 1967</th>
<th>Moisture Content (%)</th>
<th>Before Settling</th>
<th>After Overnight Settling</th>
<th>Crane Grab during Pit Unloading, (lb/grab)</th>
</tr>
</thead>
<tbody>
<tr>
<td>March 18-19</td>
<td>26</td>
<td>349</td>
<td>375</td>
<td>1615</td>
</tr>
<tr>
<td>June 13-14</td>
<td>42</td>
<td>480</td>
<td>523</td>
<td>2084</td>
</tr>
</tbody>
</table>

Incinerator residue is also affected by the density of packing and moisture content. Densities of mixed residue can be calculated from weights loaded into 55-gal drums (7.35 ft$^3$). A few examples are given in Table 2.

<table>
<thead>
<tr>
<th>Residue</th>
<th>Bulk Density of Dry Residue (lb/yd$^3$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Metal cans</td>
<td>207</td>
</tr>
<tr>
<td>Paper and wood</td>
<td>280</td>
</tr>
<tr>
<td>Clinkers over 3 in.</td>
<td>1053</td>
</tr>
<tr>
<td>Clinkers 0.25 x 3 in.</td>
<td>1787</td>
</tr>
<tr>
<td>Ash, under 0.25 in.</td>
<td>1904</td>
</tr>
<tr>
<td>Grate siftings, high glass content</td>
<td>1782</td>
</tr>
<tr>
<td>Fly ash, no carbon</td>
<td>950</td>
</tr>
</tbody>
</table>

SAMPLING OF SOLID WASTES

Refuse

Incinerators usually receive a wide variety of materials, the moisture contents of which are higher than the air-dried level. This heterogeneity and moisture content complicate both the size and number of increments that must be taken, as well as the reduction of the sample to representative amounts that can be analyzed. Sampling at the storage pit during a burning test is one practical approach.

A suggested procedure for sampling municipal, commercial, and industrial refuse at a crane-and-pit operation follows: Assume an 8-h test.

Mix refuse in the active portion of the pit by a crane bucket.

At mid-point of each test hour take an increment of 300 to 400 lb by the crane bucket and deposit it in a box on a scale.

Weigh each increment. Add weights of the 8 increments, which will total in the range of 2400 to 3200 lb. Mix the pile by the crane, cut the pile in half, and discard one half after weighing the same. Weight losses are moisture, which should be prorated.

Store the remainder (1200 to 1600 lb) in a pile and cover with plastic sheets to prevent moisture loss. The pile constitutes the sample.

Hand-sort the sample as soon as possible by the categories given or other appropriate categories, each of which is definable and fairly homogenous:

(1) corrugated boxboard
(2) newspaper
(3) magazines, catalogs and books
(4) all other paper
(5) plastic film
(6) molded plastics, rubber leather
(7) wood, excelsior
(8) grass, hay, leaves, shrubbery
(9) food waste
(10) smalls, 1 dirt
(11) metals
(12) glass, ceramics, stones

Sorting is readily done by four or five men, one of whom places the refuse on a table, 36-in. high, for others to select and place categories into tared 55-gal open-top drums or plastic bags thumb tacked or taped to a rack close at hand.
Close and tag containers as filled. Weigh each category at end of fractionation.

The difference between the total of the increments and the total of the discarded half and categories, corrected for tares, is moisture loss during fractionation. Prorate the moisture loss back to the net weights of the components in proportion to their weights.

Prepare a sample of each component for the purpose of the moisture determination and succeeding analyses. The amount and method used will vary somewhat with the type and amount of the component and the equipment available, as explained in a later section.

Transfer the samples to metal pans, such as 22 x 22 x 6 in. deep, for heat-drying at 175 to 212°F. Weigh daily to the nearest gram until a constant weight is obtained on two successive days. Drying time is about 5 days. The moisture loss is equal to the weight loss of the sample while in the oven in addition to the previous prorations. The percent moisture in the component equals 100 times (initial corrected weight minus final weight) divided by initial corrected weight.

Reduce the size of pieces of dried material in categories (1) through (9) to 2-in. maximum size, mix, cone, and quarter to obtain a 0.5-lb. sample.

Some regaining of moisture by the samples will result from exposure to ambient air in the steps described thus far. No error will result inasmuch as the regained moisture will be determined during analysis and the correction to eliminate it will be made when the analysis is converted to the dry basis. Because of imperfect sorting in the hand-sorting step, pieces of other categories will be found in the dried sample. They should be removed and weighed, and the weights should be recorded for corrections later.

Categories (10) through (12) are dried as previously described but are then given special processing, as explained later.

Reduce the 0.5-lb samples of categories (1) through (9) to less than 2 mm in a knife mill such as the Wiley No. 3. Metal, glass, stone, and the like should be kept out of the mill inasmuch as good performance with fibrous and tough materials depends on sharp knives and close tolerances. Mix, cone, and quarter for a final sample of 1 pt for the laboratory. As necessary, categories (10) through (12) are reduced to laboratory samples by other means.

Incinerator Residue

The procedure for sampling and drying incinerator residue follows the same general principles as for refuse, although with simplifications. No single method suffices because the total residue consists of grate residue, grate siftings, fly ash deposited in settling chambers and on arches, collector dust, water clarifier solids, etc. It may be necessary to sample at each location. At some plants, grate residue, siftings, and settled fly ash may be collected steadily in a single conveyor trough.

Truckloads of the quenched residue discharged from a residue conveyor should be allowed to drain before weighing. During an 8-h test, an increment of grate residue enough to fill a 55-gal drum may be taken once an hour. When the increment is taken, the total residue coming off the full width of the conveyor should be included. The eight drums of residue sample from the 8-h test are then mixed on a pile, coned, and quartered until a sample of 55 gal is obtained. Heat-dry 15 lb for a moisture determination.

Sort the 55-gal drum of sample into two categories, combustible and noncombustible, and weigh. The sample of combustible should be heat dried and reduced by shredding or Wiley No. 3 mill to less than 2 mm in size. A pint sample is ample for the laboratory analyses.

Increments of 1 qt of steadily collected fly ash may be taken each hour. Settled fly ash that accumulates over a long period in a dry, hot settling chamber should be sampled at the end of the test because it normally continues to burn after being deposited.

A 0.5 lb of dried sample of fly ash is ample for laboratory analysis.

When incinerator residue is transported in water, part of the residue remains in the water as dissolved and suspended solids. Samples of the process water discharged from the plant may be taken, and the flow, determined. By evaporating the water from the samples, the amount and analysis of the solids may be determined.
SUGGESTIONS ON TECHNIQUES FOR SAMPLING AND SAMPLE REDUCTION

Procedures for reducing samples to a size suitable for laboratory analyses are influenced by variability in the component fraction and the rapid migration of moisture. Cool surroundings and minimum air movement are desirable when sampling, sorting, and reducing the size of samples. Exposure time of the samples to the ambient air is reduced by adequate preparation and manpower. Objective techniques must be followed to prevent distortion, bias, and error in the results.

The technique described uses a minimum amount of equipment. A power shredder is optional for reducing the samples.

Corrugated Boxboard

Except for moisture content, corrugated paper boxboard is relatively uniform. During sampling, metal banding should be removed and transferred to the pile metal samples, but it is not advisable to take the time to remove metal staples except from the dried sample before final grinding. When over 100 lb of the sorted component are to be reduced by hand to 3 lb, some of the original pieces may be rejected. For example, if 60 lb are available, every other piece may be rejected to a tared drum. A coupon of about one-tenth the original area is then cut by a cutting board (purchased from a stationery supplier) from the selected pieces. The coupons are dropped into a plastic bag or metal container with minimum opening. Loose dirt on the boxboard coupons must accompany the coupons into the sample.

It will be noted that some pieces are relatively moist and limp, whereas others are dry and crisp. Each type should be represented proportionately in the final sample with a minimum loss of moisture. The correction for loss of moisture is determined by weighing the total component and the sample and by comparing this weight with the original component weight.

Newspaper

Newspaper is basically a uniform, low-ash material except for moisture and soiling. It has usually absorbed moisture from garbage, grass, and rain. Wide variation in moisture content is experienced. A sample of 6 lb can be cut readily from a lot by hand, tearing across an entire newspaper and retaining approximately the same proportion each time.

If the weight of the entire component lot were 225 lb, for example, and stored in 10 plastic bags, the lot could be reduced in half by discarding every other bag in the order filled. The weights of the remaining bags should be known. From each of the five bags retained, every other newspaper may be rejected to a tared drum, and the alternate papers are sampled as above. This procedure approaches coning and quartering but has the advantage of minimum moisture loss.

Magazines, Catalogs, Books

The ash content of magazines varies widely, being lowest in the pulp paper and highest in glossy paper, such as cover stock. Samples may be taken by cutting coupons across the entire magazine, or by opening the magazine at the center fold and tearing or cutting half across it. The moisture loss of the reduction to about 6 lb should be determined by weighing both the sample and the rejected paper and prorating as described earlier.

All Other Paper

All paper not included in the three categories just described is combined as miscellaneous paper, which is usually the major category by weight. It consists largely of packaging waste. The individual pieces range from high Btu waxed paper to wet pulpboard, with much of it contaminated with food waste. In general, the moisture content of this category is over 25 percent. Despite the variety among the pieces, sufficient homogeneity is present for reduction of the component lot by taking 1-lb grabs by hand from each bag of 30 to 50 lb and combining the grabs in a single plastic bag, all without significant loss of moisture. The final reduction to 5 to 6 lb for heat-drying will usually require further size reduction by tearing or shearing, remixing, and dividing. Moisture corrections should then be applied as before. Dirt that separates from the paper must also be proportioned fairly between the sample and rejects.

Plastic Film

Thin sheet plastic (under 1/32 in.) and plastic film consist mainly of cellophane wrappers and polyethylene, with lesser amounts of other types of plastics. The material is transparent, although colored, and nearly opaque; plastic bags are also present. Moisture and food particles adhere to the
surface rather than being absorbed, as in the case of paper. The entire, weighed lot in this category may be sheared to under 10-in. square and remixed before a sample of about 3 to 5 lb is taken by 0.5-lb hand grabs from different parts of the pile. Mixing is particularly important in this category because the calorific value (dry basis) of polyethylene is close to 19,900 Btu lb, whereas the value for cellophane is only 7500 lb. Moisture corrections for losses occurring during sample reduction should be applied. Partial and damaged rolls of plastic film are sometimes present, in which case the sample for drying should include a proper proportion of this material.

**Molded Plastic, Rubber, Leather**

This grouping is the result of experience and is a compromise for simplification of grouping of several physically tough materials. Shoes are sometimes a combination of the three types. Ladies handbags, tires, molded plastic articles, garden hose, plastic bottles, plastic toys, artificial flowers, foamed rubber, styrofoam, and a host of other articles are included. The category is under 3 percent of the total refuse today but is increasing. The moisture content is low and the calorific value is relatively high.

Inasmuch as the total moist weight has been obtained previously, it is suggested that the category be spread out on a clean area for classification into the following groupings:

1. tires
2. hose
3. bottles
4. foam
5. shoes
6. handbags
7. molded brittle shapes
8. all other

By means of a saw or shears, cut representative sections of the materials and combine in the proportion that they exist in the original category, collecting a total of 4 to 5 lb. Tire samples are obtained by sawing radial cuts toward the center of the tire. Shoe samples should include material from the toes, uppers, and heels.

**Wood**

The wood category includes pieces of lumber, twigs, and branches over 1 in., excelsior, furniture wood, boxes, picture frames, clothes pins, and painted and unpainted wood. The moisture content of green wood is usually lowest near the outer surface and highest near the center. Saw cuts across the sections are advisable for reducing the samples. Pieces for the drying pan should be split to expose drying surface if they are over 1-in. thick. A final sample of 5 or 6 lb may be sufficient.

**Grass, Hay, Leaves, Shrubbery**

These four components may be combined if the amounts are small, or they may be separated into several categories for greater accuracy if the amounts are substantial. The moisture content may exceed 50 percent. The weight that can be put into the drying pan is about 8 lb, provided that the shrubbery is hand sheared into pieces small enough to nest together.

**Food Waste**

Food waste usually contains over 50 percent moisture and consists of fats and bones, as well as cereals, fruit, vegetables, and meat. Food should be removed from bags and jars without attempting to recover minor amounts. Cut open fruit, potatoes, and other large pieces to decrease drying time. A final sample of not over 10 lb is suggested.

**Smalls, Dirt**

Smalls and dirt are the small bits under 1 in. remaining on the sorting table after other categories have been picked off. Small pieces of all other categories are present.

It is neither necessary nor advisable to produce a more thorough separation, because the resultant homogeneity of this fraction is more than adequate for 12 to 16 lb of drying sample. After drying, a pound or two may be separated by hand-picking of the glass, metal, food, and paper, from the oversize fraction on a 4 mesh/in. sieve. The fractions are analyzed separately when they contain combustibles.

**Metals**

This category includes steel and aluminum cans, foil, wire items, bolts, nails, auto parts, die and sand castings, etc. Paper labels, paint and coatings, and food remainants contribute a significant amount of that which is combustible, the extent of which can be determined after the category is dried. A
10-lb sample will fill the pan. Be sure that pressurized containers are punctured and that all other containers are open before drying.

**Glass, Ceramics, Stones**

The nonoxidizable or inert fraction of the refuse falls into this category. Labels and food are present. Food in excess of that sticking to the glass should be removed and included in the food category. Plastic and metal bottle caps belong with molded plastics and metals, respectively. A sample of 20 lb is sufficient for drying.

### REFUSE ANALYSES AND BTU DETERMINATIONS

Chemical analyses by established procedures developed for coal and coke are applicable for solid waste. The analyses of interest for incinerator refuse and residue are among the following, all obtained from the samples prepared as previously described.

#### Table 3

<table>
<thead>
<tr>
<th>Item</th>
<th>ASTM Method</th>
</tr>
</thead>
<tbody>
<tr>
<td>Moisture (Lab.)</td>
<td>D-271-68</td>
</tr>
<tr>
<td>Carbon</td>
<td>D-271-68</td>
</tr>
<tr>
<td>Hydrogen</td>
<td>D-271-68</td>
</tr>
<tr>
<td>Oxygen</td>
<td>D-271-68</td>
</tr>
<tr>
<td>Nitrogen</td>
<td>D-271-68</td>
</tr>
<tr>
<td>Sulfur</td>
<td>D-271-68</td>
</tr>
<tr>
<td>Chlorine</td>
<td>D-2361-66</td>
</tr>
<tr>
<td>Ash</td>
<td>D-271-68</td>
</tr>
</tbody>
</table>

These items total 100 percent.

The higher calorific value in Btu/lb is obtained by D-2015-66 with an oxygen bomb calorimeter.

Carbon, hydrogen, oxygen, and sulfur are of interest for the calculation of theoretical combustion air. Chlorine is assuming increasing importance because of its corrosive effects and contribution to air pollution. Chlorine in plastics is expected to increase, and the amount of chlorine in other components is largely unknown.

Proximate analyses may be run on refuse samples, but the results are more interesting than useful for municipal waste. Proximate analyses include moisture, volatile matter, fixed carbon, and ash. The same moisture and ash contents are included in the ultimate analysis. "Volatile matter" indicates the weight loss of organic matter by rapid distillation at red heat, whereas "fixed carbon" is the unvolutalized matter, mainly carbon. "Volatile matter" is not the same as "volatiles," used in sanitary engineering. The proximate analysis of a sample containing metals gives an erroneous fixed carbon and ash because the metal gains weight while oxidizing.

Because of the small amount of sample required for the analyses, less than 0.5 g for the analyses and about 0.5 g/1000 Btu/lb in the refuse, it is highly important that the final sample be nearly homogeneous. Refuse presents several limitations in this respect because finely chopped fibrous material, such as textiles, tend to ball and segregate from granular material, such as rubber and molded plastics. Food waste is somewhat greasy, adding a further complication.

Until means have been developed to mill such materials to powder, so that all organic component types can be blended together in the proportions found in the original refuse, it is advisable to analyze the components separately. The composite analysis and Btu value can be calculated from those of the components by simple proportion and addition.

### EQUIPMENT REQUIRED

To accomplish the sampling and drying of a solid waste, such as municipal incinerator refuse and residue, the following equipment and supplies are required for an incinerator test of 8 h:

- Platform scale (500 lb or larger)
- Open top box (4-x 6-x 6-ft deep or larger) to hold 300- to 400-lb or 0.25 crane grab
- Laboratory scale (20 kg)
- Drying oven (special)
- 8 galvanized-iron pans (22-x 22-x 6-in. deep)
- Plastic sheets (10-ft wide)
- 100 plastic bags (2 mill thickness, 55 gal)
- Bag rack and table (special)
- 10 steel drums (clamp top)
- 5 five-gal covered cans
- Hand shears, putty knife, thumbtacks, hammer, cord, tags, marking pens, hand saw, or band saw
- Cutting board (12 x 12 in.)
- Shovels (wide)
- Wiley No. 3 mill
- 24 qt jars with covers and rings
- Power shredder (optional)
Approximately 25 man-days are required for the preparation, sampling of refuse and residue, and preparation of final laboratory samples for analysis, based on a single, 8-h incinerator test.

**DISCUSSION**

Ideally, if the initial gross sample could be shredded without significant dust and moisture loss, it would be simple to sample the resultant product and thus obtain a single, small, representative sample for drying. Furthermore, if the dried sample could be ground to a powder, a laboratory sample could be prepared.

Because the equipment to accomplish this ideal is not available, it is necessary to sort by fairly homogeneous categories.

If available, a power shredder or chopper would be helpful in reducing each category of organic matter to a maximum size that would permit resampling for the sample to be heat-dried. The moisture loss due to the windage of a hammermill shredder could be prorated by weighing before and after shredding. Dust losses can be minimized by a cloth bag to catch the shredded sample or by other obvious means.

To utilize existing ASTM analytical tests, it is necessary that a sample be represented by 0.5 to 3.0 g. The fine shredding of fibrous material by Wiley No. 3 mill does not result in powder. Textiles form tufts or balls; paper becomes fluffy; rubber and molded plastic become small grains; and plastic film is in flake form. These forms segregate readily when mixed. Hence, it is better to sample and analyze the components separately, at least for the present, and calculate the composite analysis and Btu value from the data on the components.

Typical results have been presented earlier [3] for both incinerator refuse and residue.

The organic matter, such as paper, textiles, and food, readily exchanges moisture with the ambient air. Initially they tend to lose moisture. On removal from the dryer, the pans should be weighed promptly because the organic contents will reabsorb moisture. Drying in ambient air will not give uniform results because the final moisture content increases and decreases with air humidity. If a sample of nonputrescible refuse is weighed in its original moist condition, it may be air dried, and finally heat dried to determine the total moisture.

If it should be necessary to ship or store the samples prior to heat drying, they may be stored in double plastic bags, packed in the steel drums, and refrigerated below freezing. Decomposition is thus prevented, as well as moisture loss, until drying.

The final Wiley-milled samples may be stored in sealed glass jars, properly labelled.

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**REFERENCES**

