E. S. Domalski

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Test Procedures for the Determination of the Gross Calorific Value of Refuse and Refuse-Derived-Fuel By Conventional Bomb and Large Bomb Calorimetry

SUMMARY OF THE 1979 FISCAL YEAR RESULTS

D. R. Kirklin, J. Colbert, P. Decker, S. Abramowitz, and E. S. Domalski

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U.S. Department of Commerce
National Bureau of Standards
Washington, DC 20234

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Prepared for:
U.S. Department of Energy
Assistant Secretary for
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Office of Energy from Municipal Waste
Washington, DC 20585

and

U.S. Environmental Protection Agency
l Environmental Research Laboratory
l Hazardous Waste Research Division
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U.S. DEPARTMENT OF COMMERCE, Malcolm Baldrige, *Secretary*
NATIONAL BUREAU OF STANDARDS, Ernest Ambler, *Director*

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Resource Recovery Facility:

MSW is shredded by a primary shredder; the light fraction is separated by a light air classifier and collected in cyclone No. 1; the large particles are separated by a trommel and reshredded by a secondary shredder.

Figure 3 U.S. Bureau of Mines, College Park, Md.

Resource Recovery Facility:

MSW is shredded by a primary shredder; the light fraction is separated by a light air classifier and collected in cyclone No. 1; the small particles are separated by a trommel with 3/4 inch openings and further separated from fine glass and dirt by a No. 14 mesh screen.

Figure 4 U.S. Bureau of Mines, College Park, Md.

Resource Recovery Facility:

MSW is shredded by a primary shredder; the heavy fraction is separated from the light fraction by a light air classifier; ferrous metals are removed from this heavy fraction by a magnetic separator; and the light portion of this heavy fraction is separated by a primary air classifier and collected in cyclone No. 2; the large particles are separated by a trommel and reshredded by the secondary shredder.

Figure 5 U.S. Bureau of Mines, College Park, Md.

Resource Recovery Facility:

MSW is shredded by a primary shredder; the heavy fraction is separated from the light fraction by a light air classifier; ferrous metals are removed from this heavy fraction by a magnetic separator and the light portion of this heavy fraction is separated by a primary air classifier and collected in cyclone No. 2; the small particles are separated from the large particles by a trommel with 3/4 inch openings and further separated from fine glass and dirt by a No. 14 mesh screen.

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Resource Recovery Facility:

MSW is shredded by the primary shredder; the heavy fraction is separated from the light fraction by the light air classifier; ferrous metals are removed from this heavy fraction by a magnetic separator and the heavy portion of this heavy fraction is separated by the primary air classifier; the large particles of this heavy portion are separated by a trommel and reshredded by the secondary shredder; the lighter portion is then removed by the secondary air classifier and collected in cyclone No. 3.

Figure 7 U.S. Bureau of Mines, College Park, Md.

Resource Recovery Facility:

MSW is shredded by the primary shredder; the heavy fraction is separated from the light fraction by the light air classifier; ferrous metals are removed from this heavy fraction by a magnetic separator; and the heavy portion of this heavy fraction is separated by the primary air classifier; the large particles of this heavy portion are separated by a trommel and reshredded by the secondary shredder; the heavier portion is then separated from the lighter portion by the secondary air classifier; aluminum is removed from this heavier portion by an electrostatic separator leaving only the heavy organic material.

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Resource Recovery Facility:

MSW is shredded by the primary shredder; the heavy fraction is separated from the light fraction by the light air classifier; ferrous metals are removed from this heavy fraction by a magnetic separator and the heavier portion of this heavy fraction is separated by the primary air classifier; the small particles of this heavy portion are separated by a trommel with 3/4 inch openings and allowed to fall into a mineral jig filled with water and the organic wastes are floated off the top of the mineral jig.

Appendix A

Figure 1 RDF-3 Reduced to 0.5 mm,
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TEST PROCEDURES FOR THE DETERMINATION OF THE GROSS CALORIFIC VALUE OF REFUSE-DERIVED-FUEL BY CONVENTIONAL BOMB AND LARGE BOMB CALORIMETRY

SUMMARY OF THE 1979 FISCAL YEAR RESULTS

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ABSTRACT

This report provides the results of investigations during fiscal year 1979 to develop test procedures for the determination of the calorific value of refuse and refuse-derived-fuel (RDF) by means of oxygen bomb calorimetry. The results of 138 calorimetric measurements are discussed, along with 32 calorimeter calibration measurements, for 20 different RDF samples. In addition, determinations have been carried out on these RDF samples for air dry loss, residual moisture, furnace ash, bomb ash, and sulfur content to correct the calorimetric data for their presence; the latter group of measurements amounts to 283 experiments.

The calorimetric (and related) measurements can be categorized into five classes: (1) measurements carried out in conjunction with ASTM round robin testing of RDF-3, (2) measurements on an Americology RDF extracted using both selective and non-selective sampling methods, (3) measurements required to compare data obtained from the small (2.5 gram capacity) and large (25 gram

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[†] Summer student from the University of Maryland, Baltimore County Campus during the summer of 1978 and 1979.

capacity) bomb calorimeters, (4) measurements carried out in the large bomb calorimeter on seven processed samples of different particles sizes obtained from New Castle County, Delaware MSW, and (5) measurements on some RDF's of special interest.

Five appendices include additional information on: the evaluation of data on higher heating values obtained during ASTM round robin testing of RDF-3, the large bomb calorimeter, spectrochemical analysis of bomb residues, conversion tables, and calculation of total moisture in a RDF sample.

An evaluation of data on higher heating values determined during the ASTM round robin testing of RDF-3 showed that the repeatability (within-lab agreement) and reproducibility (between-lab agreemtn) are 250 and 750 Btu/lb when values are converted to a dry basis. These variabilities are 5 and 7.5 times greater, respectively, than the precision levels accepted in ASTM protocols for determining the higher heating values of coal or coke on a dry basis (see Appendix A).

Measurements on three RDF samples in both small (2.5 gram-capacity) and large (25 gram-capacity) bomb calorimeters showed that equivalent high precision can be obtained in both calorimeters. The large bomb calorimeter has the advantage that it can accommodate RDF samples whose particle size is as large as 25 mm (1 inch).

The mean and standard deviation of the calorific values obtained for 20 different RDF samples give 9482 and 553 Btu/lb, respectively, on a moisture-ash free basis using the furnace ash method to determine the ash content. Thse results support the generally accepted value of 9500 Btu/lb as the higher heating value for RDF on a moisture-ash free basis.

I. Introduction

This report provides the results of an investigation which was carried out at the National Bureau of Standards during fiscal year 1979 under a research agreement with the U.S. Department of Energy and the U.S. Environmental Protection Agency on the development of test procedures to determine the calorific values of refuse and refuse-derived-fuel (RDF) by means of oxygen bomb calorimetry. This is the third report of studies on the development of such test procedures. Earlier reports [1,2][+] should be consulted to obtain further information on the efforts in fiscal years 1977 and 1978.

This investigation responds to the Resource Conservation and Recovery Act of 1976 (PL 94-580) in which the National Bureau of Standards has been mandated to furnish guidelines for the development of specifications for materials which can be recovered from wastes and have been otherwise destined for disposal. The specifications refer to the physical and chemical properties and related characteristics of such materials.

The results of this report are of interest to the Committee on Performance Test Codes for Large Incinerators (PTC-33) of the American Society for Mechanical Engineers (ASME). This committee has indicated the need to find more accurate test methodology for the determination of calorific values of refuse samples which will more reliably represent the heterogeneous array of orignal solid waste. It is the opinion of many engineers that one cannot accurately sample a multi-ton pile of refuse down to a few grams for bomb calorimeter measurement. In addition, these engineers suggest that size reduction to small particle dimensions (i.e., 2 mm or 0.5 mm) could alter the chemical nature of this heterogenoeous material. The latter assumption is somewhat severe;

[+] Number in brackets identify references at the end of this report.

however, experimental evidence which would confirm or disprove this idea is needed. If improved test methodology to determine the calorific value of refuse and RDF could be found, a better means would be available to evaluate whether or not large incinerators or refuse-fired boilers are in compliance with their contract performance specification.

The results of this report are also of interest to Committee E-38 on Resource Recovery of the American Society for Testing and Materials (ASTM). This committee is involved in the development and publication of test procedures needed to characterize RDF so that it can be established as an article of commerce. A laboratory procedure which yields reproducible results will better equip commercial testing laboratories to certify accurately the energy content of RDF of various compositions. As an article of commerce RDF can be bought and sold as a regulated low-sulfur fuel to supplement coal-fired systems.

Project Objectives

The major objectives of this research project are:

(1) to investigate the size reduction and homogenization required to obtain representative samples of refuse and RDF,

(2) to evaluate calorimetric methods for conventional-size (2.5 gram capacity) combustion bomb calorimeters and an NBS large (25 gram capacity) combustion bomb calorimeter; and

(3) to develop test procedures for determining the calorific value of refuse and RDF samples using bomb calorimetric methods.

One of the most significant property of a fuel is its heating value (calorific value). For the past 100 years, the calorific value of solid fuels has been determined in a reliable way by the methods of oxygen bomb calorimetry. Traditionally, small (1-3 gram) samples are burned in an oxygen bomb calorimeter to estimate the calorific value of gross quantities of a fuel. Since the techniques of oxygen bomb calorimetry are well understood, the accuracy of estimating the calorific value of gross quantities of a fuel is dependent upon making measurements on small (1-3 gram) fuel samples. The accuracy of these measurements is, in turn, dependent upon the representativeness of the analysis samples which are extracted from gross lots of the fuel. The traditional fuels, such as coal and coke, are homogeneous enough so that techniques [3,4] have been developed for obtaining (1-3 gram) analysis samples which are representative of the gross lot of the fuel. The heterogeneity of refuse has made it necessary to re-evaluate the test procedures for determining the calorific value of coal and coke before they can be applied in a reliable manner to refuse and RDF. This heterogeneity problem can be attacked by either of two approaches. First, one may choose a representative gross sample of refuse or RDF and through appropriate particle size reduction and blending, obtain a representative (1-3 gram) sample. Second, one may construct combustion calorimeters that can handle larger samples of RDF which are more representative of the gross lot of the RDF fuel. NBS is investigating this problem from both approaches. One method involves the processing of heterogeneous samples to obtain a homogeneous sample from which a small (1-3 gram) representative sample can be selected for use in the NBS conventional-size calorimeter. The other method involves the design and the construction of large calorimeters capable of making calorific value measurements on large (kilogram-size) samples of refuse and RDF. The latter approach impacts upon the advancement of the state-of-the-art of calorimetry.

This report will present calorific values measured in the NBS conventional size (2.5 gram capacity) oxygen bomb calorimeter on highly processed "homogeneous" analysis samples of RDF. It also provides calorific values measured in the (25 gram capacity) oxygen bomb calorimeter which was built at NBS for less processed RDF samples.

The RDF samples that were studied can be placed in five arbitrary classes. The first class consists of those RDF's investigated as part of ASTM E-38.01 (Subcommittee on Energy of the ASTM Committee E-38 on Resource Recovery) Round Robin testing of RDF-3. The second class deals with an Americology RDF sample from which sub-samples were extracted for calorimetric measurements in both a selective and a non-selective manner. The third class consists of those RDF samples used in comparative studies of the same RDF investigated in both the NBS conventional size (2.5 gram capacity) and the NBS large (25 gram capacity) oxygen bomb calorimeters. The combustible fractions of a sample of municipal solid waste (MSW) from New Castle County, Delaware, which had been processed through the U.S. Bureau of Mines Resource Recovery facility in College Park, Maryland constitutes still a fourth class of RDF samples. The fifth class of RDF samples consists of several RDF's from different parts of the USA that had some unusual characteristics or history which prompted calorific value measurements to ascertain what variations might be present.

The RDF's investigated provide useful answers to all of the objectives of this research project. Comparison of the methods and results of samples which had received various degrees of processing and were investigated in both the 2.5 gram capacity and the 25 gram capacity bomb calorimeters provides information on the effects of the size reduction and homogenization. The variability of the calorimetric data provides some insight into the extraction of representative RDF samples. The comparison study on the same

RDF's investigated in both the small and large bomb calorimeters will help to evaluate the methods required to measure calorific values in a reliable manner by the use of either the conventional size or larger bomb calorimeter. Also, test procedures were developed to be used for the measurement of the calorific value of RDF samples in both bomb calorimeters. In addition, RDF's with unusual characteristics provide information on the general properties of RDF. Thus, this report addresses itself to all of the objectives of this project and also provides some general information on the properties of refuse and refuse-derived-fuels (RDF).

II. Experimental

A. Materials

1. Benzoic Acid

(a) NBS Standard Reference Material 39i with a certified energy of combustion of 26434 ± 3 $J \cdot g^{-1}$ at standard bomb conditions was used to calibrate the conventional-size (2.5 gram capacity) bomb calorimeter.

(b) Fisher Scientific Company's certified benzoic acid with a measured energy of combustion of 26437 ± 3 $J \cdot g^{-1}$ was used to calibrate the large (25 gram capacity) bomb calorimeter.

2. Oxygen

Ultra High Purity (UHP) grade oxygen was supplied by Matheson Gas Products. This oxygen is certified by the supplier to contain combustible impurities not exceeding 0.002 percent and total impurities of less than 0.05 percent.

3. ASTM Round Robin RDF-3 Samples [5]

Refuse-derived-fuel-3 (RDF-3) is defined as a shredded fuel derived from municipal solid waste (MSW) which has been processed for the removal of metal, glass, and other entrained inorganic material. Generally, this material has a particle size such that 95 weight percent passes through a 2-inch square mesh screen.

The round robin testing of RDF-3 was coordinated through ASTM Subcommittee E-38.01 on Energy. Each laboratory participating in the ASTM round robin testing program received five samples of RDF-3 weighing about 2.5 kg each. Four were used for sieve analysis while the fifth was processed after air drying to 0.5 mm particle size for physical and chemical analysis. Gram-size amounts of milled sample were used in the various analyses.

Samples of RDF-3 were prepared at the National Center for Resource Recovery (NCRR), Washington, D.C., and were distributed to various participating laboratories for two preliminary series or zeroth rounds of round robin testing. Six laboratories participated in the first and second preliminary rounds, however, the data obtained appeared to have a wide dispersion. It was felt that a period of acclimation was required for laboratory analysts to acquire some experience in processing and analyzing RDF-3 samples.

(a) First Round Robin Sample

The first round robin sample was prepared at NCRR, Washington, D.C. Eight laboratories participated in the first round robin tests, which took place in June and July of 1978.

(b) Second Round Robin Sample

The second round robin sample was prepared at NCRR, Washington, D.C. Twelve laboratories participated in the second round robin tests, which took place in December 1978 and January 1979. In an effort to ascertain whether improved precision could be obtained for higher heating values, moisture, ash, elemental analysis, and volatile matter, an inter-laboratory reference or comparison sample was also prepared. A sample of RDF-3 from NCRR was shipped to the Wisconsin Electric Power Co., Milwaukee, WI for milling down to 0.5 mm particle size. The milled sample was thereafter distributed to the participating laboratories in quantities of about 200 grams. It was hoped that the comparison sample would eliminate possible biases which might have been introduced into the particle size reduction operation at each of the participating laboratories.

(c) Third Round Robin Sample

The third round robin sample was prepared at Americology (Division of American Can Co.), Milwaukee, WI. As with the second round of testing, twelve laboratories participated in the third round of testing during May and June of 1979. Again, an interlaboratory comparison sample was prepared.

This time, a sample of RDF-3 was shipped from Americology to the Oak Ridge National Laboratory, Oak Ridge, TN, for milling to 0.5 mm particle size. Distribution of the milled sample was made to the participating laboratories in quantities of about 200 grams.

(d) Bituminous Coal Round Robin Sample

In an effort to assure that the testing laboratories had good quality control over their test procedures for higher heating value, residual moisture, and ash content determinations, another series of round robin tests was carried out using a NBS Standard Reference Material (SRM) bituminous coal sample (1632a). Quantities of 100 grams were distributed to six laboratories for test during February and March of 1980. This SRM had a particle size of about 0.25 mm (-60 mesh).

4 Teledyne National RDF

(a) RDF Pellets

A 20 kilogram sample of extruded RDF pellets was received from the Baltimore County Resource Recovery Facility operated by Teledyne National in Cockeysville, MD. The Teledyne National pellets are cylindrical in shape with a diameter of 2.5 cm (1 inch), and are broken-off into lengths of about 2.5 to 7.5 cm (1 to 3 inches). A portion of the extruded pellets was broken up in an arbor press or put through a grinder and run through a micromill to yield a 0.5 mm. (-10 mesh) powder. The powdered RDF sample was homogenized in a vee blender to provide an analysis sample.

(b) RDF-3 (Fluff) Sample

Two samples of RDF-3 (fluff) weighing approximately 3 kg each were obtained from Teledyne National's Resource Recovery facility in Cockeysville, Md. One sample remained at NBS and the other was sent to Gilbert Associates, Inc. in Reading, PA. The sample was analyzed by both laboratories using proposed ASTM E-38 test procedures following single step and two step moisture determinations.

5. ECO FUEL-II RDF

A 20 kilogram sample of ECO FUEL-II RDF was received from the Combustion Equipment Associates Facility at Bridgeport, CT and had a particle size of approximately 0.42 mm. (-40 mesh).

6. Americology RDF-3 (Fluff)

The Americology Resource Recovery Facility processes the municipal solid waste from the city of Milwaukee and supplies the Wisconsin Electric Power Company with RDF-3 which is co-fired with coal to generate electricity. A 55 gallon drum of RDF-3 (Fluff) was obtained from Americology.

7. New Castle County, Delaware MSW

A representative sample of municipal solid waste (MSW) from New Castle County, Delaware was shipped to the Bureau of Mines' College Park, Md. Resource Recovery Facility in March 1979. The College Park Resource Recovery Facility has seven sampling points within the processing stream from which combustible material can be collected. The New Castle County, Delaware, MSW was processed through the College Park Facility and representative samples were obtained from each of the seven sampling points.

8. Steam Size Reduction RDF

This RDF was produced by a novel technique at Burke, Davoud, and Associates in Richmond, VA. The process is similar to that used to make puffed cereals. The refuse is pressurized with steam in a closed container. An instanteous release of the pressure causes the steam and heated refuse to expand and thus be reduced in particle size to an RDF.

9. NCRR Storage Pit RDF (Charred Sorage Sample)

This is RDF which had charred during storage. The National Center for Resource Recovery had processed some Washington, D.C. municipal solid waste into RDF. The RDF was stored in Washington, D.C. for approximately 10 months. When the RDF was being removed for a test burn in an industrial boiler, portions of the RDF were found to have spontaneously undergone a chemical reaction to form a partly charred RDF. A grab sample of the RDF char was obtained for a few calorific value measurements.

10. NCRR RDF (Low Ash Sample)

The National Center for Resource Recovery (NCRR) prepared an RDF from office waste collected in various government buildings in the Washington, D.C. area. The RDF was prepared for a test burn in one of the boilers at the Pentagon in Washington, D.C. A grab sample was obtained from NCRR for measurement of the gross calorific value of RDF produced from this collection of office waste.

B. Oxygen Bomb Calorimeters

1. 2.5 Gram Capacity Bomb Calorimeter

The NBS conventional size (2.5 gram capacity) oxygen bomb calorimeter which was designed at NBS and built by Precision Scientific Co. is an isoperibol (isothermal jacket bomb calorimeter. The calorimeter has a history of providing high precision results with fuels and various materials of high purity.

2. 25 Gram Capacity Bomb Calorimeter

A large (25 gram capacity) oxygen bomb calorimeter was designed and built at NBS for high precision measurement on large samples. The two calorimeters are very similar in design except for the sample size capacities. Details of the large (25 gram capacity) oxygen bomb calorimeter were presented earlier [2] and are also described briefly in Appendix B.

C. Sample Preparation Apparatus

1. Steelman 450°F Electric Bake Oven

A model 568ETC A, 300 cubic foot oven was used for air-drying and total moisture determinations. It was equipped with movable shelves to accommodate drying racks whose volume was one cubic foot, and had an upper temperature limit of 450°F. The drying oven was equipped with a Portlow model RFC-15 24 hour chart recorder and programmer.

2. W. W. Grinder Corporation Hammer Mill

A model F-21-M-18, 5 horsepower hammer mill was used with either 3/4 inch or 1/4 inch screen.

3. Wiley model 4 Laboratory Mill

The Wiley model 4 laboratory mill has a 1/2 horsepower motor and performed size reduction of RDF samples through either a 2 mm or 0.5 mm screen. The mill has 6 stationary blades and 4 rotating blades.

4. Patterson Kelley Company Twin Shell Dry Blenders

These vee blenders were used with an 8 quart stainless steel twin shell and a 26 prong rotation mixer-intensifier rod to enhance the homogenization of RDF samples. Lucite shells of 1 or 3 quart capacity were also used.

5. Carver Laboratory Press, Model M

The Carver Laboratory Press was a hydraulic press capable of exerting 50,000 pounds of force. Dies of 1 3/8 or 5/8 inch diameter bores were fabricated in the NBS Instrument Shops and used in conjunction with the press to prepare pelleted RDF samples for various determinations.

D. Sample Preparation Procedure

The processing of an RDF sample involved four basic steps. RDF samples were: (1) dried, (2) milled to reduce the particle size, (3) blended to a "homogeneous state" and (4) pelletized into analysis samples.

1. Sample Drying

As-received RDF samples were dried in either a one step or two step method. The one step method was performed in the drying oven set at 107 ± 3°C for 24 hours. The two step method consisted of air-drying of the sample in the drying oven at 37 ± 3°C until the sample was in equilibrium with ambient conditions. A residual moisture determination was carried out subsequently on the analysis samples and combined with the air-dry-loss to compute the total moisture of the sample (see Appendix E).

2. Particle Size Reduction

Samples were milled to either 2 mm or 0.5 mm particle size for analysis purposes. RDF samples larger than 3/4 inch were processed through the 5 h.p. hammer mill to obtain particles that could pass through either a 3/4 inch or 1/4 inch metal screen. If more processing was required, the material was then milled in a 1/2 h.p. Wiley laboratory mill to pass through a 2 mm stainless steel screen. When further particle size reduction was required, the 2 mm material was processed through the Wiley laboratory mill again until it passed through a 0.5 mm screen.

3. Sample Homogenization

The 0.5 mm RDF was coned and quartered to obtain a laboratory sample which would fit into the 8 qt. twin-shell dry blender. The laboratory sample was then blended throughly in the twin shell blender.

4. Preparation of Pelletized Analysis Samples

After stopping the twin shell blender, analysis samples were immediately drawn from the blended RDF and pressed into pellets with the Carver Laboratory Press.

E. Calorimetric Procedure

Determination of test procedures for measuring the calorific values of refuse and RDF is one of the objectives of this project. Therefore, a detailed description of the calorimeteric procedure will be presented in the Results and Discussion Section of this report. Only a brief description of the calorimetric procedure will be presented here. A detailed description of the 25 gram capacity bomb calorimeter is provided in Appendix B.

The pelletized RDF sample was weighed into either a platinum, stainless steel, or quartz crucible. The crucible and sample were then placed in the crucible support of the bomb, which contained either 1.00 cm^3 (small bomb) or 10.0 cm^3 (large bomb) of water. A 2cm (small bomb) or 7 cm (large bomb) length of 0.003 inch diameter platinium wire was connected to the bomb electrodes and placed in contact with the sample. The bomb was sealed and then filled with 30 atm (40 atm-large bomb) of ultra high purity oxygen. The ambient temperature was recorded during the bomb-filling operation. The loaded bomb was then placed into the calorimeter vessel, which contained a measured amount of water. The calorimeter vessel was enclosed in a submarine vessel and immersed in a constant temperature water bath. The calorimeter fluid was then heated electrically to 24.98°C and then allowed to rise at a rate of 0.001°C per 100 seconds. The rate of rise was related to the stirring energy imparted by the calorimeter stirrer and the thermal leakage from the calorimeter jacket. Time and temperature measurements were recorded from a frequency counter connected to a quartz temperature probe. Integrated frequencies were recorded every 100 seconds. At a frequency corresponding to 25°C, the sample was ignited by discharging a 32,000 μf capacitor, which had been previously charged to 23V, through the platinum wire fuse. Integrated frequencies were recorded throughout the main reaction period and also during the after period when thermal euilibrium had been obtained.

The calorimeter was opened and the bomb removed. The bomb was opened and the original liquid contents and the subsequent bomb washings were weighed in a polyethylene container. Weighed aliquots of the bomb washings were analyzed for total acid and sulfur content.

The final value obtained in a calorimetric measurement is the ratio of the algebraic sum of the amount of energy released into the calorimeter, corrected for energy released by accompanying side reactions, and the calorimeter sample mass corrected for the amount of moisture and ash. Residual moisture and ash determinations were carried out concurrently on pelleted samples with the calorimetric determination.

The residual moisture sample is weighed into a dry, weighed porcelain crucible and then dried in a moisture oven maintained at 107 ± 3°C. The residual moisture was used to correct the mass of the RDF calorimetric sample to the dry basis.

The amount of ash in the calorimetric sample was determined by two methods. In one method, the mass of the bomb residue is determined by weighing the dry crucible and residue upon completion of the calorimetric determination. In the other method, an ash sample is weighed in a dry weighed porcelain crucible and placed in a furnace. The temperature of the furnace is allowed to rise slowly in a 4 hour period to 575 ± 25°C and is then maintained at that temperature for 2 hours. The amount of ash found in the determination is used to correct the mass of the calorimetric sample to an ash-free basis.

III. Results and Discussion

A. Bomb Calorimetric Measurements

1. General Discussion

Different methods for bomb calorimetric measurement of heat of combustion are available for various types of materials. Certain modifications exist depending on the precision desired and the properties of the material investigated. Apparatus and methods for the precise calorimetric measurement of the heat of combustion are present in the literature [6,7,8]. The methods followed in this laboratory are patterned after the precise measurement techniques utilized by Jessup [8].

The NBS isoperibol bomb calorimeters consist essentially of a calorimeter vessel which contains a measured amount of water. Immersed in the calorimeter water is a constant volume bomb in which combustible material are burned completely with the aid of high purity oxygen under high pressure. Also in the water are a stirring device for maintaining a uniform and meaningful calorimeter temperature, and a thermometer for measuring the calorimeter temperature. In order to control the heat transfer between the calorimeter and the environment, the calorimeter vessel is enclosed in a submarine vessel which is immersed in a constant temperature water jacket. The calorimeter vessel is separated from the submarine vessel, and hence the thermal environment of the water jacket, by a 1 cm air space.

A measurement of the heat of combustion of a given material is carried out by comparing the corrected temperature rise of the calorimeter water in an experiment in which a known amount of energy was supplied to it, with the corrected temperature rise of the calorimeter water produced in another experiment by combustion of a weighed sample of the material of interest. The temperature rise in both experiments should be, as nearly as possible, over the same temperature range from the standard initi temperature to the standard final temperature. This comparison method eliminates some of the systematic errors associated with calorimetric measurements and requires that the procedures be, as nearly as possible, identical in the two experiments which are compared. Calibration experiments are used to determine the amount of energy necessary to produce a specific corrected temperature rise. The mean of the calibration experiments is called the energy equivalent of the calorimeter. The product of the energy equivalent and the corrected temperature rise of the calorimeter in an experiment, in which a material of unknown heat of combustion is burned, gives the energy liberated in the combustion reaction and includes any side reactions which may occur.

2. Test Procedures

The procedures outlined here are applicable to both calibration experiments with benzoic acid, NBS SRM 39i, and RDF combustion experiments. Modifications necessary for either type of sample will be specified where applicable.

3. Sample Preparation

Benzoic acid (NBS SRM 39i), should be used to calibrate the bomb calorimeter. The conditions specified on the NBS certificate should be followed as closely as possible. The nature of the benzoic acid, SRM39i, is such that it can be used directly from the bottle, with no further processing. A sample size should be chosen to produce the desired temperature rise of the calorimeter. The NBS small (2.5 gram capacity) bomb calorimeter is selected to operate from 25°C to 28°C and requires approximately 1.63 grams of benzoic acid. The NBS large (25 gram capacity) bomb calorimeter is selected to operate from 25°C to 30°C and requires approximately 16.2 grams of benzoic acid. Benzoic acid was obtained commercially and its energy of combustion measured for use in calibration of the large calorimeter. The benzoic acid combustion samples are pressed into pellets and weighed in a pre-weighed combustion crucible. Platinium crucibles are used in the small calorimeter and weighings are carried out to the nearest microgram (0.000001 g). Quartz crucibles are used in the large calorimeter and weighings are carried out to the nearest ten micrograms (0.00001 g).

RDF samples for use in the small calorimeter should be material which has been milled to pass a 0.5 mm screen and then thoroughly blended. RDF samples for the large calorimeter have been from 19mm (3/4 in) to 0.5mm particle size. The choice of RDF samples from large particle size material must be done carefully and without bias. A sufficient amount of RDF material must be chosen to produce a three degree temperature rise in the small calorimeter or a five degree temperature rise in the large calorimeter. Samples are weighed in pre-weighed quartz crucibles using the appropriate balances described earlier. The weighed crucible and sample are then placed in the bomb sample holder.

4. Bomb Preparation

A measured length of firing wire is connected to the ignition electrodes of the bomb. A 3cm and 7cm fuse is used in the small and large bombs, respectively. A single loop is drawn in the center of the fuse and placed in contact with the combustion sample which had been placed in the sample holder. Platinum fuse wire is used without weighing because it does not undergo combustion. When iron fuse wire is used, its weight must be determined because a thermochemical correction for the combustion of the iron fuse wire must be applied for the quantity of iron wire burned.

A specific amount of water is placed into the bomb: 1.0 ml in the small bomb and 10.0 ml in the large bomb. The water is necessary to maintain an atmosphere that is saturated with water vapors such that any water produced in the combustion reaction will go to the liquid state so that there is no uncertainty associated with the calorimetric measurement due to the vaporization of water.

The bomb is assembled and charged with ultra high purity oxygen. The small bomb is filled to 3 MPa (20 atm) and the large bomb is filled to 4 MPa (40 atm). In the calibration experiments, the oxygen pressure is released slowly through the exit valve and then refilled with pure oxygen. This step is carried out to reduce the nitrogen impurity which is present in the one atmosphere of air initially present in the bomb. This purging step is omitted with RDF combustion experiments to ensure that there will be sufficient nitrogen for complete oxidation of the sulfur in the RDF [9].

5. Calorimeter Preparation

The calorimeter jacket temperature is adjusted to approximately 0.1 °C above the expected final temperature of the calorimeter (28°C and 30°C for the small and large calorimeters, respectively). The jacket temperature should be maintained at 0.001°C of the specified jacket temperature.

The calorimeter vessel is filled with distilled water and adjusted so that the water temperature is a few tenths of a degree below the desired initial temperature (25°C) in the experiment. The calorimeter vessel and water are placed on a balance and filled with distilled water to a constant mass for both calibration and combustion experiments. In the small calorimeter, the calorimeter vessel is filled with water to give a final weight of 3765 grams. It is then suspended and placed into a submergible calorimeter container. The bomb is placed into the calorimeter water and connections are made on the calorimeter cover for the fuse circuit and the calorimeter heater. The cover for the submergible calorimeter container is attached and the calorimeter stirrer is engaged. With the submarine sealed and all connections made, the calorimeter and submarine vessel are lowered into the constant temperature jacket bath.

The procedure is slightly different for the large calorimeter since the large calorimeter vessel contains approximately 21 liters of water; hence, it is more practical to determine the quantity of water in it volumetrically. The calorimeter vessel is filled with approximately the proper quantity of water while it is inside of the submarine vessel. The bomb is placed in the calorimeter and the water temperature is adjusted to 24.5°C. The fuse and heater leads are connected and the calorimeter vessel cover is assembled. The calorimeter cover contains a short length of a calibrated pipette. Water is added through the thermometer opening and water will rise in the pipette by capillary action to a specific level. The cover for the submarine vessel is attached and the complete system submerged into the controlled water bath.

The cover of the submarine vessel contains tubes, through which the thermometer and electrical connections are made to the calorimeter. The calorimeter stirrer is started to obtain uniform temperature drift readings between the calorimeter and jacket. Using the calorimeter heater, the desired starting temperature is reached, after which the calorimeter is ready for initial temperature measurements. The NBS fuse circuit is connected to a 32,000 microfarad capacitor which is charged to 23 volts and upon discharge ignites the fuse wire over the sample.

6. Temperature Measurements

The temperature is measured every 100 seconds with a quartz thermometer, to determine the variation of the calorimeter temperature with time. The quartz thermometer generates a frequency that rises linearly with increasing temperature to a high degree. A frequency counter is used over 100 second intervals and displays the calorimeter temperature. The display of the counter gives the average temperature over the 100 second counting intervals, providing an integrated temperature for a given calorimetric experiment. Both temperature measurement and data-logging are automatic. A least square fit of these points is performed using an exponential equation to produce the corrected temperature rise by correcting the observed temperature rise for the effects of the heat of stirring and thermal leakage.

7. Calorimeter Corrections

Corrections for side reactions producing nitric and sulfuric acids from the nitrogen and sulfur impurities in the RDF must be determined. The pressure is released from the bomb and it is opened. The crucible and residue are removed to determine the amount of non-combustibles (bomb ash) in the sample. The bomb interior is carefully washed and the washings saved for analysis of nitric and sufuric acid. Aliquots of the washings are used for these determinations. The total acid is determined by titration with a standard alkali. The sulfur content is determined gravimetrically by the precipitation of barium sulfate. A 57.7 joule correction is applied for each milliequivalent of acid and a 5860 joule correction for each gram of sulfur obtained from a calorimetric experiment.

A correction is applied for the amount of energy that had to be supplied to the calorimeter in order to ignite the sample. Since the sample is ignited by the discharge of a 32000 μf capacitor which has been charged to 23 volts, this correction is calculated from the difference between initial capacitor energy and the final capacitor energy; the energy of a capacitor is $1/2\ CV^2$. The measuring circuit accounts for 33 percent of the energy discharged.

If iron fuse wire is used, the unburned pieces must be weighed and subtracted from the initial weight of the iron fuse. 7100 joules per gram of iron burned is applied for the iron fuse correction.

8. Calculation of the Calorific Value

The total energy involved in the bomb process is calculated from the product of the actual energy equivalent (mean of the calibration experiment corrected for the heat capacities of the bomb contents) and the corrected temperature rise. The energy corrections described in the last section are subtracted from the total energy absorbed by the calorimeter to obtain the energy released by the combustion of RDF. The energy from the RDF combustion is then divided by the mass of the RDF sample to determine the calorific value (as-determined higher heating value, HHV-AD).

Appendix D is a conversion table for the calculation of data to different bases, i.e., as-determined, as-received, dry basis, and dry, ash-free basis.

B. Calibration of the Calorimeters

1. NBS 2.5 Gram Capacity Bomb Calorimeter

The NBS 2.5 gram capacity calorimeter has an energy equivalent of 14543 $J\cdot K^{-1}$. A record of the energy equivalent is maintained. We have found that the energy equivalent of this calorimeter system has not changed significantly over the last three years. Any changes to the calorimeter are recorded and an appropriate correction based upon the mass and the heat capacity of the material is applied. These calculated corrections usually amount to less than 0.1 $J\cdot K^{-1}$ and are usually beyond the measurement capabilities of the instrument. However, the energy equivalent is periodically measured to check for any unforeseen changes which might occur.

During fiscal year 1979, 23 calibration determinations were performed. The average value of these 23 determinations was 14543.64 ± 2.28 $J\cdot K^{-1}$. The estimated uncertainty is twice the standard deviation of the mean (2 sdm). The value which is presently being utilized for this calorimeter is 14543.45 $J\cdot K^{-1}$ and is based on the most recent set of 6 energy equivalent (calibration) measurements. Table 1 contains the results of the energy equivalent measurements during the 1979 fiscal year. The last six energy equivalent measurements (marked with an asterisk) are the basis of the value presently being used.

Table 2 contains a more detailed presentation of measurements performed in the last six calibration experiments, numbers 1238-1244. The headings, in the order in which they appear in the tables, are described as follows:

Expt. No., the number of the experiment, which can easily be traced back to our records.

$\Delta Uc°\ (28°)/J\cdot g^{-1}$, the energy evolved by the combustion of benzoic acid at the selected final temperature in $J\cdot g^{-1}$.

m-BA/g (vac), the mass of the benzoic acid sample, in g, reduced to mass in vacuum.

q-BA/J, the energy evolved by the combustion benzoic acid, in J.

q-ign/J, the electrical energy added to the system to ignite the sample, in J.

q-HNO3/J, the energy evolved by the formation of nitric acid in the combustion process, in J.

q-WC/J, the Washburn Correction [6,7] applied to correct the combustion data from bomb conditions to conditions in which the reactants and products are in their pure standard states at one atmosphere pressure, in J.

q-corr to t_f/J, a correction applied for the deviation of the actual final temperature from the selected standard final temperature (usually 28°C), in J.

Q-total/J, the total energy added to the calorimeter, in J.

ΔT-corr/K, the observed temperature rise of the calorimeter corrected for stirring energy and thermal leakage, in K.

E-cal/J·K^{-1}, the energy equivalent of the actual calorimeter system at the final temperature, in J·K^{-1}.

Ei-cont/J·K^{-1}, the heat capacity of the initial bomb contents, including the sample, crucible, water, and oxygen, in J·K^{-1}.

E,si-empty/J·K^{-1}, the energy equivalent of the empty calorimeter at 28°C, in J·K^{-1}.

E,si-mean/J·K^{-1}, the mean value of the measured energy equivalent, in J·K^{-1}.

Std. Dev./J·K^{-1}, the standard deviation of a measurement, in J·K^{-1} (and the percent standard deviation).

Std. Dev. Mean/J·K^{-1}, the standard deviation of the mean, in J·K^{-1} (and the percent standard deviation of the mean).

2. The NBS 25 Gram Capacity Bomb Calorimeter

The 16 gram sample size of benzoic acid that is required to calibrate the large calorimeter makes the use of the NBS SRM 39i (benzoic acid-calorimetric standard) impractical. Therefore, benzoic acid was obtained from a commercial source to calibrate this large calorimeter. Standard benzoic acid (NBS SRM 39i) has an energy of combustion under standard bomb conditions of 26,434 ± 3 $J \cdot g^{-1}$. This reduced to 26,410.68 $J \cdot g^{-1}$ under actual bomb conditions and is used to standardize the commercially obtained benzoic acid. The "commercial" benzoic acid was found to have an energy of combustion which reduces to 26,411.36 $J \cdot g^{-1}$ at actual bomb conditions. Table 3 presents the results of measurement of the energy of combustion of the "commercial" benzoic acid. The headings are explained under the calibration experiments of the 2.5 gram capacity bomb calorimeter.

Table 4 presents the results of a set of calibration experiments for the large calorimeter. The two additions to the headings described previously are as follows:

q-Fe/J, energy evolved by the combustion of the iron fuse wire, in J.

q-carbon/J, the correction applied for unburned carbon in the bomb, in J.

The calibration experiments in the large calorimeter resulted in an energy equivalent of 86,043.43 ± 9.13 $J \cdot K^{-1}$. This value is about six times the corresponding value for the conventional size calorimeter; however, the precision of calibration is essentially the same. Compare standard deviations of the mean in Tables 1 and 4, which are about 0.01 percent.

Subsequent tables which present calorimetric data on RDF samples contain addition correction headings; they are identified as follows:

Q-RDF/J, total energy delivered to the calorimeter after corrections for ignition energy, fuse energy, formation of nitric and sulfuric acids, and the like, in J.

m-RDF/g, mass of the RDF sample, in g.

HHV-AD/$MJ \cdot kg^{-1}$, higher heating value on as-determined basis, in $MJ \cdot Kg^{-1}$ (or $Btu \cdot lb^{-1}$).

C. Calorimetric Measurements on RDF

Laboratory samples of RDF undergo considerable processing so that a reasonably homogeneous sample can be prepared for calorimetric measurement. Before milling, the samples must be air-dried to remove most of the moisture present in them. The samples are then milled to 2mm or 0.5 mm powder and pressed into pellets weighing about 2.5 grams, for calorimetric measurement. The raw calorific values which are calculated from the bomb calorimetric experiments are referred to as higher heating values on an as-determined basis, HHV-AD.

The HHV-AD values are not consistent with the heating value of the fuel that is received and which will be burned in power plants or boilers. Combustion engineers are primarily interested in the higher heating value on an as-received basis, HHV-AR (also called HHV1). The sample mass which is used in the calculation of HHV-AD is corrected for its residual moisture content, and the higher heating value-moisture free, HHV-MF (also called HHV2), is obtained. HHV1 is obtained from HHV2 by adding the total moisture to the dry sample mass of HHV2. Also of interest is the higher heating value on a moisture-ash-free basis, HHV-MAF (also called HHV3). HHV3 is obtained from HHV2 by correcting the dry sample mass of HHV2 for its ash content. HHV3 is important because these data are used for comparison of heating values determined in different laboratories, under different conditions, on the same RDF sample. The precision obtainable on HHV3 is indicative of the experimental control which prevails during the calorimetric measurements. The ash content of analysis samples of RDF vary, sometimes to a significant degree. Since these ash contents are used to correct HHV2 to obtain HHV3, it has been proposed [1] that precisions can be enhanced if the calorimetric sample masses are corrected for ash content based upon measurement of the bomb residue (i.e. the actual ash content of the calorimetric sample). Use of the ash content determined from the bomb residue weighings eliminates the assumption that an equivalent ash content determined from the ASTM furnace ash protocol is applicable to combustion bomb calorimetric data. This assumption is not completely valid. In the furnace, combustion takes place in air at atmospheric pressure at about 575 or 725°C; in the bomb, combustion takes place in pure oxygen under a pressure of 30 atmospheres at about 1500 to 2000°C. The composition and character of the two ash residues (i.e., furnace ash vs. bomb ash) are distinctly different.

In summary, HHV3 can be calculated from either the bomb ash, yielding HHV3-B, or from the furnace ash, yielding HHV3-F. Higher heating values, therefore, can be calculated on four different bases from HHV-AD, namely: HHV1, HHV2, HHV3-B, and HHV3-F.

Higher heating value, residual moisture, and ash experiments are performed concurrently. Since the residual moisture content and the ash content are used to correct the mass of the calorimetric sample, the three samples must be prepared under the same conditions. Therefore, a routine laboratory measurement of the higher heating value requires the preparation of three samples. As part of the calorimetric measurements, the bomb residue and sulfur content are also measured.

The RDF calorimetric measurements were previously placed into five classes, namely: (1) measurements carried out in conjunction with ASTM E-38.01 round robin testing; (2) measurements carried out on an Americology RDF sample, from which sub-samples were extracted in a selective and non-selective manner; (3) measurements required to compare the characteristics of the small and large bomb calorimeters; (4) large bomb calorimeter measurements of the combustible fractions of New Castle County, Delaware MSW which had been processed at the U.S. Bureau of Mines, College Park, MD facility; and (5) measurements on some representative RDF's of special interest. Discussion of RDF measurements is according to each particular class.

A set of results (usually three tables) is provided for each distinct RDF sample studied. The first table of a set gives detailed calorimetric data for each experiment which was carried out on a particular RDF sample. Since RDF's are fuels which have low sulfur contents, additional corrections will appear in the tables, as follows:

$q\text{-}H_2SO_4/J$, the energy involved by the formation of sulfuric acid in the bomb process, in J.

The second table of a set contains the analytical data necessary to calculate higher heating values from the as-determined base to the four different bases.

The third table of a set will present the calculated higher heating values for the four different bases. Hence, for each RDF sample studied, three tables are presented: (a) calorimetric data, (b) analytical data, and (c) calculated higher heating values for three different bases, HHV2, HHV3-F, and HHV3-B.

1. ASTM Subcommittee E-38.01 Round Robin Testing on RDF-3

As mentioned earlier, two zeroth rounds of round robin testing were carried out over the period of 1 October 1977 to 31 January 1978, in order to allow laboratory analysts an opportunity to become more acclimated with the new and/or modified testing protocols. The provisional protocols issued by ASTM Subcommittee E-38.01 on Energy required that duplicate measurements be performed on consecutive days for all of the following chemical tests: residual moisture, higher heating value, ash content, volatile matter, elemental analysis, and ash fusion temperatures. Our interest focused primarily on the higher heating value, moisture, and ash content determinations. A complete technical review and evaluation of data have been made at NBS on the results of the ASTM E-38.01 program to develop test procedures for the characterization of RDF-3. The program was carried out by a large number of testing laboratories over the period September 1977 through March 1980. This technical review and the evaluations can be found in Appendix A of this report.

The milled RDF sample (0.5 mm particle size) was coned and quartered; each quarter was treated as a sub-sample of the total and had higher heating value, residual moisture, and ash content determinations made on it. Therefore, the precision associated with a set of measurements is a function of both our sampling and measurement techniques.

a. First Round Robin Sample

Gross calorific values determined on two sets of samples for the first round of testing of RDF-3 are shown in Table 5. One set of samples was equilibrated in a constant humidity atmosphere at 45 percent relative humidity, while the second set was dried at 105°C and exposed to the moisture in the laboratory for several days. Both samples yielded different residual moisture and bomb ash values, as shown in tables 6 and 7. Furnace ash and sulfur content determinations were carried out on the samples equilibrated at 45 percent relative humidity, and were assumed to be same for samples which were dried and subsequently exposed to laboratory moisture conditions. These observations strongly suggest that the residual moisture content of an RDF sample is related to the relative humdity present in the testing laboratory. The RDF sample which had been previously dried and then exposed to laboratory moisture conditions had a lower residual moisture content, a higher bomb ash content, and a lower calorific value. It is possible that combustible substances may have vaporized during the drying at 105°C, thus enriching the sample in inerts (ash) and perhaps also decreasing its volatile character. Tables 8 and 9 provide calorific values for the first round robin sample, calculated to different bases. The data are, in general, in good agreement. Comparison of data on HHV2 shows the largest dispersion, but this is only about 2 percent.

b. Second Round Robin Sample

Calorific values are provided for the second round of testing on RDF-3 samples in table 10 on both an interlaboratory comparison sample and an RDF sample processed down to 0.5 mm particles at NBS in the usual manner. Both samples were equilibrated in a constant humidity atmosphere at 45 percent relative humidity. Although both the interlaboratory comparison sample and the regularly processed sample were extracted from the same gross laboratory sample, the higher heating values on an as-determined basis differ by about 3.5 percent. The overall hetergeneous nature of refuse is probably responsible for this.

The results of determinations of residual moisture, ash content, and sulfur content are shown for the interlaboratory comparison sample and the RDF sample processed down to 0.5 mm in tables 11 and 12, respectively. Note that the residual moisture determinations carried out on pellets which were equilibrated in a constant 45 percent relative humidity environment are about the same value, 6.1 wt. percent. However, residual moisture determinations performed on loose powder which was exposed to laboratory moisture conditions are more variable, 3.3-3.5 wt. percent. The differences between the furnace ash determined on loose and pelleted samples do not appear significant for either interlaboratory comparison or regularly processed samples. However, when differences between corresponding bomb ash values are examined, they are significant (compare bomb ash values of 18.82 and 13.43 wt. percent in tables 11 and 12, respectively).

Calorific values calculated to different bases are reported in tables 13 and 14 for the interlaboratory comparison sample and the RDF sample processed to 0.5 mm at NBS, respectively. Agreement is good between higher heating values on a moisture-ash-free basis (HHV3), in general, but systematic differences do exist between values based upon furnace ash and bomb ash.

c. Third Round Robin Sample

In table 15, one finds data on calorific value determinations for the interlaboratory comparison sample and the RDF sample which was processed to 0.5 mm powder at NBS for the third round of testing of RDF-3. The as-determined calorific values (HHV-AD) for the interlaboratory comparison sample are about 2 percent lower than those for the RDF which underwent processing at NBS. This agreement is good. Data for residual moisture, ash content, and sulfur content are given in table 16 for the interlaboratory comparison sample and in table 17 for the regularly processed round robin sample. Pelleted RDF samples were not kept in a constant humidity atmosphere, but were exposed to ambient laboratory conditions. A comparison of analytical measurements on the interlaboratory RDF sample and the in-house processed sample (compare tables 16 and 17) are in good agreement. Calorific values which have been calculated to different bases for the interlaboratory comparison sample and the in-house processed sample are provided in tables 18 and 19, respectively. The calorific values for the interlaboratory RDF sample are all lower when compared with the sample which was processed at NBS. The differences, however, are not serious; the range is between 2 and 3 percent.

d. Bituminous Coal Sample

In order to provide some reassurance to the RDF-3 round robin testing program that the participating laboratories could carry out the determinations for higher heating value, residual moisture, ash, and sulfur content in a satisfactory manner, an NBS Standard Reference Material (SRM 1632a) sample of a bituminous coal was distributed for testing. The round robin on the SRM bituminous coal sample was an important round because it could provide the comparison in the levels of precision, i.e., repeatability and reproducibility of test results, between the analysis of coal and RDF-3. This comparison would not only furnish some quantitative estimate of what kind of variability one might expect in the results of tests carried out on a heterogeneous material like RDF-3, but also would provide an indication of what the current state-of-the-art of the ASTM protocol was in this testing program.

The results of duplicate calorimetric and analytical measurements which were performed on the SRM bituminous coal sample are found in tables 20 and 21. The results are in good agreement. Table 22 gives the calculation of calorific values to different bases. Here also, agreement appears good.

A statistical analysis of both the NBS data and those of the other participating laboratories which have been part of the ASTM testing program on RDF-3, can be found in Appendix A. The repeatability interval (which is an estimate of within laboratory measurement agreement) and the reproducibility interval (which is an estimate of between laboratory measurement agreement) have been calculated for higher heating value, residual moisture and ash content determinations. Repeatability intervals for HHV2 (dry basis), residual moisture, and ash content are 250 Btu/lb, 0.75 wt. percent, and 2.5 wt. percent, respectively. Compare these data above with equivalent values calculated for ASTM protocols for coal and coke: HHV2 (dry basis) is 50 Btu/lb, residual moisture is 0.3 wt. percent, and ash content amounts to 0.5 wt. percent.

Reproducibility intervals for HHV2 (dry basis), residual moisture, and ash content are 750 Btu/lb, 3.00 wt. percent, and 0.5 wt. percent, respectively. ASTM coal and coke protocols report comparable reproducibility intervals of 100 Btu/lb, 0.5 wt. percent, and 1.0 wt. percent, respectively.

The heterogeneity of RDF at particle sizes of both 25 mm (1 inch) and 0.5 mm limits the ability to which a representative sample can be extracted. Before improvements in the levels of precision, i.e., repeatability and reproducibility can be expected, further development in sampling and/or separation techniques for 25 mm as well as 0.5 mm particle size samples should be pursued.

2. Americology RDF Sample

A sample of RDF-3, which was prepared and collected by Americology (Division of the American Can Company) for Wisconsin Electric Power Company, was received at NBS in May 1977. The Americology RDF-3 was sampled in two ways to determine the effect of different sampling techniques on a 20 kilogram gross sample. The first method was to take a grab sample of approximately one kilogram directly from the 2 inch particle size material. The one kilogram sample was processed to 0.5mm particle size in two steps using a Wiley, Model 4, laboratory mill which is equipped with 2mm and 0.5mm screens. The 0.5mm material was blended thoroughly in a vee blender and appropriate amounts of analysis samples were drawn. This method should give useful information on the effects of selective sampling.

In the other method, the remainder of the Americology RDF-3 was ground to 6 mm (1/4 inch) particle size in the 5 hp hammer mill. It was coned and quartered to select a one kilogram laboratory sample. The one kilogram sample was processed to 0.5mm and blended in a similar manner to that of the grab sample. This material should be more representative of the 20 kilogram gross sample and also appeared more homogeneous than the grab sample. The only difference in the sample treatment of the two samples is in their initial selection; the first sample was selected directly from 2 inch particle size material in a selective or grab fashion. The second sample was obtained by coning and quartering the 1/4 inch particle size material and is more representative of the sample as a whole.

Tables 23, 24, and 25 contain the results of measurements on the Americology grab sample and tables 26, 27, and 28 contain comparable measurements on the Americology representative sample. Comparison of the average calorific values calculated to different bases in tables 25 and 28 shows that the differences between the mean values for HHV-AD, HHV2, HHV3-F, and HHV3-B are 201, 364, 133, and 457 Btu/lb, respectively. These differences are significant and reflect the heterogeneity of refuse and RDF over which only minimal control is possible using present processing methods.

The pelleted samples of the Americology grab sample were equilibrated in a constant humidity atmosphere at 30 percent relative humidity, while the pelleted samples of the Americology representative sample were equilibrated at 45 percent relative humidity.

These equilibrations are reflected in the residual moisture determinations reported for these samples in tables 24 and 27; compare mean values of 3.01 wt. percent for the grab sample in table 24 with 30 percent relative humidity equilibration and 5.19 wt. percent for the representative sample in table 27 with 45 percent relative humidity equilibration. Systematic differences in bomb ash and furnace ash mean values are apparent from an examination of tables 24 and 27. The differences between samples are due to the heterogeneity of RDF, while the differences within a sample are due to the difference in ashing conditions in the bomb and furnace.

3. Large and Small Calorimeter Comparison Measurements

Calorific values were measured in both the large 25.0 gram capacity bomb calorimeter and the small 2.5 gram capacity bomb calorimeter to evaluate the performance of the large bomb calorimeter system. Three different RDF's which had been characterized previously at NBS were chosen for this study. These RDF's were: (1) Teledyne National RDF, (2) ECO-FUEL-II RDF, and (3) Americology RDF. Although the large bomb calorimeter is capable of making measurements on RDF's which have received less size reduction and could be as large as 1 inch in particle size, 0.5mm RDF samples were used in order that two equivalent sets of samples could be prepared for measurements in both the large and small bomb calorimeters. Results obtained in the large calorimeter could then be compared to the results obtained in our small calorimeter which had been in service for many years at NBS and had a history of consistently providing calorimetric measurements of high precision.

a. Teledyne National RDF

Tables 29, 31, and 32 present results obtained in the small bomb calorimeter for the Teledyne National RDF sample. Tables 30, 31, and 32 give the Teledyne National RDF results obtained in the large bomb calorimeter. In table 32, the mean values in $MJ \cdot kg^{-1}$ ($Btu \cdot lb^{-1}$) for HHV-AD, HHV2, HHV3-F and HHV3-B are: 19.35 (8318), 20.38 (8762), 24.55 (10553), and 24.03 (10331) for the small bomb calorimeter. Also in table 32, the corresponding values from the large bomb calorimeter in $MJ \cdot kg^{-1}$ ($Btu \cdot lb^{-1}$) are: 19.50 (8385), 20.54 (8832), 24.42 (10499), and 24.31 (10451). Theoretically, the two sets of samples are identical and should reproduce identical results; however, in practice differences do occur because of random errors, systematic errors, and errors introduced due to the heterogeneity of RDF.

Since the calorific values which were calculated to different bases for the Teledyne National RDF differed by approximately one percent in comparison with values measured in the small and large bomb calorimeters, we sought an explanation. We felt that the stainless steel crucibles used in the large calorimeter were being oxidized in the bomb combustion process, and we knew from many years of experience that the platinium crucibles used in the small calorimeter were not being oxidized. This was confirmed when the crucibles were weighed before and after calorimetric experiments, revealing significant mass changes in the stainless steel crucibles and insignificant changes in the platinium crucibles. Since the actual bomb ash residues contained large amounts of silica (SiO_2), we decided to use quartz crucibles in both the large and small calorimeters in all subsequent RDF experiments. Quartz tubing of the required diameter was used to prepare these crucibles for each bomb calorimeter. The quartz crucibles easily withstand the thermal shock experienced in the combustion of RDF and other samples, and after the amount of ash residue is measured, they can be easily cleaned by heating the crucible and ash residue, and then immersing the hot crucible and residue into cold water. The rapid contraction of the hot residue frees it from the crucible bottom and walls, and it can then be cleaned out easily. The quartz crucibles can thus be used several times.

b. ECO FUEL-II RDF

Tables 33, 35, and 36 and tables 34, 35, and 36 present the results measured in the small and large calorimeters, respectively, for ECO FUEL-II RDF. The mean values in $\mathrm{MJ \cdot kg^{-1}}$ ($\mathrm{Btu \cdot lb^{-1}}$) for HHV-AD, HHV2, HHV3-F, and HHV3-B which can be found in table 36 are 18.78 (8072), 19.09 (8206), 21.98 (9451), and 21.64 (9306) for the small calorimeter, and 18.77 (9071), 19.08 (8205), 21.98 (9449), and 21.64 (9302) for the large calorimeter. On comparing the two sets of mean values, the differences are less than the standard deviation obtained for one set of measurements. However, ECO FUEL-II RDF has some particles as small as 250 m (60 mesh) and is probably the most homogeneous RDF which is available commercially. These calorimetric results clearly indicate that the new large 25.0 gram capacity bomb calorimeter gives the same results as the small 2.5 gram capacity bomb calorimeter, and because of its size, could possibly even provide more precise results.

c. Americology RDF

Americology RDF, although not as homogeneous as ECO FUEL-II RDF, should theoretically have identical results in the two calorimeters. Tables 37, 39, and 40, and tables 38, 39, and 40 contain the experimental results measured in the small and large calorimeter, respectively. The mean values in $\mathrm{MJ \cdot kg^{-1}}$ ($\mathrm{Btu \cdot lb^{-1}}$) of HHV-AD, HHV2, HHV3-F and HHV3-B which can be found in table 40 are 15.67 (6736), 16.63 (7150), 22.96 (9871), and 22.69 (9756) for the small calorimeter, and 15.70 (6750), 16.66 (7164), 23.01 (9892) and 22.67 (9746) for the large calorimeter. Again, on comparing the two sets of mean values, the differences are less than the standard deviation obtainable for one set of measurements. We feel that the impact of using quartz crucibles in both calorimeters is also present. Clearly, the two calorimeters have identical results on the same RDF which has received the same amount of processing (i.e. particle size reduction). Also, the large 25.0 gram capacity calorimeter has been proven to be very acceptable for measuring calorific values of heterogeneous materials as precisely as the conventional small 2.5 gram capacity bomb calorimeter.

4. Bureau of Mines Processing of New Castle County, Delaware MSW

This set of results are for RDF samples obtained in various stages of processing through the Bureau of Mines Resource Recovery Pilot Plant in College Park, MD. Some of these samples had received less processing (i.e. contain larger particle sizes); however, all had calorimetric measurements performed on them in the large calorimeter.

To evaluate the applicability of the NBS large gram capacity oxygen bomb calorimeter, 21 kilograms (46.3 lbs) of RDF was obtained from New Castle County, Delaware, Municipal Solid Waste (MSW), which had been processed through the U.S. Bureau of Mines Resource Recovery pilot plant located in College Park, MD [10]. The pilot plant consists of shredders, magnetic separators, trommels, air classifiers, cyclones, and mineral jigs. Samples from seven collection points in the pilot plant had been collected and included in this study. The overall flow-diagram for this pilot plant is shown in figure 1. The flow-paths through which each of the seven samples had passed are provided in figures 2 to 8, respectively. The seven fractions (or bags) and their as-received masses were:

1.	Cyclone No. 1, Secondary shred, minus 2 inches	3268.3 g
2.	Cyclone No. 2, Secondary shred, minus 2 inches	2672.5 g
3.	Cyclone No. 3, Secondary shred, as-collected	3540.3 g
4.	Cyclone No. 1, Trommel Undersize + 14 mesh	2287.5 g
5.	Cyclone No. 2, Trommel Undersize + 14 mesh	2361.2 g
6.	Heavy Organic Combustibles	3664.8 g
7.	Organic Wastes, Jig Overflow	3203.1 g
	TOTAL MASS	20996.7 g

Each of the seven separate laboratory samples received the following treatment:

1. air-drying in a convection oven @ 37±3°C
2. milling to reduce particle size,
    a. samples 1-5 were mostly paper and plastic, and were milled in the Wiley Mill to 2mm particle size;
    b. samples 6 and 7 were large pieces of wood, rubber and plastic and were milled in the hammer mill to 6mm (1/4 inch)
3. splitting to obtain a laboratory sample of workable size by successive coning and quartering;
4. blending of the laboratory sample in the vee-blenders to obtain a homogeneous sample;
5. selecting analysis specimens of approximately 2.5 or 25 grams each:
    a. 3 specimens for calorific value measurements
    b. 2 specimens for residual moisture measurements
    c. 2 specimens for furnace ash measurements
6. analysis specimens were pelletized with the exception of those from samples 6 and 7, which did not pelletize well.

The format throughout this report of presenting three tables for each RDF sample is also followed for these samples. In addition, the air-drying moisture data are included with the analytical results since these samples had high moistures, characteristic of field samples of RDF. Total moistures (T.M.) used to calculate HHV1 (as received) were calculated using the residual moisture (R.M.) fraction, and the air day loss (A.D.L.) fraction, according to the following equation:

T.M. = R.M. (1 - A.D.L.) + A.D.L.; see also Appendix E.

Tables 41, 45, and 52 present the results of measurements on cyclone 1 secondary shred (-2 inch) RDF. The mean values of HHV1, HHV2, HHV3-F and HHV3-B in $MJ \cdot kg^{-1}$ ($Btu \cdot lb^{-1}$) were 16.72 (7189), 19.32 (8305), 22.08 (9493), and 21.80 (9374), respectively. The calorific values were measured with better than 0.4 percent precision.

Tables 42, 47, and 53 present the results of measurements on cyclone 2-secondary shred (-2 inch) RDF. The mean values in $MJ \cdot kg^{-1}$ ($Btu \cdot lb^{-1}$) for HHV1, HHV2, HHV3-F and HHV3-B were 17.32 (7444), 20.00 (8598), 21.81 (9379), and 21.59 (9282), respectively. The precision observed for these measurements was 0.2 percent.

Tables 43, 49, and 54 present the calorimetric and analytical results of cyclone No. 3 as-collected RDF. The mean values of HHV1, HHV2, HHV3-F, and HHV3-B in $MJ \cdot kg^{-1}$ ($Btu \cdot lb^{-1}$) were 16.29 (7003), 20.35 (8751), 22.38 (9621), and 22.47 (9662); they were measured with a precision of approximately 0.7 percent.

The three RDF's from the cyclones were composed mostly of light paper and plastics with ash contents from 8 to 13 wt. percent and had HHV3 values of approximately 22 $MJ \cdot kg^{-1}$ (9400 $Btu \cdot lb^{-1}$). The RDF from cyclones No.s 1 and 2 which did not pass through the trommel but was retained on a 1.4mm (14 mesh) screen, consisted of heavier organics and contained considerably more ash (22-35 wt. percent).

These ashes are believed to be fairly representative of the recovery process utilized. The light air-classified fraction from the first air classifier contains the greatest percentage of ash since the only processing involved was primary shredding, separation into large and small particle sizes by the trommel, and secondary shredding of the large particles which passed through the trommel. The large particles which passed through the trommel were mostly composed of light paper and plastics. They showed a characteristic moisture of approximately 14 wt. percent and a moderate ash content of approximately 8 wt. percent. However, the small particle heavies from cyclone No. 1 which were separated by the trommel sieve openings were concentrated with the majority of the noncombustible materials, such as fine metal particles, small pieces of glass, and inorganic particles. The ash content data contain values having the largest magnitude approximately 34 wt. percent (see table 46) of all samples processed at College Park, MD. However, the heavy fraction from the first air classifier was composed of metals, wet paper, and plastics, which could account for their failure to be air classified. This material was then subjected to a magnetic separation process to remove ferrous metals before a second air classification step took place. With most of the metals removed from the heavies of the first air classifier, the material passed through a second air classifier to cyclone No. 2 and through the same trommel sizing step that was used on cyclone No. 1 material. The large particles which passed through the trommel had moisture characteristics of light paper and plastics but show less evidence of ash (approximately 8.3 wt. percent in table 47), since the magnetic separation had removed most metallic particles which were attached to the large pieces of paper. However, the undersize material from the trommel was heavier because of moisture and not because of metallic content, and we measured an increase in moisture to 13 wt. percent, and a decrease in ash to 23 wt. percent (see table 48) over the material from cyclone No. 1. It appeared that the cyclone No. 1 material contained only the absorbed moisture on paper and plastic particles but unusually high ash values since no attempt (other than air classification) had been made to separate metallic particles. Meanwhile the cyclone No. 2 material was subjected to a magnetic separation step before air classification and the small particle heavies contain considerable amounts of moisture but less than the small heavies from cyclone No. 1.

Tables 41, 46, and 55 present the results of cyclone No. 1 trommel undersize but larger than 14 mesh RDF. The calculated mean values in $MJ \cdot kg^{-1}$ ($Btu \cdot lb^{-1}$) were 12.20 (5244), 14.19 (6100), 21.53 (9257), and 20.73 (8914).

Tables 42, 48, and 56 contain the results for cyclone No. 2 trommel undersize, but larger than 14 mesh RDF. The calculated mean values in $MJ \cdot kg^{-1}$ ($Btu \cdot lb^{-1}$) were 12.46 (5359), 16.34 (7026), 2123 (9127), and 21.20 (9116).

Even though the composition of these last two RDF's was different from the first three RDF's, the HHV3 was still approximately 21 $MJ \cdot kg^{-1}$ (9000 $Btu \cdot lb^{-1}$), which appears to be characteristic of RDF's in general. However, there was a decrease in the precision obtainable on these more heterogeneous samples. Except for HHV3-B, where the ash content was based on the bomb residue, the precision is between 1.0 and 1.8 wt. percent. However, the actual ash (based on the bomb residue) gave HHV3-B with a precision of 0.2 to 0.5 wt. percent, which was as expected since the selection of equivalent or representative samples was more difficult with high ash content samples.

The remaining two RDF fractions contain large pieces of wood, shoes, plastic, putrescibles and yard waste. The material had components too large for the Wiley laboratory mill, and therefore was an opportunity to test the large calorimeter on samples with minimal processing. These last two RDF fractions were processed through our 5hp hammermill equipped with a 6mm (1/4 inch) screen. Usually the combustion samples are pelletized to get controlled burning from the sample surface, however; the composition of these samples was such that they did not pelletize well and consequently were burned as loose material in the combustion crucible. The base material burned more rapidly because of the increased surface area and made it difficult to collect all of the bomb combustion residue. Therefore, HHV3-B will not be reported for these samples because quantitative bomb residues could not be determined.

Table 44 contains the experimental results for the heavy organic combustibles (high tension non-conductors) and organic wastes from the jig overflow RDF's. In tables 57 and 58, the mean values of HHV1, HHV2, and HHV3-F for the heavy organic combustibles RDF in $MJ \cdot kg^{-1}$ ($Btu \cdot lb^{-1}$) were 20.85 (8965), 21.92 (9422), and 24.61 (10579), while those for the jig overflow RDF were 19.57 (8416), 20.69 (8896), and 23.37 (10006), respectively. The precision of the heavy organic combustibles RDF measurements was 0.2 percent and that of the jig overflow RDF was 1.28 percent. The low precision of the jig overflow RDF could be due to an unexplained large systematic error in the first combustion experiment (Number 1046). Omission of experiment number 1046 gave mean values of HHV1, HHV2, and HHV3 in $MJ \cdot kg^{-1}$ ($Btu \cdot lb^{-1}$) of 19.72 (8478), 20.84 (8962), and 23.45 (10 080) for the jig overflow RDF. Also the precision had increased to 0.04 percent. Since the ash content was approximately 11 percent, selection of identical samples should not have been difficult and the experimental results should have been more repeatable.

The heavies from the second air classifier were subjected to another magnetic separation step and the remaining material was sent through the sizing trommel similar to that used for the material from cyclones No. 1 and 2. The small particle material that did not pass through the trommel was composed of mostly glass and small pieces of wood and plastic. This small-particle material was sent to a mineral jig for separation of the organic waste (small pieces of wood and plastic) from the recoverable glass particles. This material did not contain much absorbed moisture and only a moderate amount of ash, since it had had metal and inorganic (glass) materials removed. Therefore, we measured and calculated approximately 5 wt. percent moisture and 11 wt. percent ash.

The large-particle material that passed through the sizing trommel was secondary shredded and then air classified into two fractions. The heavies from the third air classifier were passed over an electrostatic separator to remove the aluminum. The remaining material was very similar to the organic waste from the jig overflow except that it was larger in particle size (i.e. bigger pieces of wood, plastics, etc.). The residual moisture and ash values were 5 and 11 wt. percent, respectively, values similar to the those from the jig overflow organic wastes. The light fraction from the air classifier was processed through cyclone No. 3 and then collected. This material contained only 9 percent ash since most of the metals and inorganic materials had been removed, but did contain the heavier paper and plastics that were not air classified in two earlier attempts. This material did show a larger moisture content of 20 wt. percent, similar to the fraction from cyclone No. 2.

The above discussion dealt primarily with the combustible output of New Castle County, Delaware MSW that was processed through the Bureau of Mines Resource Recovery facility located in College Park, Maryland. The process described is used to recover several recoverable fractions from municipal solid waste (MSW), namely combustible fraction, glass fraction, ferrous fraction and non-ferrous fraction. The combined results from the seven combustible fractions from the Bureau of Mines Resource Recovery facility provided some important information on the combustible portion of MSW.

A composite calorific value can be calculated by addition of the fraction of the heating value supplied by each component according to the following equation:

$$\mathrm{HHV}(i) = \sum_{j=1}^{7} \frac{m_j}{m_T} \mathrm{HHV}(i,j)$$

However, this assumed that the mass of each sample is selected in the same relative proportion as compared to the total mass collected at each point. The calculated composite HHV(i) in $\mathrm{MJ \cdot kg^{-1}}$ ($\mathrm{Btu \cdot lb^{-1}}$) would be:

HHV1 = 16.91 (7270)

HHV2 = 19.35 (8319)

HHV3-F = 22.56 (9701)

Measurements in the large bomb calorimeter were made on samples with particle size from 6mm (1/4 inch) to 2mm. The precisions observed were of the order of 1 percent or better. These measurements show that the large bomb calorimeter is applicable to RDF samples which have experienced less processing than those measured in the small (conventional-size) bomb calorimeter.

5. Representative or Unique RDF's

Several RDF's which had some especially interesting characteristics were investigated to evaluate the calorimetric procedures used in the small (2.5 gram capacity) calorimeter. In addition to the evaluation of our test procedures, the effect of some special properties on the calorific value of RDF was studied. The RDF samples used in the small calorimeter were processed to 0.5mm particle size. As usual, three tables are presented for each RDF studied: one on the calorimetric data, one on the analytical data, and one on the calorific values which have been calculated to different bases.

a. Teledyne National RDF

A Teledyne National RDF sample was obtained to evaluate the effect of the method used to determine the total moisture content of a given RDF. Two techniques have been tested. One was identical to that used for coal and coke samples and is referred to as the dual-step method, in which the RDF sample was air-dried to obtain a residual moisture content which was in equilibrium with ambient laboratory conditions. This was followed by a residual moisture determination on an analysis sample on the same day of the calorimetric determination. The air-dry loss was combined with the residual moisture to determine the total moisture of the as-received RDF.

The other method, which is referred to as the single-step method, determined the total moisture in one step at 107 ± 3°C. Tables 59, 60, and 61 and 62, 63, and 64 provide the experimental data for the Teledyne National RDF, which had undergone single-stage and dual-stage moisture determinations, respectively.

The NBS portion of the Teledyne National RDF was split into two equivalent samples, one for the single-step moisture part of the study and the other for the two-step moisture part of the study. The fraction of the sample which was used in the single step moisture experiments was placed into two drying pans and treated at 107 ± 3°C until dry. The measured values for total moisture were 21.79 and 21.65 percent, with a mean value of 21.72 percent. The two-step moisture portion was also placed into two drying pans, but was only heated to 37 ± 3°C until the samples were in equilibrium with ambient laboratory moisture conditions. The moisture loss at 37°C (air-dry-loss) was found to be 17.67 and 17.57 percent, with a mean value of 17.62 percent. The mean value of the air-dry-loss was later combined with residual moistures that were determined on the day that the heating value measurements were made. Residual moisture values of 6.25, 6.21, 6.10 and 6.12 percent were combined with the mean of the air-dry-loss, 17.62 percent, to yield total moistures of 22.77, 22.74, 22.65 and 22.66 percent, with a mean of 22.71 percent. The moisture content depends upon the ambient relative humidity and, therefore, inherent in the two-step moisture determination was the assumption that the relative humidity on the day of the air-dry-loss determination was the same as on the day of the residual moisture determination. Obviously, no such assumption is necessary for the single-step moisture technique, and for that reason it is a superior method.

Tables 60 and 63, which report residual moisture, ash, and sulfur content data, show reasonably good agreement between the furnace ash, bomb ash, and sulfur determinations. Mean calorific values for HHV2, HHV3-F, and HHV3-B are given in table 61 for the single-stage moisture method; they are, in $MJ\cdot kg^{-1}$ ($Btu\cdot lb^{-1}$), as follows: 19.28 (8291), 20.75 (8922), and 20.53 (8824), respectively. Similarly, mean calorific values for HHV-AD, HHV2, HHV3-F, and HHV3-B are given in table 64 for the two-stage moisture method; they are, in $MJ\cdot kg^{-1}$ ($Btu\cdot lb^{-1}$), as follows: 1825 (7846), 19.45 (8362), 20.98 (9021), and 20.79 (8938), respectively.

b. Steam Size Reduction RDF

A novel approach to the processing of MSW to an RDF is under development at Burke, Davoud and Associates in Richmond, Va. The process consists of introducing compacted MSW (∿ 20 $lb\cdot ft^3$) into a cylindrical pressure vessel 1 ft in diameter and 8 ft in length (wall thickness 1 inch) and filling the vesseling with steam until a pressure of 200 psig is reached. The pressure vessel has been dubbed: The Great Garbage Gun. The pressure of 200 psig is maintained for about 45 minutes and temperatures of around 380°F (193°C) are attained. This period is referred to as the "cook time". After this period, the door at one end of the cylinder is opened instantly and the steam and hot MSW expand rapidly into the air. Rapid release of steam and hot MSW is an important feature. The hot MSW undergoes high turbulence and is sheared into a finely divided material as it is falls onto a sandy land fill or is caught on a large plastic sheet. A sample of this product was sent to us by Mr. Donald K. Walter (DOE Office of Energy from Municipal Waste) for higher heating value determination. The DOE sample appeared to have an average particle size of about 0.125 inch.

Tables 65, 66, and 67 present the data for the RDF made in the Steam Size Reduction. This RDF sample had a furnace ash and bomb ash of 16.55 and 16.82 wt. percent, respectively, and is shown in table 66 along with data on residual moisture and sulfur content. In table 67, the mean values of HHV1, HHV3-F, HHV3-F and HHV3-B are reported in MJ·kg^{-1} (Btu·lb^{-1}) to be: 16.46 (7076), 17.17 (7383), 20.58 (8846), and 20.64 (8875), respectively.

The more highly processed RDF samples, such as that from Teledyne National (HHV2, 8598 Btu·lb^{-1}) and ECO FUEL-II (HHV2, 8205 Btu·lb^{-1}) are about 11 and 16 percent higher in calorific value than the Steam Size Reduction RDF sample we studied; however, the Burke-Davoud process is less complex. The sample we received may not have been representative of the original batch and looked different from other RDF samples which have been studied in our laboratory. It had a sweet odor reminiscent of brown sugar which is obtained from cooling semicellulose to glucose.

c. NCRR Storage Pile RDF

Some densified RDF which had been stored at the NCRR Resource Recovery Facility in Washington, D.C. for approximately 10 months had spontaneously charred during storage. As part of our RDF test method development program, we undertook to determine the calorific value of this charred sample. The extent of charring of the storage pile RDF could be estimated by examining the difference between the actually determined calorific value and the mean calorific value of most RDF samples, 2210 MJ·kg^{-1} (9500 Btu·lb^{-1}), on a moisture-ash-free basis.

The gross calorific value of the NCRR storage pile char RDF was comparable to that measured for other RDF's. This material was much like coal and did not pelletize very well. It had been milled down to a particle size of 0.5 mm (32 mesh) and we experienced sample containment problems during our combustion experiments. To make reliable measurements on this RDF char, it is probably necessary to follow the coal processing techniques and mill the sample to 0.25mm (60 mesh). In addition, the ashing technique may not have been correct. The ash remaining after treatment at 575°C was always larger in mass than the bomb residue. The ASTM editorial draft standard for the ashing of an RDF sample suggests 575°C as the ashing temperature. Established ASTM protocols for ashing coal and coke samples use 750°C. Apparently, a char must be ashed at temperatures higher than 575°C. In general, a modified set of procedures must be used for RDF char.

Tables 68, 69, and 70 provide the data determined for the NCRR charred storage pit sample. Table 68 gives the details of three calorimetric measurements on this sample, while table 69 gives data on residual moisture, ash, and sulfur content. The furnace ash (31.03 wt. percent) is larger than the bomb ash (25.85 wt. percent) by about 5 wt. percent. As mentioned earlier, the conditions of the two ash forming methods are quite different. The ashing temperature used in the ASTM ashing protocol (furnace ash method) is 575°C, and a much higher ash content was found here than with the bomb ash method. ASTM ashing protocols for coal use 750°C for the ashing temperature, while coke is ashed at 950°C. Temperatures attained in the combustion bomb during a calorimetric measurement are of the order 1500-2000°C; hence, a more complete ashing of a char sample is obtained. Table 70 shows the calorific values calculated to different bases. Mean values in $MJ \cdot kg^{-1}$ ($Btu \cdot lb^{-1}$) for HHV1, HHV2, HHV3-F, and HHV3-B are: 16.06 (6906), 16.41 (7057), 23.81 (10237), and 22.17 (9530), respectively.

d. <u>NCRR Low Ash RDF</u>

An RDF sample was collected by NCRR and was primarily office wastes from various government buildings in the Washington, D.C. area. This RDF was expected to have a low ash content. Heavy inerts, metals, non-conductors, and food wastes were expected to be present in very low concentrations. The overall processing of office wastes should be easier because of an expected low ash content. Correspondingly, the calculated higher heating value on a moisture-ash-free basis (HHV3) should also be lower than the usual RDF sample.

Tables 71, 72, and 73 provide data determined for the NCRR Low Ash RDF sample. Table 71 gives the details of three bomb calorimetric experiments in which the following calorific values were calculated on an as-determined basis in $MJ \cdot kg^{-1}$ ($Btu \cdot lb^{-1}$): 15.71 (6753), 15.68 (6741), and 15.62 (6715). Table 72 shows an average residual moisture, furnace ash, bomb ash, and sulfur content in wt. percent of: 5.27, 11.80, 11.29, and 0.095, respectively. Calorific values which have been calculated to different bases are found in table 73; average values for HHV1, HHV2, HHV3-F, and HHV3-B in $MJ \cdot kg^{-1}$ ($Btu \cdot lb^{-1}$) are: 15.67 (6736), 16.54 (7111), 18.75 (8062), and 18.64 (8015), respectively.

IV. Summary and Conclusions

This report provides data on 106 calorimetric measurements for 20 different RDF samples. In conjunction with the above calorimetric measurements, 32 calibration measurements were carried out from time to time on both small and large bomb calorimeters. Along with the calorimetric measurements, experiments were performed to determine the air dry loss, residual moisture, furnace ash, bomb ash, and sulfur content so that the calorimetric data could be corrected for their presence in the various RDF samples. A total of 283 such experiments were conducted. Critical examination and evaluation of the experimental results allow us to make certain conclusions with respect to the objectives of this interagency research agreement. The objectives, which are also stated in the Introduction, are as follows:

(1) to investigate the size reduction and homogenization required to obtain representative samples of refuse and RDF,

(2) to evaluate calorimetric methods for conventional-size (2.5 gram capacity) combustion bomb calorimeters. and an NBS large (25 gram capacity) combustion bomb calorimeter; and

(3) to develop test procedures for determining the calorific value of refuse and RDF samples using bomb calorimetric methods.

The measurement and the evaluation of data on higher heating values determined during ASTM round robin testing of RDF-3 respond to the first objective. It was found that the heterogeneity of RDF-3 and RDF which has been milled down to 0.5 mm particles causes the extraction of a representative sample from a larger sample lot to be difficult. The results of the statistical study on the ASTM testing of RDF-3 for three round robins shows that the within laboratory agreement for HHV2 (dry basis) is 250 Btu$\cdot$lb^{-1}; compare this value with the maximum acceptable variability for coal and coke which is 50 Btu$\cdot$lb^{-1}. Similarly, between laboratory agreement on processed RDF-3 samples for HHV2 (dry basis) is 750 Btu$\cdot$lb^{-1}; again, compare this value with the maximum acceptable limit for coal and coke which is 100 Btu·lb^{-1}. These levels of precision identify the current state-of-the-art with respect to the determination of the calorific value of refuse and RDF samples using the tentative ASTM procedures. Improvement in the levels of precision will come forth as progress is attained in separation, size reduction, and, in particular, in sampling procedures. As has been mentioned already, the heterogeneity of refuse and RDF samples milled down to 0.5 mm particles makes extraction of a representative sample difficult.

Studies on the grab and representative samples of Americology RDF provide additional responses to the first objective. The difference in HHV2 for the two samples amount to about 5.5 percent (364 Btu·lb^{-1}), while the measurement error (standard deviation) is about one percent ($\sim$ 70 Btu·lb^{-1}). Examination of the ash content data for the two Americology RDF samples shows that the average values for the furnace ash differ by 2.9 wt. percent, while the average values for the bomb ash differ by 1.2 wt. percent. These differences are due in part to the assumption that furnace ash data can be applied to higher heating values to calculate HHV3, and in part due to the overall heterogeneity of RDF. Here also, improvements in the agreement between a grab and representative sample will develop when better sampling procedures can be found.

Our studies on combustion measurements of three RDF samples (Teledyne National, ECO FUEL-II, and Americology) in the small (2.5 gram capacity) and large (25 gram capacity) bomb calorimeters respond to the second objective. The one percent difference in calculated calorific values between determinations carried out in the small and large calorimeters on the Teledyne National sample was due to the reactivity of stainless steel crucibles used in the large bomb, as compared to platinum crucibles used in the small bomb. A search for a suitable replacement for stainless steel resulted in the use of quartz crucibles for the small and large bomb calorimeters. Quartz crucibles were used for the subsequent measurements on ECO FUEL-II and Americology RDF samples. The results showed that the calorimetric data on ECO FUEL-II agreed to within 0.01 percent and on Americology RDF to within 0.2 percent. In summation, calorific values of equivalent high precision could be obtained in the large bomb calorimeter as well as the small one.

The calorimetric measurements performed on the New Castle County, Delaware MSW, which had been processed through the U.S. Bureau of Mines Resource Recovery facility in College Park, MD were all performed in the 25 gram capacity combustion bomb calorimeter. Samples which were composed of either 6 mm or 2 mm particles were burned in this calorimeter. The precisions observed were one percent or better and showed that RDF samples of larger particle size could be accommodated. The 6mm material was very difficult to pelletize, and therefore, the combustion of the material was less controlled. However, the calorific values determined on the 6mm material were measured with the same precision as those from 2mm or 0.5mm material. Therefore, for large (25 gram) samples, processing to only 6mm produces an RDF sample homogeneous enough to choose representative samples for bomb calorimetric measurements at the one percent level. This is an important finding, since a great deal of time and effort are consumed in the particle size reduction process. The main advantage of the large bomb calorimeter is in the time saved by being able to select and burn a sample after only limited processing.

The third objective, to develop test procedures, is realized in the descriptive content of the sections of this report under: II Experimental and III Results and Discussion. The step-wise methodology is outlined in detail and is also accompanied by interpretive and evaluative commentary. Data for the common measurement statistics (i.e., mean, standard deviation, and percent standard deviation) are given in all tables in which data on calorific values are calculated to different bases. A summary of the mean calorific values obtained in calorimetric measurements on the RDF's studied is provided in Table 74.

The present study, which introduces the 25 gram capacity bomb calorimeter for the combustion of refuse and RDF samples of large particle size, has also established the foundation for the next phase of research and development in the combustion calorimetry of larger samples. We have made some progress in the proper direction, however, there is still a need to develop a combustion calorimeter which can accommodate a sample several kilograms in size. The system developed should probably be a flow (constant pressure), rather a bomb (constant volume), system. One ton of refuse or RDF is equivalent to about 1,000,000 or 10^6 grams. Calorimeters which accommodate gram-size amounts of sample provide calorific values representing 1 to 25 ppm of a one ton field sample. A combustion calorimeter which accommodates kilogram-size samples will provide the additional assurance needed by combustion engineers who seek data on calorific values that such a sample represents multi-ton quantities of refuse and RDF. Such a system would provide reliability heretofore not available to the engineering community.

V. References

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TABLE 1. ENERGY EQUIVALENT VALUES

DETERMINED DURING FY 1979

(2.5 GRAM CAPACITY BOMB CALORIMETER)

Date	Expt. No.	E,si-empty/J·K^{-1}
07-31-78	1175	14550.22
08-01-78	1176	14550.44
08-17-78	1191	14550.51
08-18-78	1192	14543.31
08-18-78	1193	14545.06
12-26-78	1201	14552.37
12-27-78	1202	14543.17
01-04-79	1205	14534.19
01-04-79	1206	14539.05
01-05-79	1207	14537.68
01-05-79	1208	14541.32
01-06-79	1209	14542.14
01-07-79	1217	14544.70
01-17-79	1218	14546.37
01-18-79	1219	14541.72
01-19-79	1220	14542.89
01-19-79	1221*	14537.99
04-02-79	1238*	14535.57
04-05-79	1239*	14539.68
04-06-79	1240*	14554.84
04-09-79	1241*	14545.42
04-10-79	1242*	14546.47
04-11-79	1244	14538.72

E,si-mean/J·K^{-1} = 14543.64

Std. Dev./J·K^{-1} = 5.46 (0.04%)

Std. Dev. Mean/J·K^{-1} = 1.14 (0.01%)

*(Most recent calibration results, see Table 2 for details)

TABLE 2. DETERMINATION OF THE ENERGY EQUIVALENT
CALIBRATION EXPERIMENTS ON STANDARD SAMPLE BENZOIC ACID
(2.5 GRAM CAPACITY BOMB CALORIMETER, SIX TYPICAL EXPERIMENTS)

Expt. No.	1238	1239	1240	1241	1242	1244
$\Delta Uc(28°C)/J\cdot g^{-1}$	26410.68	26410.68	26410.67	26410.68	26410.68	26410.68
m-BA/g(vac.)	1.633500	1.633078	1.629424	1.628883	1.632755	1.632368
q-BA/J	43141.83	43130.72	43034.17	43019.92	42122.17	43111.96
q-ign/J	1.13	1.28	1.10	1.09	1.52	1.10
q-HNO_3/J	55.46	1.37	4.92	3.55	3.10	4.04
q-WC/J	34.20	34.58	34.38	34.48	34.55	34.44
q-corr to t_f/J	0.02	0.02	0.03	0.04	0.02	0.00
Q-Total/J	43232.64	43167.96	43074.60	43059.07	43161.38	43151.54
ΔT-corr/K	2.970405	2.965121	2.955640	2.956477	2.963287	2.964199
E-cal/$J\cdot K^{-1}$	14554.46	14558.59	14573.70	14564.32	14565.37	14557.57
Ei-cont/$J\cdot K^{-1}$	18.89	18.91	18.85	18.90	18.90	18.85
E,si-empty/$J\cdot K^{-1}$	14535.57	14539.68	14554.84	14545.42	14546.47	14538.72

E,si-mean/$J\cdot K^{-1}$	14543.45
Std. Dev./$J\cdot K^{-1}$	6.96 (0.05%)
Std. Dev. Mean/$J\cdot K^{-1}$	2.84 (0.02%)

TABLE 3. DETERMINATION OF THE ENERGY OF COMBUSTION OF "COMMERCIAL" BENZOIC ACID

(2.5 GRAM CAPACITY BOMB CALORIMETER)

Expt. No.	1188	1189	1190
E-cal/J·K^{-1}	14564.92	14564.54	14564.54
ΔT-corr/K	2.977312	2.379834	2.293676
Q-Total/J	43364.31	34661.17	33406.33
q-ign/J	1.03	0.94	0.95
q-WC/J	35.38	27.16	26.13
q-HNO$_3$/J	3.57	4.16	3.59
q-corr to t_f/J	-5.61	-3.62	-3.37
q-BA/J	43329.95	34632.53	33379.03
m-BA/g(vac.)	1.640286	1.311060	1.263567
ΔU°(28°C)/J·g^{-1}	26416.09	26415.66	26416.52

E,si-mean/J·g^{-1}	26416.09
Std. Dev./J·g^{-1}	0.00(0.00%)
Std. Dev. Mean/J·g^{-1}	0.00(0.00%)

TABLE 4. DETERMINATION OF THE ENERGY EQUIVALENT

CALIBRATION EXPERIMENTS ON "COMMERCIAL" BENZOIC ACID

(25 GRAM CAPACITY BOMB CALORIMETER)

Expt. No.	2042	2043	2047	2048	2049	2050
ΔUc(30°C)/J·g^{-1}	26411.36	26411.36	26411.36	26411.36	26411.36	26411.36
m-BA/g(vac.)	16.277573	16.252797	16.331530	16.228149	16.277317	16.209109
q-BA/J	429912.79	429258.41	431337.89	428607.41	429906.02	428104.55
q-ign/J	2.99	5.21	1.41	1.67	1.88	2.20
q-HNO$_3$	629.42	140.51	113.81	57.81	128.40	114.36
q-WC/J	500.61	507.93	511.51	507.83	508.65	507.05
q-corr to t$_f$/J	-1.27	-0.89	-1.34	-0.76	-1.14	-0.71
q-Fe/J	57.35	60.35	26.50	28.00	25.64	33.24
q-carbon/J	-100.62	-52.44	-11.80	-17.70	-13.77	-13.11
Q-Total/J	431001.28	429919.08	431977.98	429184.26	430555.68	428647.57
ΔT-corr/K	5.002353	4.987062	5.014116	4.979931	4.995473	4.973686
E-cal/J·K^{-1}	86159.71	86206.88	86152.37	86182.77	86189.17	86203.19
Ei-cont/J·K^{-1}	139.40	139.32	138.83	138.60	138.63	138.74
E,si-empty/J·K^{-1}	86020.31	86067.56	86013.54	86044.17	86050.54	86064.45

E,si-mean/J·K^{-1}	86043.43
Std. Dev./J·K^{-1}	22.36(0.026%)
Std. Dev. Mean/J·K^{-1}	9.13 (0.011%)

TABLE 5. DETERMINATION OF CALORIFIC VALUES

ASTM ROUND ROBIN TESTING OF RDF-3 (FIRST ROUND)

(2.5 GRAM CAPACITY BOMB CALORIMETER)

	Samples Equilibrated at 45% R.H.				Dried Samples			
Expt. No.	1167	1168	1169	1170	1171	1172	1173	1174
E-cal/J·K^{-1}	14558.69	14558.65	14558.59	14558.59	14558.64	14558.62	14558.59	14558.60
ΔT-corr/K	2.573101	2.485633	2.520173	2.470169	2.445994	2.466048	2.457463	2.453624
Q-total/kJ	37.461	36.187	36.690	35.962	35.610	35.902	35.777	35.721
q-ign/kJ	0.001	0.001	0.001	0.001	0.001	0.001	0.001	0.001
q-Fe/kJ	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
q-HNO_3/kJ	0.060	0.059	0.058	0.060	0.054	0.057	0.058	0.056
q-H_2SO_4/kJ	0.017	0.016	0.016	0.016	0.016	0.016	0.017	0.016
Q-RDF/kJ	37.383	36.111	36.615	35.885	35.539	35.828	35.702	35.648
m-RDF/g	2.28741	2.194897	2.229699	2.188164	2.184476	2.217163	2.231947	2.198730
HHV-AD/MJ·kg^{-1}	16.34	16.45	16.42	16.40	16.27	16.16	16.00	16.21
/(Btu·lb^{-1})	(7026)	(7073)	(7060)	(7051)	(68994)	(6947)	(6877)	(6970)

MJ·kg^{-1} = 429.9226 Btu·lb^{-1} was used to calculate the Btu·lb^{-1} values presented in parenthesis.

TABLE 6. DETERMINATION OF RESIDUAL MOISTURE, ASH, AND SULFUR CONTENT

ASTM ROUND ROBIN TESTING OF RDF-3 (FIRST ROUND)

(2.5 GRAM CAPACITY BOMB CALORIMETER)

(SAMPLES EQUILIBRATED AT 45% R.H.)

Expt. No.	Residual Moisture wt. %	Furnace Ash* wt. %	Bomb Ash* wt. %	Sulfur Content* wt. %
1167	5.069	25.54	23.83	0.1368
1168	5.108	24.20	23.34	0.1319
1169	4.968	23.40	23.42	0.1301
1170	4.979	23.59	23.69	0.1347
Mean	5.031	24.18	23.57	0.1334
Std. Dev.	0.068	0.97	0.23	0.003
% Std. Dev.	1.36%	4.00%	0.97%	2.22%

*(Data are reduced to a dry basis.)

TABLE 7. DETERMINATION OF RESIDUAL MOISTURE, ASH, AND SULFUR CONTENT

ASTM ROUND ROBIN TESTING OF RDF-3 (FIRST ROUND)

(2.5 GRAM CAPACITY BOMB CALORIMETER)

DRIED SAMPLES AT 105°C

Expt. No.	Air Dry Moisture wt.%	Residual Moisture wt.%	Furnace Ash* wt.%	Bomb Ash* wt.%	Sulfur Content* wt.%
1171	9.537	4.338	--	23.76	--
1172	--	4.146	--	24.37	--
1173	--	4.269	--	25.15	--
1174	--	4.252	--	23.85	--
Mean	--	4.251	--	24.28	--
Std. Dev.	--	0.079	--	0.64	--
% Std. Dev.	--	1.87%	--	2.63%	--

*(Data are reduced to a dry basis.)

TABLE 8. CALORIFIC VALUES CALCULATED TO DIFFERENT BASES

ASTM ROUND ROBIN TESTING OF RDF-3 (FIRST ROUND)

(2.5 GRAM CAPACITY BOMB CALORIMETER)

SAMPLES EQUILIBRATED AT 45% R.H.

	HHV-AD	HHV2	HHV3-F	HHV3-B
xpt. No.	MJ·kg^{-1} (Btu·lb^{-1})	MJ·kg^{-1} (Btu·lb^{-1})	MJ·kg^{-1} (Btu·lb^{-1})	MJ·kg^{-1} (Btu·lb^{-1})
1167	16.34 (7026)	17.22 (7401)	23.12 (9940)	22.60 (9717)
1168	16.45 (7073)	17.34 (7454)	22.87 (9833)	22.62 (9723)
1169	16.42 (7060)	17.28 (7429)	22.56 (9699)	22.56 (9701)
1170	16.40 (7051)	17.26 (7420)	22.59 (9711)	22.62 (9724)
Mean	16.40 (7052)	17.27 (7426)	22.78 (9796)	22.60 (9716)
td. Dev.	0.05 (20)	0.05 (22)	0.26 (114)	0.02 (11)
Std. Dev.	0.28%	0.29%	1.16%	0.11%

J·kg^{-1} = 429.9226 Btu·lb^{-1} was used to calculate the Btu·lb^{-1} values presented in parenthesis.

TABLE 9. CALORIFIC VALUES CALCULATED TO DIFFERENT BASES
ASTM ROUND ROBIN TESTING OF RDF-3 (FIRST ROUND)
(2.5 GRAM CAPACITY BOMB CALORIMETER)

DRIED SAMPLES AT 105°C

	HHV-AD	HHV2	HHV3-F	HHV-B
xpt. No.	MJ·kg^{-1} (Btu·lb^{-1})	MJ·kg^{-1} (Btu·lb^{-1})	MJ·kg^{-1} Btu·lb^{-1})	MJ·kg^{-1} (Btu·lb^{-1})
1171	16.27 (6994)	16.99 (7305)	22.41 (9635)	22.28 (9578)
1172	16.16 (6947)	16.88 (7256)	22.26 (9570)	2232 (9597)
1173	16.00 (6877)	16.71 (7182)	22.03 (9473)	22.32 (9595)
1174	16.21 (6970)	16.93 (7280)	22.33 (9602)	22.24 (9559)
ean	16.16 (6947)	16.88 (7256)	22.26 (9570)	22.29 (9582)
td. Dev.	0.12 (50)	0.12 (53)	0.16 (70)	0.04 (18)
Std. Dev.	0.72%	0.73%	0.73%	0.18%

J·kg^{-1} = 429.9226 Btu·lb^{-1} was used to calculate the Btu·lb^{-1} values presented in parenthesis.

TABLE 10. DETERMINATION OF CALORIFIC VALUES

ASTM ROUND ROBIN TESTING OF RDF-3 (SECOND ROUND)

(2.5 GRAM CAPACITY BOMB CALORIMETER)

	Interlab Comparison Sample*				RDF-3 PROCESSED TO 0.5 mm*			
Expt. No.	1222	1223	1224	1225	1226	1227	1228	1229
E-cal/J·K^{-1}	14561.78	14561.83	14561.77	14561.83	14561.81	14561.82	14561.86	14561.86
ΔT-corr/K	2.436375	2.523139	2.441165	2.514091	2.577896	2.583504	2.657341	2.623227
Q-total/kJ	35.478	36.742	35.548	36.610	37.539	37.621	38.696	38.199
q-ign/kJ	0.001	0.001	0.001	0.001	0.001	0.001	0.001	0.001
q-Fe/kJ	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
q-HNO_3/kJ	0.069	0.076	0.080	0.081	0.061	0.068	0.068	0.064
q-H_2SO_4/kJ	0.031	0.032	0.033	0.032	0.017	0.016	0.017	0.016
Q-RDF/kJ	35.377	36.633	35.433	36.496	37.460	37.536	38.609	38.118
m-RDF/g	2.136382	2.195192	2.123979	2.192481	2.162675	2.186798	2.223242	2.220071
HHV-AD/MJ·kg^{-1}	16.56	16.69	16.68	16.65	17.32	17.16	17.34	17.17
(Btu·lb^{-1})	(7119)	(7175)	(7172)	(7156)	(7447)	(7380)	(7466)	(7382)

*(Sample equilibrated at 45% R.H. prior to DET'N.)

MJ·kg^{-1} = 429.9226 Btu·lb^{-1} was used to calculate the Btu·lb^{-1} values presented in parenthesis.

TABLE 11. DETERMINATION OF RESIDUAL MOISTURE, ASH, AND SULFUR CONTENT

ASTM ROUND ROBIN TESTING OF RDF-3 (SECOND ROUND)

INTERLAB COMPARISON SAMPLE

(2.5 GRAM CAPACITY BOMB CALORIMETER)

Expt. No.	Air Dry Moisture wt.%	Residual Moisture (Loose) wt.%	Residual Moisture (Pellets) wt.%	Furnace Ash* (Loose) wt.%	Furnace Ash* (Pellets) wt.%	Bomb Ash* wt.%	Sulfur Content* wt.%
1222	18.42	3.271	6.126	19.95	19.84	19.40	0.2606
1223	--	3.325	6.202	19.43	19.82	18.32	0.2661
1224	--	3.352	6.218	20.35	20.11	18.62	0.2858
1225	--	3.458	6.167	20.16	20.28	18.96	0.2656
Mean	--	3.352	6.178	19.97	20.01	18.82	0.2695
Std. Dev.	--	0.079	0.0408	0.40	0.22	0.46	0.011
% Std. Dev.	--	2.34%	0.661%	1.99%	1.11%	2.46%	4.13%

*(Data are reduced to a dry basis.)

TABLE 12. DETERMINATION OF RESIDUAL MOISTURE, ASH, AND SULFUR CONTENT

ASTM ROUND ROBIN TESTING OF RDF-3 (SECOND ROUND)

RDF-3 PROCESSED TO 0.5mm POWDER

(2.5 GRAM CAPACITY BOMB CALORIMETER)

Expt. No.	Residual Moisture (Loose) wt.%	Residual Moisture (Pellets) wt.%	Furnace Ash* (Loose) wt.%	Furnace Ash* (Pellets) wt.%	Bomb Ash* wt.%	Sulfur Content* wt.%
1226	2.452	6.068	--	14.40	12.82	0.1464
1227	2.256	6.061	14.00	14.66	13.81	0.1317
1228	2.341	6.086	14.62	14.75	13.22	0.1386
1229	2.378	6.145	--	14.68	13.88	0.1312
Mean	2.357	6.090	14.31	14.62	13.43	0.1370
Std. Dev.	0.081	0.038	0.44	0.15	0.50	0.0071
% Std. Dev.	3.46%	0.626%	3.06%	1.05%	3.75%	5.21%

*(Data are reduced to a dry basis.)

TABLE 13. CALORIFIC VALUES CALCULATED TO DIFFERENT BASES

ASTM ROUND ROBIN TESTING OF RDF-3 (SECOND ROUND)

INTERLAB COMPARISON SAMPLE

(2.5 GRAM CAPACITY BOMB CALORIMETER)

Expt. No.	HHV-AD $MJ\cdot kg^{-1}$ ($Btu\cdot lb^{-1}$)	HHV2 $MJ\cdot kg^{-1}$ ($Btu\cdot lb^{-1}$)	HHV3-F $MJ\cdot kg^{-1}$ ($Btu\cdot lb^{-1}$) (Loose)	HHV3-F $MJ\cdot kg^{-1}$ ($Btu\cdot lb^{-1}$) (Pellets)	HHV3-B $MJ\cdot kg^{-1}$ ($Btu\cdot lb^{-1}$)
1222	16.56 (7119)	17.64 (7584)	22.04 (9474)	22.00 (9460)	21.89 (9409)
1223	16.69 (7175)	17.79 (7649)	22.08 (9494)	22.19 (9541)	21.78 (9365)
1224	16.68 (7172)	17.79 (7648)	22.33 (9602)	22.27 (9572)	21.86 (9397)
1225	16.65 (7156)	17.74 (7626)	22.22 (9552)	22.25 (9567)	21.89 (9411)
Mean	16.64 (7156)	17.74 (7627)	22.17 (9530)	22.18 (9535)	21.85 (9396)
Std. Dev.	0.06 (26)	0.07 (30)	0.13 (58)	0.12 (52)	0.05 (21)
% Std. Dev.	0.36%	0.40%	0.61%	0.54%	0.23%

$MJ\cdot kg^{-1} = 429.9226\ Btu\cdot lb^{-1}$ was used to calculate the $Btu\cdot lb^{-1}$ values presented in parenthesis.

TABLE 14. CALORIFIC VALUES CALCULATED TO DIFFERENT BASES

ASTM ROUND ROBIN TESTING OF RDF-3 (SECOND ROUND)

RDF-3 PROCESSED TO 0.5mm POWDER

(2.5 GRAM CAPACITY BOMB CALORIMETER)

	HHV-AD	HHV2	HHV3-F	HHV3-F	HHV3-B
	$MJ\cdot kg^{-1}$ $(Btu\cdot lb^{-1})$	$MJ\cdot kg^{-1}$ $(Btu\cdot lb^{-1})$	$MJ\cdot kg^{-1}$ $(Btu\cdot lb^{-1})$	$MJ\cdot kg^{-1}$ $(Btu\cdot lb^{-1})$	$MJ\cdot kg^{-1}$ $(Btu\cdot lb^{-1})$
Expt. No.			(Loose)	(Pellets)	
1226	17.32 (7447)	18.44 (7928)	--	21.54 (9262)	21.15 (9094)
1227	17.16 (7380)	18.27 (7856)	21.25 (9135)	21.41 (9206)	21.20 (9115)
1228	17.34 (7466)	18.49 (7950)	21.66 (9311)	21.69 (9325)	21.31 (9161)
1229	17.17 (7382)	18.29 (7865)	--	21.44 (9219)	21.24 (9133)
Mean	17.26 (7419)	18.37 (7900)	21.46 (9223)	21.52 (9253)	21.23 (9126)
Std. Dev.	0.10 (44)	0.11 (46)	6.29 (124)	0.12 (54)	0.07 (28)
%Std. Dev.	0.60%	0.59%	1.35%	0.58%	0.31%

$MJ\cdot kg^{-1}$ = 429.9226 $Btu\cdot lb^{-1}$ was used to calculate the $Btu\cdot lb^{-1}$ values presented in parenthesis.

TABLE 15. DETERMINATION OF CALORIFIC VALUES

ASTM ROUND ROBIN TESTING OF RDF-3 (THIRD ROUND)

(2.5 GRAM CAPACITY BOMB CALORIMETER)

	INTERLAB COMPARISON SAMPLE					RDF-3 PROCESSED TO 0.5 mm		
Expt. No.	1254	1255	1256	1257	1259	1260	1261	1262
E-cal/J·K^{-1}	14562.25	14562.22	14562.21	14562.21	14562.18	14562.21	14562.16	14562.21
ΔT-corr/K	2.855619	2.863918	2.844921	2.743247	2.845226	2.892132	2.881416	2.879575
Q-Total/kJ	41.584	41.705	41.428	40.676	41.433	42.116	40.785	41.933
q-ign/kJ	0.001	0.001	0.001	0.001	0.001	0.001	0.001	0.001
q-Fe/kJ	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
q-HNO_3/kJ	0.097	0.098	0.099	0.093	0.101	0.104	0.101	0.101
q-H_2SO_4/kJ	0.069	0.070	0.069	0.069	0.067	0.069	0.067	0.070
Q-RDF/kJ	41.417	41.536	41.260	40.512	41.263	41.941	40.626	41.761
m-RDF/g	2.623375	2.586581	2.581279	2.575939	2.547319	2.576651	2.527990	2.572948
HHV-AD/MJ·kg^{-1}	15.79	16.06	15.98	15.73	16.20	16.28	16.07	16.23
/(Btu·lb^{-1})	(6788)	(6904)	(6872)	(6761)	(6964)	(6998)	(6909)	(6978)

MJ·kg^{-1} = 429.9226 Btu·lb^{-1} was used to calculate the Btu·lb^{-1} values presented in parenthesis.

TABLE 16. DETERMINATION OF RESIDUAL MOISTURE, ASH, AND SULFUR CONTENT

ASTM ROUND ROBIN TESTING OF RDF-3 (THIRD ROUND)

INTERLAB COMPARISON SAMPLE

(2.5 GRAM CAPACITY BOMB CALORIMETER)

Expt. No.	Air Dry Moisture wt.%	Residual Moisture wt.%	Furnace Ash* wt.%	Bomb Ash* wt.%	Sulfur Content* wt.%
1254	26.35	4.522	21.29	21.25	0.4689
1255	--	4.458	22.05	19.74	0.4857
1256	--	4.382	22.31	19.97	0.4764
1257	--	4.376	22.54	21.29	0.4804
Mean	--	4.435	22.05	20.56	0.4779
Std. Dev.	--	0.069	0.54	0.82	0.0071
% Std. Dev.	--	1.562%	2.46%	4.00%	1.48%

*(Data are reduced to a dry basis.)

TABLE 17. DETERMINATION OF RESIDUAL MOISTURE, ASH, AND SULFUR CONTENT

ASTM ROUND ROBIN TESTING OF RDF-3 (THIRD ROUND)

RDF-3 PROCESSED TO 0.5mm POWDER

(2.5 GRAM CAPACITY BOMB CALORIMETER)

Expt. No.	Residual Moisture wt.%	Furnace Ash* wt.%	Bomb Ash* wt.%	Sulfur Content* wt.%
1259	4.773	22.83	20.82	0.4734
1260	4.740	22.08	20.87	0.4813
1261	4.843	22.60	21.45	0.4788
1262	4.826	22.56	20.79	0.4859
Mean	4.796	22.52	20.98	0.4799
Std. Dev.	0.048	0.32	0.31	0.0052
% Std. Dev.	0.991%	1.40%	1.49%	1.09%

*(Data are reduced to a dry basis.)

TABLE 18. CALORIFIC VALUES CALCULATED TO DIFFERENT BASES

ASTM ROUND ROBIN TESTING OF RDF-3 (THIRD ROUND)

INTERLAB COMPARISON SAMPLE

(2.5 GRAM CAPACITY BOMB CALORIMETER)

	HHV-AD	HHV2	HHV3-F	HHV3-B
Expt. No.	$MJ \cdot kg^{-1}$ ($Btu \cdot lb^{-1}$)	$MJ \cdot kg^{-1}$ ($Btu \cdot lb^{-1}$)	$MJ \cdot kg^{-1}$ ($Btu \cdot lb^{-1}$)	$MJ \cdot kg^{-1}$ ($Btu \cdot lb^{-1}$)
1254	15.79 (6788)	16.54 (7109)	21.01 (9032)	21.00 (9028)
1255	16.06 (6904)	16.81 (7226)	21.56 (9270)	20.94 (9003)
1256	15.98 (6872)	16.72 (7187)	21.52 (9251)	20.89 (8980)
1257	15.73 (6761)	16.45 (7071)	21.23 (9129)	20.90 (8983)
Mean	15.89 (6831)	16.63 (7148)	21.33(9170)	20.93 (8998)
Std. Dev.	0.16 (68)	0.16 (71)	0.26 (111)	0.05 (22)
% Std. Dev.	0.99%	0.99%	1.22%	0.246%

$MJ \cdot kg^{-1}$ = 429.9226 $Btu \cdot lb^{-1}$ was used to calculate the $Btu \cdot lb^{-1}$ values presented in parenthesis.

TABLE 19. CALORIFIC VALUES CALCULATED TO DIFFERENT BASES

ASTM ROUND ROBIN TESTING OF RDF-3 (THIRD ROUND)

RDF-3 PROCESSED TO 0.5mm POWDER

(2.5 GRAM CAPACITY BOMB CALORIMETER)

	HHV-AD	HHV2	HHV3-F	HHV3-B
Expt. No.	$MJ\cdot kg^{-1}$ ($Btu\cdot lb^{-1}$)	$MJ\cdot kg^{-1}$ ($Btu\cdot lb^{-1}$)	$MJ\cdot kg^{-1}$ ($Btu\cdot lb^{-1}$)	$MJ\cdot kg^{-1}$ ($Btu\cdot lb^{-1}$
1259	16.20 (6964)	17.01 (7313)	22.04 (9477)	21.48 (9237)
1260	16.28 (6998)	17.09 (7346)	21.93 (9428)	21.59 (9284)
1261	16.07 (6909)	16.89 (7261)	21.82 (9380)	21.50 (9243)
1262	16.23 (6978)	17.05 (7332)	22.02 (9467)	21.53 (9257)
Mean	16.19 (6962)	17.01 (7313)	21.95 (9438)	21.53 (9255)
Std. Dev.	0.09 (38)	0.09 (37)	0.10 (44)	0.05 (21)
% Std. Dev.	0.55%	0.51%	0.47%	0.23%

$MJ\cdot kg^{-1} = 429.9226\ Btu\cdot lb^{-1}$ was used to calculate the $Btu\cdot lb^{-1}$ values presented in parenthesis.

TABLE 20. DETERMINATION OF CALORIFIC VALUES
ASTM ROUND ROBIN TESTING OF RDF-3
NBS (SRM 1632a) BITUMINOUS COAL SAMPLE

(2.5 GRAM CAPACITY BOMB CALORIMETER)

Expt. No.	1012	1013
$E\text{-cal}/J\cdot K^{-1}$	14385.21	14385.21
$\Delta T\text{-corr}/K$	1.907333	1.916937
Q-total/kJ	27.437	27.576
q-ign/kJ	0.001	0.001
q-Fe/kJ	0.000	0.000
$q\text{-}HNO_3/kJ$	0.101	0.100
$q\text{-}H_2SO_4/kJ$	0.084	0.096
q-RDF/kJ	27.252	27.377
m-RDF/g	1.047333	1.052734
$HHV\text{-}AD/MJ\cdot kg^{-1}$	26.020	26.006
$/(Btu\cdot lb^{-1})$	(11187)	(11181)

$MJ\cdot kg^{-1} = 429.9226\ Btu\cdot lb^{-1}$ was used to calculate the $Btu\cdot lb^{-1}$ values presented in parenthesis.

TABLE 21. DETERMINATION OF RESIDUAL MOISTURE, ASH, AND SULFUR CONTENT

ASTM ROUND ROBIN TESTING OF RDF-3

NBS (SRM 1632A) BITUMINOUS COAL SAMPLE

(2.5 GRAM CAPACITY BOMB CALORIMETER)

Expt. No.	Residual Moisture wt. %	Furnace Ash wt. %	Bomb Ash wt. %	Sulfur Content wt. %
1012	1.15	22.19	--	1.38
1013	1.14	22.13	--	1.58
Mean	1.145	22.15	--	1.48

TABLE 22. CALORIFIC VALUES CALCULATED TO DIFFERENT BASES

ASTM ROUND ROBIN TESTING OF RDF-3

NBS (SRM 1632a) BITUMINOUS COAL SAMPLE

(2.5 GRAM CAPACITY BOMB CALORIMETER)

	HHV-AD	HHV2	HHV3-F	HHV3-B
Expt. No.	MJ·kg^{-1} (Btu·lb^{-1})	MJ·kg^{-1} (Btu·lb^{-1})	MJ·kg^{-1} (Btu·lb^{-1})	MJ·kg^{-1} (Btu·lb^{-1})
1012	26.021	26.323	33.829	--
	(11187)	(11317)	(14544)	
1013	26.007	26.307	33.783	--
	(11181)	(11310)	(14524)	
Mean	26.014	26.315	33.806	--
	(11184)	(11314)	(14534)	

MJ·kg^{-1} = 429.9226 Btu·lb^{-1} was used to calculate the Btu·lb^{-1} values presented in parenthesis.

TABLE 23. DETERMINATION OF CALORIFIC VALUES

AMERICOLOGY RDF, "GRAB" SAMPLE

(2.5 GRAM CAPACITY BOMB CALORIMETER)

Expt. No.	1128	1129	1130	1131	1132	1133	1134
E-cal/J·K^{-1}	14558.81	14558.88	14558.88	14558.84	14558.90	14558.85	14558.90
ΔT-corr/K	2.132686	2.169917	2.225881	2.215068	2.202305	2.187771	2.169966
Q-total/kJ	31.049	31.592	32.406	32.249	32.063	31.851	31.592
q-ign/kJ	0.001	0.000	0.001	0.001	0.001	0.001	0.001
q-Fe/kJ	0.000	0.000	0.000	0.000	0.000	0.000	0.000
q-HNO_3/kJ	0.057	0.054	0.057	0.060	0.055	0.056	0.053
q-H_2SO_4/kJ	0.029	0.028	0.030	0.029	0.029	0.029	0.029
Q-RDF/kJ	30.963	31.508	32.319	32.159	31.978	31.766	31.509
m-RDF/g	2.098995	2.121740	2.129789	2.129859	2.139318	2.143760	2.143270
HHV-AD/MJ·kg^{-1}	14.75	14.85	15.17	15.10	14.95	14.82	14.70
/(Btu·lb^{-1})	(6432)	(6384)	(6524)	(6491)	(6426)	(6371)	(6320)

MJ·kg^{-1} = 429.9226 Btu·lb^{-1} was used to calculate the Btu·lb^{-1} values presented in parenthesis.

TABLE 24. DETERMINATION OF RESIDUAL MOISTURE, ASH, AND SULFUR CONTENT

AMERICOLOGY RDF "GRAB" SAMPLE

(2.5 GRAM CAPACITY BOMB CALORIMETER)

Expt. No.	Residual Moisture** wt.%	Furnance Ash* wt.%	Bomb Ash* wt.%	Sulfur Content* wt.%
1128	2.9774	29.53	29.25	0.2410
1129	2.9664	29.22	28.91	0.2357
1130	3.0585	30.43	26.94	0.2445
1131	3.0499	29.40	27.40	0.2391
1132	3.0275	--	28.24	0.2391
1133	3.0069	--	28.88	0.2355
1134	2.9520	--	29.59	0.2382
Mean	3.0055	29.65	28.46	0.2390
Std. Dev.	0.0417	0.54	0.98	0.0031
% Std. Dev.	1.39%	1.82%	3.45%	1.30%

*(Data are reduced to a dry basis.)

**(Sample equilibrated at 30% R.H. prior to DET'N.)

TABLE 25. CALORIFIC VALUES CALCULATED TO DIFFERENT BASES

AMERICOLOGY RDF "GRAB" SAMPLE

(2.5 GRAM CAPACITY BOMB CALORIMETER)

	HHV-AD	HHV2	HHV3-F	HHV3-B
Expt. No.	$MJ \cdot kg^{-1}$ ($Btu \cdot lb^{-1}$)	$MJ \cdot kg^{-1}$ ($Btu \cdot lb^{-1}$)	$MJ \cdot kg^{-1}$ ($Btu \cdot lb^{-1}$)	$MJ \cdot kg^{-1}$ ($Btu \cdot lb^{-1}$)
1128	14.75 (6342)	15.21 (6539)	21.62 (9294)	21.50 (9242)
1129	14.85 (6384)	15.31 (6582)	21.76 (9357)	21.54 (9259)
1130	15.17 (6524)	15.65 (6726)	22.24 (9561)	21.41 (9206)
1131	15.10 (6491)	15.57 (6693)	22.13 (9514)	21.44 (9219)
1132	14.95 (6426)	15.41 (6626)	21.91 (9418)	21.48 (9233)
1133	14.82 (6371)	15.28 (6568)	21.72 (9336)	21.48 (9235)
1134	14.70 (6320)	15.16 (6516)	21.55 (9263)	21.53 (9255)
Mean	14.91 (6408)	15.37 (6607)	21.85 (9392)	21.48 (9236)
Std. Dev.	0.18 (76)	0.18 (78)	0.26 (112)	0.04 (19)
% Std. Dev.	1.19%	1.19%	1.19%	0.20%

$MJ \cdot kg^{-1}$ = 429.9226 $Btu \cdot lb^{-1}$ was used to calculate the $Btu \cdot lb^{-1}$ values presented in parenthesis.

TABLE 26. DETERMINATION OF CALORIFIC VALUES

AMERICOLOGY RDF, REPRESENTATIVE SAMPLE

(2.5 GRAM CAPACITY BOMB CALORIMETER)

Expt. No.	1177	1178	1180	1184	1194	1195	1197	1198	1200
E-cal/J·K^{-1}	14558.47	14558.43	14558.49	14558.56	14558.45	14558.50	14558.47	14558.40	14558.43
ΔT-corr/K	2.127851	2.172785	2.146900	2.300874	2.175473	2.201080	2.167258	2.145680	2.147408
Q-total/J	30.978	31.632	31.256	33.497	31.672	32.044	31.552	31.238	31.263
q-ign/J	0.001	0.001	0.001	0.001	0.001	0.001	0.001	0.001	0.001
q-Fe/J	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
q-HNO_3/J	0.0058	0.050	0.052	0.056	0.050	0.060	0.064	0.050	0.058
q-H_2SO_4/J	0.023	0.026	0.026	0.027	0.026	0.027	0.027	0.025	0.024
Q-RDF/J	30.897	31.556	31.176	33.414	31.595	31.957	31.461	31.161	31.180
m-RDF/g	2.023184	2.030289	2.028917	2.164149	2.043847	2.053797	2.077729	2.041180	1.979378
HHV-AD/MJ·kg^{-1}	15.27	15.44	15.37	15.44	15.46	15.56	15.14	15.27	15.31
/(Btu·lb^{-1})	(6566)	(6682)	(6606)	(6638)	(6646)	(6689)	(6510)	(6563)	(6582)

MJ·kg^{-1} = 429.9226 Btu·lb^{-1} was used to calculate the Btu·lb^{-1} values presented in parenthesis.

TABLE 27. DETERMINATION OF RESIDUAL MOISTURE, ASH, AND SULFUR CONTENT

AMERICOLOGY RDF, REPRESENTATIVE SAMPLE

(2.5 GRAM CAPACITY BOMB CALORIMETER)

Expt. No.	Residual Moisture Content** wt.%	Furnace Ash* wt.%	Bomb Ash* wt.%	Sulfur Content* wt.%
1177	5.131	26.727	30.24	0.2012
1178	5.240	26.896	28.98	0.2285
1180	--	--	29.88	0.2317
1184	5.175	27.098	29.26	0.2238
1194	--	--	29.15	0.2276
1195	5.211	26.544	28.66	0.2362
1197	--	--	30.49	0.2309
1198	--	--	30.26	0.2238
1200	--	--	29.64	0.2102
Mean	5.189	26.816	29.62	0.2238
Std. Dev.	0.047	0.237	0.64	0.0112
% Std. Dev.	0.907%	0.882%	2.17%	4.99%

*(Data are reduced to a dry basis.)

**(Sample equilibrated at 45% R.H. prior to DET'N.)

TABLE 28. CALORIFIC VALUES CALCULATED TO DIFFERENT BASES

AMERICOLOGY RDF, REPRESENTATIVE SAMPLE

(2.5 GRAM CAPACITY BOMB CALORIMETER)

	HHV-AD	HHV2	HHV3-F	HHV3-B
Expt. No.	$MJ\cdot kg^{-1}$ ($Btu\cdot lb^{-1}$)	$MJ\cdot kg^{-1}$ ($Btu\cdot lb^{-1}$)	$MJ\cdot kg^{-1}$ ($Btu\cdot lb^{-1}$)	$MJ\cdot kg^{-1}$ ($Btu\cdot lb^{-1}$)
1177	15.27 (6566)	16.11 (6925)	22.01 (9462)	22.53 (9708)
1178	15.54 (6682)	16.39 (7048)	22.40 (9630)	22.60 (9718)
1180	15.37 (6606)	16.21 (6968)	22.15 (9521)	22.61 (9722)
1184	15.44 (6638)	16.28 (7001)	22.25 (9566)	22.54 (9689)
1194	15.46 (6646)	16.31 (7010)	22.28 (9579)	22.53 (9687)
1195	15.56 (6689)	16.41 (7056)	22.42 (9641)	22.53 (9688
1197	15.14 (6510)	15.97 (6866)	21.82 (9382)	22.47 (9658)
1198	15.27 (6563)	16.10 (6922)	22.00 (9459)	22.58 (9708)
1200	15.31 (6582)	16.15 (6942)	22.06 (9486)	22.46 (9655)
Mean	15.37 (6609)	16.21 (6971)	22.16 (9525)	22.54 (9693)
Std. Dev.	0.14 (60)	0.15 (63)	0.20 (86)	0.06 (24)
% Std. Dev.	0.90%	0.91%	0.90%	0.25%

$MJ\cdot kg^{-1} = 429.9226\ Btu\cdot lb^{-1}$ was used to calculate the $Btu\cdot lb^{-1}$ values presented in parenthesis.

TABLE 29. DETERMINATION OF CALORIFIC VALUES

CALORIMETER COMPARISON MEASUREMENTS

TELEDYNE NATIONAL RDF

(2.5 GRAM CAPACITY BOMB CALORIMETER)

Expt No.	1233	1234	1235
E-cal/$J \cdot K^{-1}$	14561.54	14561.63	14561.62
ΔT-corr/K	2.660558	2.652241	2.634082
Q-total/kJ	38.742	38.621	38.357
q-ign/kJ	0.001	0.001	0.001
q-Fe/kJ	0.000	0.000	0.000
q-HNO_3/kJ	0.069	0.068	0.067
q-H_2SO_4/kJ	0.16	0.17	0.018
Q-RDF/kJ	38.656	38.535	38.271
m-RDF/g	2.001529	1.986701	1.979255
HHV-AD/$MJ \cdot kg^{-1}$	19.31	19.40	19.34
/($Btu \cdot lb^{-1}$)	(8303)	(8339)	(8313)

TABLE 30. DETERMINATION OF CALORIFIC VALUES

CALORIMETER COMPARISON MEASUREMENTS

TELEDYNE NATIONAL RDF

(25 GRAM CAPACITY BOMB CALORIMETER)

Expt. No.	2051	2052	2053	2054	2055	2056
E-cal/J·k^{-1}	86220.02	82218.55	86219.98	86218.54	86182.10	86181.92
ΔT-corr/K	4.516466	4.506280	4.509029	4.497590	4.515228	4.495162
Crucible material	316SS	316SS	316SS	316SS	Pt	316SS
Q-total/kJ	389.410	388.525	388.768	387.776	389.132	387.402
q-ign/kJ	0.002	0.002	0.002	0.002	0.001	0.001
q-Fe/kJ	0.036	0.037	0.032	0.033	0.027	0.030
q-HNO_3/kJ	0.643	0.476	0.560	0.585	0.674	0.653
q-H_2SO_4/kJ	0.161	0.130	0.147	0.150	0.173	0.152
Q-RDF/kJ	388.568	387.880	388.027	387.006	388.257	386.565
m-RDF/g	20.00515	19.94353	19.98236	19.96562	19.78861	19.60786
HHV-AD/MJ·kg^{-1}	19.42	19.45	19.42	19.38	19.63	19.71
/(Btu·lb^{-1})	(8351)	(8362)	(8348)	(8333)	(8439)	(8476)

TABLE 31. DETERMINATION OF RESIDUAL MOISTURE, ASH, AND SULFUR CONTENT

CALORIMETER COMPARISON MEASUREMENTS

TELEDYNE NATIONAL RDF

(2.5 GRAM CAPACITY BOMB CALORIMETER)
(25 GRAM CAPACITY BOMB CALORIMETER)

Expt. No.	Residual** Moisture wt.%	Furnace Ash* wt.%	Bomb Ash* wt.%	Sulfur Content* wt.%
1233	5.036	15.843	15.53	0.1462
1234	--	--	14.61	0.1544
1235	5.052	15.793	15.40	0.1601
2051	5.107	15.996	15.19	0.1444
2052	- -	--	15.40	0.1170
2053	5.067	15.866	15.60	0.1324
2054	--	--	15.69	0.1348
2055	--	--	15.38	0.1573
2056	--	--	15.67	0.1397
Mean	5.065	15.874	15.386	0.1429
Std. Dev.	0.030	0.086	0.332	0.0137
% Std. Dev.	0.592%	0.545%	2.156%	9.598%

*(Data are reduced to a dry basis.)

**(Sample equilibrated at 45% R.H. prior to DET'N.)

TABLE 32. CALORIFIC VALUES CALCULATED TO DIFFERENT BASES

CALORIMETER COMPARISON MEASUREMENTS

TELEDYNE NATIONAL RDF

(2.5 GRAM CAPACITY BOMB CALORIMETER)

Expt. No.	HHV-AD MJ·kg^{-1} (Btu·lb^{-1})	HHV2 MJ·kg^{-1} (Btu·lb^{-1})	HHV3-F MJ·kg^{-1} (Btu·lb^{-1})	HHV3-B MJ·kg^{-1} (Btu·lb^{-1})
	(2.5 GRAM CAPACITY BOMB CALORIMETER)			
1233	19.31 (8303)	20.34 (8746)	24.21 (10409)	24.08 (10355)
1234	19.40 (8339)	20.43 (8784)	24.86 (10686)	23.93 (10287)
1235	19.34 (8313)	20.37 (8757)	24.57 (10564)	24.07 (10350)
Mean	19.35 (8318)	20.38 (8762)	24.55 (10553)	24.03 (10331)
Std. Dev.	0.04 (19)	0.05 (20)	0.32 (139)	0.09 (38)
% Std. Dev.	0.22%	0.22%	1.32%	0.37%
	(25.0 GRAM CAPACITY BOMB CALORIMETER)			
2051	19.42 (8351)	20.46 (8796)	24.32 (10456)	24.13 (10372)
2052	19.45 (8362)	20.49 (8808)	24.35 (10470)	24.22 (10411)
2053	19.42 (8348)	20.45 (8794)	24.31 (10453)	24.24 (10419)
2054	19.38 (8333)	20.42 (8778)	24.27 (10434)	24.22 (10411)
2055	19.63 (8439)	20.68 (8890)	24.58 (10567)	24.43 (10505)
2056	19.71 (8476)	20.77 (8928)	24.69 (10613)	24.63 (10587)
Mean	19.50 (8385)	20.54 (8832)	24.42 (10499)	24.31 (10451)
Std. Dev.	0.14 (58)	0.14 (61)	0.17 (73)	0.19 (80)
% Std. Dev.	0.69%	0.69%	0.70%	0.76%

MJ·kg^{-1} = 429.9226 Btu·lb^{-1} was used to calculate the Btu·lb^{-1} values presented in parenthesis.

TABLE 33. DETERMINATION OF CALORIFIC VALUES
CALORIMETER COMPARISON MEASUREMENTS
ECO FUEL-II RDF
(2.5 GRAM CAPACITY BOMB CALORIMETER)

Expt. No.	1245	1246	1247
E-cal/$J\cdot K^{-1}$	14561.78	14561.99	148562.00
ΔT-corr/K	2.781353	3.055371	3.055691
Q-total/kJ	40.501	44.492	44.497
q-ign/kJ	0.001	0.001	0.001
q-Fe/kJ	0.000	0.000	0.000
q-HNO_3/kJ	0.102	0.109	0.108
q-H_2SO_4/kJ	0.060	0.067	0.069
Q-RDF/kJ	40.339	44.315	44.319
m-RDF/g	2.147869	2.359926	2.361323
HHV-AD/$MJ\cdot kg^{-1}$	18.78	18.79	18.77
/($Btu\cdot lb^{-1}$)	(8074)	(8073)	(8069)

$MJ\cdot kg^{-1}$ = 429.9226 $Btu\cdot lb^{-1}$ was used to calculate the $Btu\cdot lb^{-1}$ values presented in parenthesis.

TABLE 34. DETERMINATION OF CALORIFIC VALUES

CALORIMETER COMPARISON MEASUREMENTS

ECO FUEL-II RDF

(25 GRAM CAPACITY BOMB CALORIMETER)

Expt. No.	2057	2059	2060	2061	2062
E-cal/J·K^{-1}	86200.33	86206.47	86206.09	86205.58	86205.28
ΔT-corr/K	5.089892	5.098377	5.103465	5.122380	5.123829
Q-total/kJ	438.750	439.513	439.950	441.578	441.654
q-ign/kJ	0.001	0.001	0.001	0.001	0.002
q-Fe/kJ	0.000	0.000	0.000	0.000	0.000
q-HNO_3/kJ	0.980	0.972	0.935	0.966	0.938
q-H_2SO_4/kJ	0.554	0.567	0.613	0.683	0.564
Q-RDF/kJ	437.214	437.973	438.401	439.928	440.150
m-RDF/g	23.29107	23.32159	23.35230	23.43669	23.44928
HHV-AD/MJ·kg^{-1}	18.77	18.78	18.77	18.77	18.77
/(Btu·lb^{-1})	(8070)	(8074)	(8071)	(8070)	(8070)

MJ·kg^{-1} = 429.9226 Btu·lb^{-1} was used to calculate the Btu·lb^{-1} values presented in parenthesis.

TABLE 35. DETERMINATION OF RESIDUAL MOISTURE, ASH, AND SULFUR CONTENT

CALORIMETER COMPARISON MEASUREMENTS

ECO FUEL-II RDF

(2.5 GRAM CAPACITY BOMB CALORIMETER)

(25 GRAM CAPACITY BOMB CALORIMETER)

Expt. No.	Residual Moisture** wt.%	Furnace Ash* wt.%	Bomb Ash* wt.%	Sulfur Content* wt.%
1245	1.629	13.149	11.70	0.4841
1246	--	--	11.92	0.4907
1247	1.625	13.199	11.85	0.5093
2057	--	--	11.83	0.4127
2059	--	--	11.78	0.4219
2060	--	--	11.71	0.4551
2061	--	--	11.87	0.5053
2062	--	--	11.79	0.4174
Mean	1.627	13.714	11.806	0.4621
Std. Dev.	0.002	0.035	0.077	0.0405
% Std. Dev.	0.144%	0.266%	0.648%	8.772%

*(Data are reduced to a dry basis.)

**(Sample equilibrated to 30% R.H. prior to DET'N.)

TABLE 36. CALORIFIC VALUES CALCULATED TO DIFFERENT BASES
CALORIMETER COMPARISON MEASUREMENTS
ECO FUEL-II RDF

	HHV-AD	HHV2	HHV3-F	HHV3-B
Expt. No.	MJ·kg^{-1} (Btu·lb^{-1})	MJ·kg^{-1} (Btu·lb^{-1})	MJ·kg^{-1} (Btu·lb^{-1})	MJ·kg^{-1} (Btu·lb^{-1})
	(2.5 GRAM CAPACITY BOMB CALORIMETER)			
1245	18.78 (8074)	19.09 (8208)	21.99 (9453)	21.62 (9295)
1246	18.79 (8073)	19.09 (8207)	21.99 (9452)	21.67 (9317)
1247	18.77 (8069)	19.08 (8203)	21.97 (9447)	21.64 (9305)
Mean	18.78 (8072)	19.09 (8206)	21.98 (9451)	21.64 (9306)
Std. Dev.	0.01 (3)	0.01 (3)	0.01 (3)	0.03 (11)
% Std. Dev.	0.03%	0.03%	0.03%	0.12%
	(25 GRAM CAPACITY BOMB CALORIMETER)			
2058	18.77 (8070)	19.08 (8204)	21.98 (9449)	21.64 (9305)
2059	18.78 (8074)	19.09 (8207)	21.99 (9453)	21.64 (9303)
2060	18.77 (8071)	19.08 (8205)	21.98 (9449)	21.61 (9293)
2061	18.77 (8070)	19.08 (8204)	21.98 (9448)	21.65 (9309)
2062	18.77 (8070)	19.08 (8203)	21.98 (9448)	21.63 (9300)
Mean	18.77 (8071)	19.08 (8205)	21.98 (9449)	21.64 (9302)
Std. Dev.	0.004 (2)	0.004 (2)	0.005 (2)	0.01 (6)
% Std. Dev.	0.02%	0.02%	0.02%	0.06%

MJ·kg^{-1} = 429.9226 Btu·lb^{-1} was used to calculate the Btu·lb^{-1} values presented in parenthesis.

TABLE 37. DETERMINATION OF CALORIFIC VALUES
CALORIMETER COMPARISON MEASUREMENTS
AMERICOLOGY RDF
(2.5 GRAM CAPACITY BOMB CALORIMETER)

Expt. No.	1250	1251	1252
E-cal/J·K^{-1}	14562.39	14562.39	14562.36
ΔT-corr/K	2.965378	2.972371	2.944801
Q-total/kJ	43.183	43.285	42.883
q-ign/kJ	0.001	0.001	0.001
q-Fe/kJ	0.000	0.000	0.000
q-HNO$_3$/kJ	0.034	0.041	0.033
q-H$_2$SO$_4$/kJ	0.037	0.036	0.035
Q-RDF/kJ	43.111	43.206	42.814
m-RDF/g	2.761429	2.750133	2.730152
HHV-AD/MJ·kg^{-1}	15.61	15.71	15.68
/(Btu·lb^{-1})	(6712)	(6754)	(6742)

MJ·kg^{-1} = 429.9226 Btu·lb^{-1} was used to calculate the Btu·lb^{-1} values presented in parenthesis.

TABLE 38. DETERMINATION OF CALORIFIC VALUES
CALORIMETER COMPARISON MEASUREMENTS
AMERICOLOGY RDF
(25 GRAM CAPACITY BOMB CALORIMETER)

Expt. No.	2064	2065	2066	2067	2068	2069
E-cal/J$\cdot$K^{-1}	86211.66	86204.17	86205.00	86203.11	86204.06	86202.97
ΔT-corr/K	4.997517	4.963379	4.975904	4.974333	4.975251	4.936259
Q-total/kJ	430.844	427.864	428.948	428.803	428.887	425.520
q-ign/kJ	0.003	0.003	0.002	0.006	0.002	0.003
q-Fe/kJ	0.000	0.000	0.000	0.000	0.000	0.000
q-HNO_3/kJ	0.672	0.666	0.670	0.697	0.715	0.725
q-H_2SO_4/kJ	0.306	0.303	0.295	0.371	0.318	0.320
Q-RDF/kJ	429.863	426.892	427.980	427.730	427.852	424.472
m-RDF/g	27.3768	27.1314	27.1988	27.1076	27.2651	27.2717
HHV-AD/MJ$\cdot$kg^{-1}	15.70	15.73	15.74	15.79	15.69	15.56
/(Btu$\cdot$lb^{-1})	(6751)	(6765)	(6765)	(6784)	(6746)	(6692)

MJ$\cdot$kg^{-1} = 429.9226 Btu$\cdot$lb^{-1} was used to calculate the Btu$\cdot$lb^{-1} values presented in parenthesis.

TABLE 39. DETERMINATION OF RESIDUAL MOISTURE, ASH, AND SULFUR CONTENT

CALORIMETER COMPARISON MEASUREMENTS

AMERICOLOGY RDF

Expt. No.	Residual Moisture wt.%	Furnace Ash* wt.%	Bomb Ash* wt.%	Sulfur Content* wt.%
(2.5 GRAM CAPACITY BOMB CALORIMETER)				
1250	5.764	27.441	27.06	0.2398
1251	--	--	26.43	0.2404
1252	5.794	27.709	26.66	0.2329
(25 GRAM CAPACITY BOMB CALORIMETER)				
2064	--	--	26.43	0.2023
2065	--	--	26.64	0.2023
2066	--	--	26.55	0.1967
2067	--	--	26.17	0.2478
2068	--	--	26.74	0.2115
2069	--	--	26.44	0.2126
Mean	5.779	27.575	26.57	0.2207
Std. Dev.	0.021	0.190	0.25	0.0195
% Std. Dev.	0.359%	0.689%	0.940%	8.832%

*(Data are reduced to a dry basis.)

TABLE 40. CALORIFIC VALUES CALCULATED TO DIFFERENT BASES
CALORIMETER COMPARISON MEASUREMENTS
AMERICOLOGY RDF

	HHV-AD	HHV2	HHV3-F	HHV3-B
Expt. No.	MJ·kg^{-1} (Btu·lb^{-1})	MJ·kg^{-1} (Btu·lb^{-1})	MJ·kg^{-1} (Btu·lb^{-1})	MJ·kg^{-1} (Btu·lb^{-1})
(2.5 GRAM CAPACITY BOMB CALORIMETER)				
1250	15.61 (6712)	16.57 (7124)	22.88 (9836)	22.72 (9766)
1251	15.71 (6754)	16.67 (7169)	23.02 (9898)	22.66 (9744)
1252	15.68 (6742)	16.64 (7156)	22.98 (9880)	22.69 (9757)
Mean	15.67 (6736)	16.63 (7150)	22.96 (9871)	22.69 (9756)
Std. Dev.	0.05 (21.63)	0.05 (23)	0.07 (32)	0.03 (11)
% Std. Dev.	0.32%	0.32%	0.32%	0.11%
(25 GRAM CAPACITY BOMB CALORIMETER)				
2064	15.70 (6751)	16.66 (7165)	23.01 (9882)	22.65 (9738)
2065	15.73 (6765)	16.70 (7179)	23.06 (9913)	22.76 (9786)
2066	15.74 (6765)	16.70 (7180)	23.06 (9913)	22.74 (9775)
2067	15.79 (6784)	16.75 (7200)	23.12 (9941)	22.68 (9753)
2068	15.69 (6746)	16.65 (7160)	23.00 (9886)	22.73 (9773)
2069	15.56 (6692)	16.52 (7102)	22.81 (9806)	22.46 (9654)
Mean	15.70 (6750)	16.66 (7164)	23.01 (9892)	22.67 (9746)
Std. Dev.	0.07 (32)	0.08 (34)	0.11 (46)	0.11 (48)
% Std. Dev.	0.47%	0.47%	0.47%	0.50%

MJ·kg^{-1} = 429.9226 Btu·lb^{-1} was used to calculate the Btu·lb^{-1} values presented in parenthesis.

TABLE 41. DETERMINATION OF CALORIFIC VALUES
NEW CASTLE COUNTY, DELAWARE MSW, PROCESSED
THROUGH THE BUREAU OF MINES FACILITY, COLLEGE PARK, MD.
(25 GRAM CAPACITY BOMB CALORIMETER)

Processing Path	Cyclone No. 1, Secondary Shred, 2 inch pieces			Cyclone No. 1, Trommel Undersize, +14 mesh		
Expt. No.	1011	1014	1015	1029	1030	1031
E-cal/J·K^{-1}	86206.63	86204.13	86101.90	85787.94	85787.02	85787.83
ΔT-corr/K	5.038961	4.708003	4.748646	3.406100	3.427473	3.512099
Q-total/KJ	434.392	405.849	408.867	292.202	294.033	301.295
q-ign/kJ	0.003	0.002	0.003	0.003	0.003	0.003
q-Fe/kJ	0.000	0.000	0.000	0.000	0.000	0.000
q-HNO_3/kJ	0.669	0.632	0.634	0.540	0.565	0.583
q-H_2SO_4/kJ	0.181	0.174	0.174	0.267	0.275	0.289
Q-RDF/kJ	433.539	405.041	408.056	291.392	293.190	300.420
m-RDF/g	23.7834	22.1607	22.2285	22.1384	22.0979	22.0558
HHV-AD/MJ·kg^{-1}	18.23	18.28	18.36	13.16	13.27	13.62
/(Btu·lb^{-1})	(7837)	(7858)	(7892)	(5659)	(5704)	(5856)

MJ·kg^{-1} = 429.9226 Btu·lb^{-1} was used to calculate the Btu·lb^{-1} values presented in parenthesis.

TABLE 42. DETERMINATION OF CALORIFIC VALUES

NEW CASTLE COUNTY, DELAWARE MSW, PROCESSED

THROUGH THE BUREAU OF MINES FACILITY, COLLEGE PARK, MD.

(25 GRAM CAPACITY BOMB CALORIMETER)

Processing Path	Cyclone No. 2, Secondary Shred, 2 inch pieces			Cyclone No. 2, Trommel Undersize, +14 mesh		
Expt. No.	1018	1019	1020	1035	1037	1038
E-cal/J·K^{-1}	85788.62	85787.73	85788.33	85793.38	85795.21	85793.81
ΔT-corr/K	5.046045	5.094753	5.096709	5.107338	5.111198	5.012434
Q-total/kJ	432.893	437.067	437.238	438.176	438.516	430.036
q-ign/kJ	0.002	0.003	0.003	0.002	0.002	0.002
q-Fe/kJ	0.000	0.000	0.000	0.000	0.000	0.000
q-HNO_3/kJ	0.628	0.671	0.671	0.817	0.840	0.843
q-H_2SO_4/kJ	0.157	0.157	0.162	0.347	0.337	0.340
Q-RDF/kJ	432.106	436.236	436.402	437.008	437.337	428.851
m-RDF/g	22.8831	23.0440	23.0188	28.4674	28.5886	28.4646
HHV-AD/MJ·kg^{-1}	18.88	18.93	18.96	15.35	15.30	15.07
/(Btu·lb^{-1})	(8118)	(8137)	(8151)	(6600)	(6577)	(6477)

MJ·kg^{-1} = 429.9226 Btu·lb^{-1} was used to calculate the Btu·lb^{-1} values presented in parenthesis.

TABLE 43. DETERMINATION OF CALORIFIC VALUES
NEW CASTLE COUNTY, DELAWARE MSW, PROCESSED
THROUGH THE BUREAU OF MINES FACILITY, COLLEGE PARK, MD.
(25 GRAM CAPACITY BOMB CALORIMETER)

Processing Path	Secondary Shred, Cyclone No. 3, as collected		
Expt. No.	1023	1024	1025
$E\text{-cal}/J \cdot K^{-1}$	85787.89	85788.16	85789.37
$\Delta T\text{-corr}/K$	5.130192	5.118568	5.047555
Q-total/kJ	440.108	439.113	433.026
q-ign/kJ	0.002	0.003	0.003
q-Fe/kJ	0.000	0.000	0.000
$q\text{-}HNO_3/kJ$	0.710	0.731	0.686
$q\text{-}H_2SO_4/kJ$	0.152	0.146	0.152
Q-RDF/kJ	439.245	438.233	432.186
m-RDF/g	22.6636	22.6696	22.5781
$HHV\text{-}AD/MJ \cdot kg^{-1}$	19.38	19.33	19.14
$/(Btu \cdot lb^{-1})$	(8332)	(8311)	(8229)

$MJ \cdot kg^{-1} = 429.9226\ Btu \cdot lb^{-1}$ was used to calculate the $Btu \cdot lb^{-1}$ values presented in parenthesis.

TABLE 44. DETERMINATION OF CALORIFIC VALUES
NEW CASTLE COUNTY, DELAWARE MSW, PROCESSED
THROUGH THE BUREAU OF MINES FACILITY, COLLEGE PARK, MD.
(25 GRAM CAPACITY BOMB CALORIMETER)

Processing Path	Heavy Organic Combustibles			Organic Wastes, Jig Over Flow		
Expt. No.	1041	1042	1043	1046	1047	1048
E-cal/J$\cdot$K^{-1}	85772.98	85771.23	85772.43	85770.95	85772.87	85771.14
ΔT-corr/K	1.427986	1.451410	1.403397	1.320258	1.426406	1.222589
Q-total/kJ	122.483	124.489	120.373	113.240	122.347	104.863
q-ign/kJ	0.001	0.002	0.003	0.002	0.002	0.003
q-Fe/kJ	0.000	0.000	0.000	0.000	0.000	0.000
q-HNO$_3$/kJ	0.227	0.281	0.270	0.144	0.154	0.130
q-H$_2$SO$_4$/kJ	0.070	0.065	0.057	0.047	0.050	0.044
Q-RDF/kJ	122.185	124.141	120.042	113.048	122.141	104.686
m-RDF/g	5.8045	5.9240	5.7158	5.8160	6.1435	5.2689
HHV-AD/MJ$\cdot$kg^{-1}	21.05	20.96	21.00	19.44	19.88	19.87
/(Btu$\cdot$lb^{-1})	(9050)	(9009)	(9029)	(8357)	(8548)	(8542)

MJ$\cdot$kg^{-1} = 429.9226 Btu$\cdot$lb^{-1} was used to calculate the Btu$\cdot$lb^{-1} values presented in parenthesis.

TABLE 45. DETERMINATION OF AIR DRY LOSS, RESIDUAL MOISTURE, ASH, AND SULFUR CONTENT

NEW CASTLE COUNTY, DELAWARE MSW, PROCESSED

THROUGH THE BUREAU OF MINES FACILITY, COLLEGE PARK, MD.

CYCLONE NO. 1, SECONDARY SHRED, 2 INCH PIECES

TOTAL WEIGHT OF SAMPLE, 3268.3 GRAMS

(25 GRAM CAPACITY BOMB CALORIMETER)

Expt. No.	Air Dry Loss wt.%	Residual Moisture wt.%	Furnace Ash* wt.%	Bomb Ash* wt.%	Sulfur Content* wt.%
1011	8.761	5.324	12.503	11.824	0.137
1014	8.454	5.338	12.517	11.460	0.142
1015	8.481	--	--	10.932	0.141
Mean	8.565	5.331	12.510	11.405	0.140
Std. Dev.	0.170	0.010	0.010	0.449	0.003
% Std. Dev.	1.985%	0.183%	0.080%	3.932%	1.890%

*(Data are reduced to a dry basis.)

TABLE 46. DETERMINATION OF AIR DRY LOSS, RESIDUAL MOISTURE, ASH, AND SULFUR CONTENT
NEW CASTLE COUNTY, DELAWARE MSW, PROCESSED
THROUGH THE BUREAU OF MINES FACILITY, COLLEGE PARK, MD.
CYCLONE NO. 1, TROMMEL UNDERSIZE, +14 MESH
TOTAL WEIGHT OF SAMPLE, 2237.5 GRAMS
(25 GRAM CAPACITY BOMB CALORIMETER)

Expt. No.	Air Dry Loss wt.%	Residual Moisture wt.%	Furnace Ash* wt.%	Bomb Ash* wt.%	Sulfur Content* wt.%
1029	8.535	5.966	34.458	32.512	0.219
1030	8.732	5.830	33.766	32.134	0.225
1031	--	--	--	30.079	0.238
Mean	8.634	5.898	34.112	31.575	0.227
Std. Dev.	0.139	0.096	0.489	1.309	0.010
% Std. Dev.	1.615%	1.633%	1.435%	4.147%	4.272%

*(Data are reduced to a dry basis.)

TABLE 47. DETERMINATION OF AIR DRY LOSS, RESIDUAL MOISTURE, ASH, AND SULFUR CONTENT

NEW CASTLE COUNTY, DELAWARE MSW, PROCESSED

THROUGH THE BUREAU OF MINES FACILITY, COLLEGE PARK, MD.

CYCLONE NO. 2, SECONDARY SHRED, 2 INCH PIECES

TOTAL WEIGHT OF SAMPLE, 2672.5 GRAMS

(25 GRAM CAPACITY BOMB CALORIMETER)

Expt. No.	Air Dry Loss wt.%	Residual Moisture wt.%	Furnace Ash* wt.%	Bomb Ash* wt.%	Sulfur Content* wt.%
1018	8.316	5.429	8.238	7.133	0.124
1019	8.720	5.323	8.387	7.312	0.123
1020	8.471	--	--	7.637	0.127
Mean	8.502	5.376	8.313	7.361	0.125
Std. Dev.	0.204	0.075	0.106	0.256	0.002
% Std. Dev.	2.40%	1.398%	1.269%	3.471%	1.670%

*(Data are reduced to a dry basis.)

TABLE 48. DETERMINATION OF AIR DRY LOSS, RESIDUAL MOISTURE, ASH, AND SULFUR CONTENT

NEW CASTLE COUNTY, DELAWARE MSW, PROCESSED

THROUGH THE BUREAU OF MINES FACILITY, COLLEGE PARK, MD.

CYCLONE NO. 2, TROMMEL UNDERSIZE, +14 MESH

TOTAL WEIGHT OF SAMPLE, 2361.2 GRAMS

(25 GRAM CAPACITY BOMB CALORIMETER)

Expt. No.	Air Dry Loss wt.%	Residual Moisture wt.%	Furnace Ash* wt.%	Bomb Ash* wt.%	Sulfur Content* wt.%
1035	18.207	6.797	22.176	22.359	0.223
1037	--	6.704	23.867	22.215	0.216
1038	--	--	--	24.214	0.219
Mean	18.207	6.750	23.022	22.929	0.219
Std. Dev.	--	0.066	1.196	1.115	0.004
% Std. Dev.	--	0.977%	5.193%	4.862%	1.601%

*(Data are reduced to a dry basis.)

TABLE 49. DETERMINATION OF AIR DRY LOSS, RESIDUAL MOISTURE, ASH, AND SULFUR CONTENT

NEW CASTLE COUNTY, DELAWARE MSW, PROCESSED

THROUGH THE BUREAU OF MINES FACILITY, COLLEGE PARK, MD.

SECONDARY SHRED, CYLCONE NO. 3, AS-COLLECTED

TOTAL WEIGHT OF SAMPLE, 3540.4 GRAMS

(25 GRAM CAPACITY BOMB CALORIMETER)

Expt. No.	Air Dry Loss wt.%	Residual Moisture wt.%	Furnace Ash* wt.%	Bomb Ash* wt.%	Sulfur Content* wt.%
1023	15.766	5.212	9.182	9.516	0.121
1024	15.298	5.308	8.887	10.944	0.116
1025	--	--	--	7.751	0.121
Mean	15.532	5.260	9.034	9.404	0.119
Std. Dev.	0.331	0.068	0.208	1.599	0.003
% Std. Dev.	2.131%	1.288%	2.308%	17.009%	2.419%

*(Data are reduced to a dry basis.)

TABLE 50. DETERMINATION OF AIR DRY LOSS, RESIDUAL MOISTURE, ASH, AND SULFUR CONTENT

NEW CASTLE COUNTY, DELAWARE MSW, PROCESSED

THROUGH THE BUREAU OF MINES FACILITY, COLLEGE PARK, MD.

HEAVY ORGANIC COMBUSTIBLES

TOTAL WEIGHT OF SAMPLE, 3664.8 GRAMS

(25 GRAM CAPACITY BOMB CALORIMETER)

Expt. No.	Air Dry Loss wt.%	Residual Moisture wt.%	Furnace Ash* wt.%	Bomb Ash* wt.%	Sulfur Content* wt.%
1041	0.718	4.207	11.671	--	0.215
1042	--	4.117	10.192	--	0.196
1043	--	--	--	--	0.178
Mean	0.718	4.162	10.931	--	0.196
Std. Dev.	--	0.064	1.046	--	0.019
% Std. Dev.	--	1.527%	9.570%	--	9.424%

*(Data are reduced to a dry basis.)

TABLE 51. DETERMINATION OF AIR DRY LOSS, RESIDUAL MOISTURE, ASH, AND SULFUR CONTENT

NEW CASTLE COUNTY, DELAWARE MSW, PROCESSED

THROUGH THE BUREAU OF MINES FACILITY, COLLEGE PARK, MD.

ORGANIC WASTES, JIG OVERFLOW

TOTAL WEIGHT OF SAMPLE, 3202.1 GRAMS

(25 GRAM CAPACITY BOMB CALORIMETER)

Expt. No.	Air Dry Loss wt.%	Residual Moisture wt.%	Furnace Ash* wt.%	Bomb Ash* wt.%	Sulfur Content* wt.%
1046	0.784	4.560	11.415	--	0.145
1047	--	4.735	10.774	--	0.145
1048	--	--	--	--	0.151
Mean	0.784	4.648	11.094	--	0.147
Std. Dev.	--	0.123	0.453	--	0.003
% Std. Dev.	--	2.656%	4.081%	--	2.357%

*(Data are reduced to a dry basis.)

TABLE 52. CALORIFIC VALUES CALCULATED TO DIFFERENT BASES
NEW CASTLE COUNTY, DELAWARE MSW, PROCESSED
THROUGH THE BUREAU OF MINES FACILITY, COLLEGE PARK, MD.
CYCLONE NO. 1, SECONDARY SHRED, 2 INCH PIECES
(25 GRAM CAPACITY BOMB CALORIMETER)

	HHV1	HHV2	HHV3-F	HHV3-B
Expt. No.	$MJ\cdot kg^{-1}$ ($Btu\cdot lb^{-1}$)	$MJ\cdot kg^{-1}$ ($Btu\cdot lb^{-1}$)	$MJ\cdot kg^{-1}$ ($Btu\cdot lb^{-1}$)	$MJ\cdot kg^{-1}$ ($Btu\cdot lb^{-1}$)
1011	16.67 (7165)	19.25 (8278)	22.01 (9463)	21.84 (9388)
1014	16.71 (7185)	19.31 (8300)	22.07 (9488)	21.81 (9375)
1015	16.79 (7217)	19.39 (8337)	22.17 (9529)	21.77 (9360)
Mean	16.72 (7189)	19.32 (8305)	22.08 (9493)	21.80 (9374)
Std. Dev.	0.06 (26)	0.07 (30)	0.08 (33)	0.03 (14)
% Std. Dev.	0.36%	0.36%	0.35%	0.15%

$MJ\cdot kg^{-1}$ = 429.9226 $Btu\cdot lb^{-1}$ was used to calculate the $Btu\cdot lb^{-1}$ values presented in parenthesis.

TABLE 53. CALORIFIC VALUES CALCULATED TO DIFFERENT BASES
NEW CASTLE COUNTY, DELAWARE MSW, PROCESSED
THROUGH THE BUREAU OF MINES FACILITY, COLLEGE PARK, MD.
CYCLONE NO. 2, SECONDARY SHRED, 2 INCH PIECES
(25 GRAM CAPACITY BOMB CALORIMETER)

	HHV1	HHV2	HHV3-F	HHV3-B
Expt. No.	MJ·kg^{-1} (Btu·lb^{-1})	MJ·kg^{-1} (Btu·lb^{-1})	MJ·kg^{-1} (Btu·lb^{-1})	MJ·kg^{-1}(Btu·lb
1018	17.28 (7428)	19.96 (8580)	21.77 (9358)	21.49 (9239)
1019	17.32 (7447)	20.01 (8601)	21.82 (9382)	21.59 (9280)
1020	17.35 (7458)	20.04 (8614)	21.85 (9396)	21.69 (9326)
Mean	17.32 (7444)	20.00 (8598)	21.81 (9379)	21.59 (9282)
Std. Dev.	0.04 (15)	0.04 (17)	0.04 (19)	0.10 (44)
% Std. Dev.	0.20%	0.20%	0.20%	0.47%

MJ·kg^{-1} = 429.9226 Btu·lb^{-1} was used to calculate the Btu·lb^{-1} values presented in parenthesis.

TABLE 54. CALORIFIC VALUES CALCULATED TO DIFFERENT BASES
NEW CASTLE COUNTY, DELAWARE MSW, PROCESSED THROUGH
THE BUREAU OF MINES FACILITY, COLLEGE PARK, MD.
SECONDARY SHRED, CYLCONE NO. 3, AS-COLLECTED
(25 GRAM CAPACITY BOMB CALORIMETER)

	HHV1	HHV2	HHV3-F	HHV3-B
Expt. No.	$MJ \cdot kg^{-1}$ ($Btu \cdot lb^{-1}$)	$MJ \cdot kg^{-1}$ ($Btu \cdot lb^{-1}$)	$MJ \cdot kg^{-1}$ ($Btu \cdot lb^{-1}$)	$MJ \cdot kg^{-1}$ ($Btu \cdot lb^{-1}$)
1023	16.37 (7038)	20.46 (8795)	22.49 (9669)	22.61 (9720)
1024	16.33 (7020)	20.40 (8772)	22.43 (9644)	22.91 (9850)
1025	16.17 (6951)	20.20 (8686)	22.21 (9549)	21.90 (9416)
Mean	16.29 (7003)	20.35 (8751)	22.38 (9621)	22.47 (9662)
Std. Dev.	0.11 (46)	0.13 (57)	0.15 (63)	0.52 (223)
% Std. Dev.	0.66%	0.66%	0.66%	2.31%

$MJ \cdot kg^{-1}$ = 429.9226 $Btu \cdot lb^{-1}$ was used to calculate the $Btu \cdot lb^{-1}$ values presented in parenthesis.

TABLE 55. CALORIFIC VALUES CALCULATED TO DIFFERENT BASES
NEW CASTLE COUNTY, DELAWARE MSW, PROCESSED THROUGH
THE BUREAU OF MINES FACILITY, COLLEGE PARK, MD.
CYCLONE NO. 1, TROMMEL UNDERSIZE, +14 MESH
(25 GRAM CAPACITY BOMB CALORIMETER)

	HHV1	HHV2	HHV3-F	HHV3-B
Expt. No.	MJ·kg^{-1} (Btu·lb^{-1})	MJ·kg^{-1} (Btu·lb^{-1})	MJ·kg^{-1} (Btu·lb^{-1})	MJ·kg^{-1} (Btu·lb
1029	12.03 (5171)	13.99 (6014)	21.23 (9127)	20.73 (8911)
1030	12.12 (5212)	14.10 (6062)	21.40 (9200)	20.78 (8932)
1031	12.44 (5350)	14.47 (6223)	21.97 (9445)	20.70 (8900)
Mean	12.20 (5244)	14.19 (6100)	21.53 (9257)	20.73 (8914)
Std. Dev.	0.22 (94)	0.25 (109)	0.39 (167)	0.04 (16)
% Std. Dev.	1.79%	1.79%	1.80%	0.18%

MJ·kg^{-1} = 429.9226 Btu·lb^{-1} was used to calculate the Btu·lb^{-1} values presented in parenthesis.

TABLE 56. CALORIFIC VALUES CALCULATED TO DIFFERENT BASES
NEW CASTLE COUNTY, DELAWARE MSW, PROCESSED
THROUGH THE BUREAU OF MINES FACILITY, COLLEGE PARK, MD.
CYCLONE NO. 2, TROMMEL UNDERSIZE, +14 MESH
(25 GRAM CAPACITY BOMB CALORIMETER)

	HHV1	HHV2	HHV3-F	HHV3-B
Expt. No.	$MJ \cdot kg^{-1}$ ($Btu \cdot lb^{-1}$)	$MJ \cdot kg^{-1}$ ($Btu \cdot lb^{-1}$)	$MJ \cdot kg^{-1}$ ($Btu \cdot lb^{-1}$)	$MJ \cdot kg^{-1}$ ($Btu \cdot lb^{-1}$)
1035	12.56 (5399)	16.46 (7078)	21.39 (9195)	21.20 (9116)
1037	12.51 (5379)	16.40 (7053)	21.31 (9162)	21.09 (9067)
1038	12.32 (5298)	16.16 (6946)	20.99 (9024)	21.32 (9165)
Mean	12.46 (5359)	16.34 (7026)	21.23 (9127)	21.20 (9116)
Std. Dev.	0.12 (53)	0.16 (70)	0.21 (91)	0.11 (49)
% Std. Dev.	1.00%	1.00%	0.99%	0.54%

$MJ \cdot kg^{-1} = 429.9226\ Btu \cdot lb^{-1}$ was used to calculate the $Btu \cdot lb^{-1}$ values presented in parenthesis.

TABLE 57. CALORIFIC VALUES CALCULATED TO DIFFERENT BASES
NEW CASTLE COUNTY, DELAWARE MSW, PROCESSED
THROUGH THE BUREAU OF MINES FACILITY, COLLEGE PARK, MD.
HEAVY ORGANIC COMBUSTIBLES
(25 GRAM CAPACITY BOMB CALORIMETER)

	HHV1	HHV2	HHV3-F	HHV3-B
Expt. No.	$MJ\cdot kg^{-1}$ ($Btu\cdot lb^{-1}$)	$MJ\cdot kg^{-1}$ ($Btu\cdot lb^{-1}$)	$MJ\cdot kg^{-1}$ ($Btu\cdot lb^{-1}$)	$MJ\cdot kg^{-1}$ ($Btu\cdot lb^{-1}$)
1041	20.90 (8986)	21.97 (9444)	24.66 (10603)	--
1042	20.81 (8945)	21.87 (9401)	24.55 (10556)	--
1043	20.85 (8965)	21.92 (9422)	24.61 (10579)	--
Mean	20.85 (8965)	21.92 (9422)	24.61 (10579)	--
Std. Dev.	0.05 (20)	0.05 (22)	0.05 (24)	--
% Std. Dev.	0.23%	0.23%	0.22%	--

$MJ\cdot kg^{-1}$ = 429.9226 $Btu\cdot lb^{-1}$ was used to calculate the $Btu\cdot lb^{-1}$ values presented in parenthesis.

TABLE 58. CALORIFIC VALUES CALCULATED TO DIFFERENT BASES
NEW CASTLE COUNTY, DELAWARE MSW, PROCESSED
THROUGH THE BUREAU OF MINES FACILITY, COLLEGE PARK, MD.
ORGANIC WASTES, JIG OVERFLOW
(25 GRAM CAPACITY BOMB CALORIMETER)

	HHV1	HHV2	HHV3-F	HHV3-B
Expt. No.	$MJ \cdot kg^{-1}$ ($Btu \cdot lb^{-1}$)	$MJ \cdot kg^{-1}$ ($Btu \cdot lb^{-1}$)	$MJ \cdot kg^{-1}$ ($Btu \cdot lb^{-1}$)	$MJ \cdot kg^{-1}$ ($Btu \cdot lb^{-1}$)
1046	19.29 (8291)	20.39 (8764)	22.93 (9858)	--
1047	19.73 (8480)	20.85 (8964)	23.45 (10083)	--
1048	19.71 (8476)	20.84 (8959)	23.44 (10077)	--
Mean	19.57 (8416)	20.69 (8896)	23.27 (10006)	--
Std. Dev.	0.25 (108)	0.27 (114)	0.30 (128)	--
% Std. Dev.	1.28%	1.28%	1.28%	--

$MJ \cdot kg^{-1}$ = 429.9226 $Btu \cdot lb^{-1}$ was used to calculate the $Btu \cdot lb^{-1}$ values presented in parenthesis.

TABLE 59. DETERMINATION OF CALORIFIC VALUES
TELEDYNE NATIONAL RDF, SINGLE STAGE MOISTURE
(2.5 GRAM CAPACITY BOMB CALORIMETER)

Expt. No.	1272	1273	1274	1275
E-cal/J$\cdot$K^{-1}	14561.90	14561.91	14561.99	14562.01
ΔT-corr/K	3.026075	3.029582	3.147106	3.172096
Q-total/kJ	44.065	44.116	45.828	46.192
q-ign/kJ	0.001	0.001	0.001	0.002
q-Fe/kJ	0.000	0.000	0.000	0.000
q-HNO_3/kJ	0.074	0.078	0.083	0.073
q-H_2SO_4/kJ	0.025	0.026	0.026	0.025
Q-RDF/kJ	43.965	44.011	45.718	46.092
m-RDF/g	2.28167	2.28249	2.36948	2.38905
HHV-AD/MJ$\cdot$kg^{-1}	--	--	--	--
/(Btu$\cdot$lb^{-1})	--	--	--	--
HHV2/MJ$\cdot$kg^{-1}	19.27	19.28	19.29	19.29
/(Btu$\cdot$lb^{-1})	(8284)	(8290)	(8295)	(8295)

MJ$\cdot$kg^{-1} = 429.9226 Btu$\cdot$lb^{-1} was used to calculate the Btu$\cdot$lb^{-1} values presented in parenthesis.

TABLE 60. DETERMINATION OF RESIDUAL MOISTURE, ASH, AND SULFUR CONTENT

TELEDYNE NATIONAL RDF, SINGLE STAGE MOISTURE

(2.5 GRAM CAPACITY BOMB CALORIMETER)

Expt. No.	Residual Moisture wt.%	Furnace Ash* wt.%	Bomb Ash* wt.%	Sulfur Content* wt.%
1272	--	7.100	6.137	0.1900
1273	--	7.080	6.085	0.1933
1274	--	7.054	6.109	0.1891
1275	--	7.059	5.845	0.1801
Mean	--	7.073	6.044	0.1881
Std. Dev.	--	0.021	0.134	0.0056
% Std. Dev.	--	0.298%	2.223%	3.001%

*(Data are reduced to a dry basis.)

TABLE 61. CALORIFIC VALUES CALCULATED TO DIFFERENT BASES

TELEDYNE NATIONAL RDF, SINGLE STAGE MOISTURE

(2.5 GRAM CAPACITY BOMB CALORIMETER)

	HHV-AD	HHV2	HHV3-F	HHV3-B
Expt. No.	MJ·kg^{-1} (Btu·lb^{-1})	MJ·kg^{-1} (Btu·lb^{-1})	MJ·kg^{-1} (Btu·lb^{-1})	MJ·kg^{-1} (Btu·lb^{-1})
1272	--	19.27 (8284)	20.74 (8917)	20.53 (8826)
1273	--	19.28 (8290)	20.75 (8921)	20.53 (8827)
1274	--	19.29 (8295)	20.76 (8925)	20.55 (8835)
1275	--	19.29 (8295)	20.76 (8925)	20.49 (8809)
Mean	--	19.28 (8291)	20.75 (8922)	20.53 (8824)
Std. Dev.	--	0.01 (5)	0.01 (4)	0.03 (11)
% Std. Dev.	--	0.05%	0.05%	0.12%

MJ·kg^{-1} = 429.9226 Btu·lb^{-1} was used to calculate the Btu·lb^{-1} values presented in parenthesis.

TABLE 62. DETERMINATION OF CALORIFIC VALUES

TELEDYNE NATIONAL RDF, TWO STAGE MOISTURE

(2.5 GRAM CAPACITY BOMB CALORIMETER)

Expt. No.	1268	1269	1270	1271
E-cal/$J \cdot K^{-1}$	14562.13	14562.12	14562.00	14562.03
ΔT-corr/K	3.145622	3.119644	2.988314	3.015041
Q-total/kJ	45.807	45.429	43.516	43.905
q-ign/kJ	0.001	0.001	0.001	0.001
q-Fe/kJ	0.000	0.000	0.000	0.000
q-HNO_3/kJ	0.082	0.081	0.081	0.083
q-H_2SO_4/kJ	0.030	0.032	0.030	0.030
Q-RDF/kJ	45.694	45.314	43.404	43.791
m-RDF/g	2.506865	2.483743	2.374997	2.398991
HHV-AD/$MJ \cdot kg^{-1}$ /($Btu \cdot lb^{-1}$)	18.23 (7836)	18.24 (7844)	18.28 (7857)	18.25 (7848)

$MJ \cdot kg^{-1}$ = 429.9226 $Btu \cdot lb^{-1}$ was used to calculate the $Btu \cdot lb^{-1}$ values presented in parenthesis.

TABLE 63. DETERMINATION OF RESIDUAL MOISTURE, ASH, AND SULFUR CONTENT

TELEDYNE NATIONAL RDF, TWO STAGE MOISTURE

(2.5 GRAM CAPACITY BOMB CALORIMETER)

Expt. No.	Residual Moisture wt.%	Furnace Ash* wt.%	Bomb Ash* wt.%	Sulfur Content* wt.%
1268	6.251	7.288	6.393	0.2170
1269	6.212	7.260	6.599	0.2345
1270	6.102	7.284	6.435	0.2330
1271	6.116	7.371	6.332	0.2282
Mean	6.170	7.301	6.440	0.2282
Std. Dev.	0.073	0.048	0.114	0.0079
% Std. Dev.	1.179%	0.664%	1.775%	3.471

*(Data are reduced to a dry basis.)

TABLE 64. CALORIFIC VALUES CALCULATED TO DIFFERENT BASES

TELEDYNE NATIONAL RDF, TWO STAGE MOISTURE

(2.5 GRAM CAPACITY BOMB CALORIMETER)

	HHV-AD	HHV2	HHV3-F	HHV3-B
Expt. No.	$MJ\cdot kg^{-1}$ $(Btu\cdot lb^{-1})$	$MJ\cdot kg^{-1}$ $(Btu\cdot lb^{-1})$	$MJ\cdot kg^{-1}$ $(Btu\cdot lb^{-1})$	$MJ\cdot kg^{-1}$ $(Btu\cdot lb^{-1})$
1268	18.23 (7836)	19.44 (8359)	20.97 (9016)	20.77 (8930)
1269	18.24 (7844)	19.45 (8363)	20.98 (9018)	20.83 (8954)
1270	18.28 (7857)	19.46 (8368)	20.99 (9025)	20.80 (8943)
1271	18.25 (7848)	19.44 (8359)	20.99 (9024)	20.76 (8924)
Mean	18.25 (7846)	19.45 (8362)	20.98 (9021)	20.79 (8938)
Std. Dev.	0.02 (9)	0.01 (4)	0.01 (4)	0.03 (13)
% Std. Dev.	0.11%	0.05%	0.05%	0.15%

$MJ\cdot kg^{-1}$ = 429.9226 $Btu\cdot lb^{-1}$ was used to calculate the $Btu\cdot lb^{-1}$ values presented in parenthesis.

TABLE 65. DETERMINATION OF CALORIFIC VALUES
STEAM SIZE REDUCTION RDF
(2.5 GRAM CAPACITY BOMB CALORIMETER

Expt. No.	1258	1263	1264
E-cal/J·K^{-1}	14562.00	14562.27	14562.26
ΔT-corr/K	2.791100	2.970305	2.991348
Q-total/kJ	40.644	43.254	43.561
q-ign/kJ	0.001	0.001	0.001
q-Fe/kJ	0.000	0.000	0.000
q-HNO_3/kJ	0.064	0.065	0.070
q-H_2SO_4/kJ	0.023	0.028	0.026
Q-RDF/kJ	40.556	43.161	43.464
m-RDF/g	2.457883	2.635031	2.634345
HHV-AD/MJ·kg^{-1}	16.50	16.38	16.50
/(Btu·lb^{-1})	(7094)	(7042)	(7093)

MJ·kg^{-1} = 429.9226 Btu·lb^{-1} was used to calculate the Btu·lb^{-1} values presented in parenthesis.

TABLE 66. DETERMINATION OF RESIDUAL MOISTURE, ASH, AND SULFUR CONTENT

STEAM SIZE REDUCTION RDF

(2.5 GRAM CAPACITY BOMB CALORIMETER)

Expt. No.	Residual Moisture wt.%	Furnace Ash* wt.%	Bomb Ash* wt.%	Sulfur Content* wt.%
1258	4.159	16.64	16.34	0.1673
1263	4.136	16.46	17.39	0.1890
1264	--	--	16.74	0.1752
Mean	4.148	16.55	16.82	0.1772
Std. Dev.	0.016	0.125	0.53	0.011
% Std. Dev.	0.39%	0.758%	3.15%	6.20%

*(Data are reduced to a dry basis.)

TABLE 67. CALORIFIC VALUES CALCULATED TO DIFFERENT BASES

STEAM SIZE REDUCTION RDF

(2.5 GRAM CAPACITY BOMB CALORIMETER)

	HHV-AD	HHV2	HHV3-F	HHV3-B
Expt. No.	$MJ\cdot kg^{-1}$ $(Btu\cdot lb^{-1})$	$MJ\cdot kg^{-1}$ $(Btu\cdot lb^{-1})$	$MJ\cdot kg^{-1}$ $(Btu\cdot lb^{-1})$	$MJ\cdot kg^{-1}$ $(Btu\cdot lb^{-1})$
1258	16.50 (7094)	17.21 (7401)	20.63 (8868)	20.58 (8846)
1263	16.38 (7042)	17.09 (7347)	20.48 (8803)	20.68 (8892)
1264	16.50 (7093)	17.21 (7400)	20.63 (8867)	20.67 (8888)
Mean	16.46 (7076)	17.17 (7383)	20.58 (8846)	20.64 (8875)
Std. Dev.	0.07 (30)	0.07 (31)	0.09 (37)	0.06 (25)
% Std. Dev.	0.42%	0.42%	0.42%	0.29%

$MJ\cdot kg^{-1}$ = 429.9226 $Btu\cdot lb^{-1}$ was used to calculate the $Btu\cdot lb^{-1}$ values presented in parentehsis.

TABLE 68. DETERMINATION OF CALORIFIC VALUES

NCRR STORAGE PIT RDF, CHARRED STORAGE SAMPLE

(25 GRAM CAPACITY BOMB CALORIMETER)

Expt. No.	1003	1004	1006
$E\text{-cal}/J\cdot K^{-1}$	86208.25	86197.94	86210.35
$\Delta T\text{-corr}/K$	4.657703	2.858432	4.788010
Q-total/kJ	401.532	243.391	412.776
q-ign/kJ	0.002	0.001.	0.002
q-Fe/kJ	0.000	0.000	0.000
$q\text{-}HNO_3/kJ$	0.433	0.228	0.777
$q\text{-}H_2SO_4/kJ$	0.334	0.262	0.317
Q-RDF/kJ	400.764	245.900	411.680
m-RDF/g	25.0601	15.1636	25.7628
$HHV\text{-}AD/MJ\cdot kg^{-1}$	15.99	16.22	15.98
$/(Btu\cdot lb^{-1})$	(6875)	(6972)	(6870)

$MJ\cdot kg^{-1} = 429.9226\ Btu\cdot lb^{-1}$ was used to calculate the $Btu\cdot lb^{-1}$ values presented in parenthesis.

TABLE 69. DETERMINATION OF RESIDUAL MOISTURE, ASH, AND SULFUR CONTENT

NCRR STORAGE PIT RDF, CHARRED STORAGE SAMPLE

(25 GRAM CAPACITY BOMB CALORIMETER)

Expt. No.	Residual Moisture wt.%	Furnace Ash* wt.%	Bomb Ash* wt.%	Sulfur Content* wt.%
1003	2.618 (pellet)	30.915	24.93	0.232
1004	1.900 (loose)	31.1383	22.94	0.301
1006	--	--	29.68	0.216
Mean	2.259	31.027	25.85	0.250
Std. Dev.	0.508	0.158	3.46	0.045
% Std. Dev.	22.48%	0.509%	13.40%	18.09%

*(Data are reduced to a dry basis.)

TABLE 70. CALORIFIC VALUES CALCULATED TO DIFFERENT BASES

NCRR STORAGE PIT RDF, CHARRED STORAGE SAMPLE

(25 GRAM CAPACITY BOMB CALORIMETER)

	HHV-AD	HHV2	HHV3-F	HHV3-B
Expt. No.	$MJ\cdot kg^{-1}$ ($Btu\cdot lb^{-1}$)	$MJ\cdot kg^{-1}$ ($Btu\cdot lb^{-1}$)	$MJ\cdot kg^{-1}$ ($Btu\cdot lb^{-1}$)	$MJ\cdot kg^{-1}$ ($Btu\cdot lb^{-1}$)
1003	15.99 (6875)	16.30 (7009)	23.67 (10178)	21.72 (9336)
1004	16.22 (6972)	16.53 (7107)	24.01 (10321)	21.45 (9222)
1006	15.98 (6870)	16.41 (7055)	23.75 (10213)	23.33 (10032)
Mean	16.06 (6906)	16.41 (7057)	23.81 (10237)	22.17 (9530)
Std. Dev.	0.13 (58)	0.11 (49)	0.17 (75)	1.02 (438)
% Std. Dev.	0.83%	0.69%	0.73%	4.60%

$MJ\cdot kg^{-1}$ = 429.9226 $Btu\cdot lb^{-1}$ was used to calculate the $Btu\cdot lb^{-1}$ values presented in parenthesis.

TABLE 71. DETERMINATION OF CALORIFIC VALUES

NCRR (LOW ASH) RDF SAMPLE

(2.5 GRAM CAPACITY BOMB CALORIMETER)

Expt. No.	1265	1266	1267
E-cal/$J \cdot K^{-1}$	14562.01	14561.99	14562.04
ΔT-corr/K	2.574470	2.545666	2.598256
Q-total/kJ	37.489	37.070	37.836
q-ign/kJ	0.001	0.001	0.001
q-Fe/kJ	0.000	0.000	0.000
q-HNO_3/kJ	0.057	0.054	0.054
q-H_2SO_4/kJ	0.012	0.013	0.013
Q-RDF/kJ	37.420	37.002	37.768
m-RDF/g	2.382203	2.359875	2.417902
HHV-AD/$MJ \cdot kg^{-1}$	15.71	15.68	15.62
/($Btu \cdot lb^{-1}$)	(6753)	(6741)	(6715)

$MJ \cdot kg^{-1} = 429.9226\ Btu \cdot lb^{-1}$ was used to calculate the $Btu \cdot lb^{-1}$ values presented in parenthesis.

TABLE 72. DETERMINATION OF RESIDUAL MOISTURE, ASH, AND SULFUR CONTENT

NCRR (LOW ASH) RDF SAMPLE

(2.5 GRAM CAPACITY BOMB CALORIMETER)

Expt. No.	Residual Moisture wt.%	Furnace Ash* wt.%	Bomb Ash* wt.%	Sulfur Content* wt.%
1265	5.295	11.781	11.256	0.0881
1266	5.235	11.824	11.454	0.0995
1267	--	--	11.148	0.0961
Mean	5.265	11.803	11.286	0.0946
Std. Dev.	0.042	0.030	0.155	0.0059
% Std. Dev.	0.806%	0.258%	1.375%	6.189%

*(Data are reduced to a dry basis.)

TABLE 73. CALORIFIC VALUES CALCULATED TO DIFFERENT BASES
NCRR (LOW ASH) RDF SAMPLE
(2.5 GRAM CAPACITY BOMB CALORIMETER)

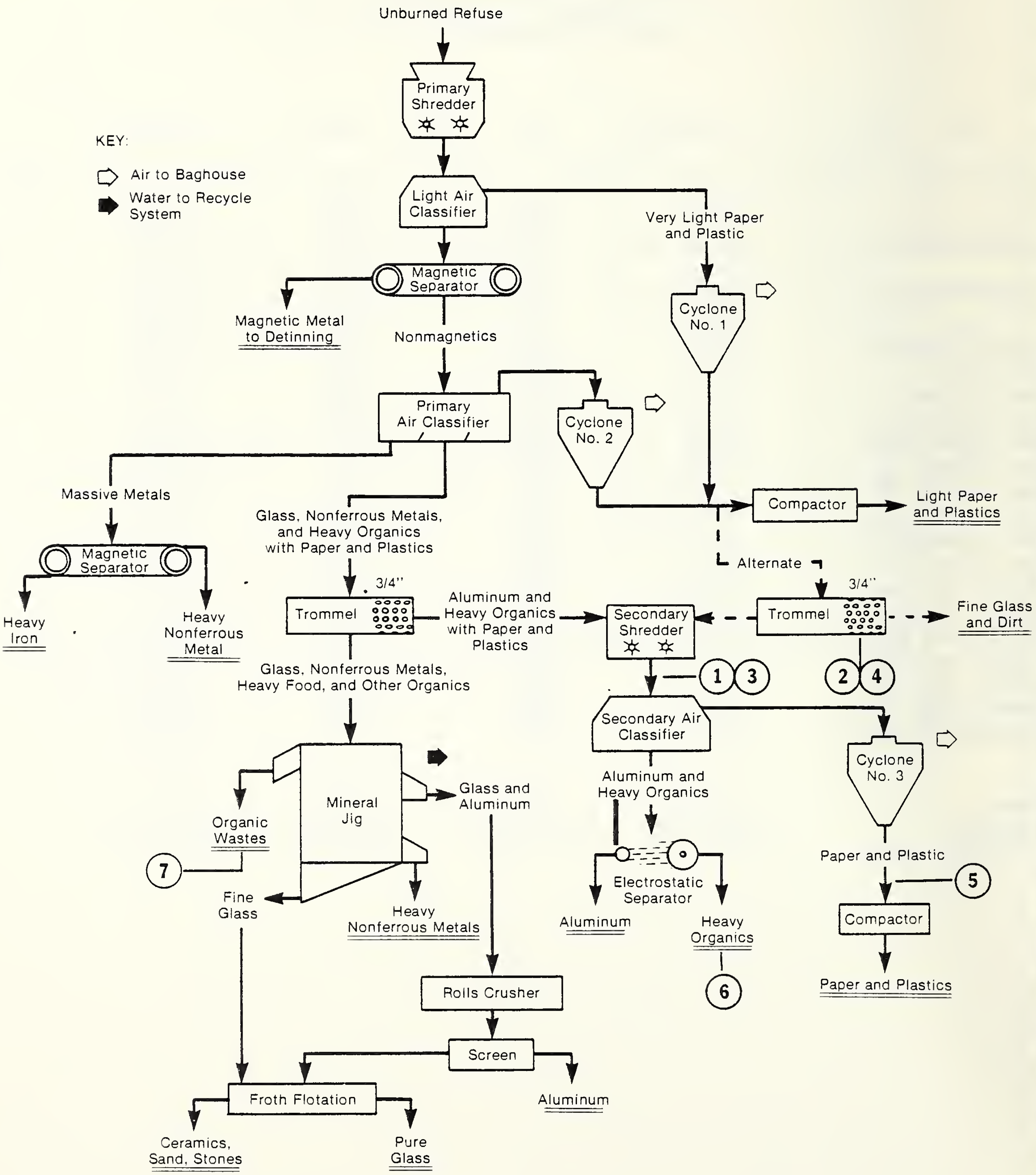
	HHV-AD	HHV2	HHV3-F	HHV3-B
Expt. No.	$MJ \cdot kg^{-1}$ $(Btu \cdot lb^{-1})$	$MJ \cdot kg^{-1}$ $(Btu \cdot lb^{-1})$	$MJ \cdot kg^{-1}$ $(Btu \cdot lb^{-1})$	$MJ \cdot kg^{-1}$ $(Btu \cdot lb^{-1})$
1265	15.71 (6753)	16.58 (7129)	18.80 (8082)	18.68 (8033)
1266	15.68 (6741)	16.55 (7116)	18.77 (8068)	18.69 (8036)
1267	15.62 (6715)	16.49 (7089)	18.69 (8037)	18.56 (7978)
Mean	15.67 (6736)	16.54 (7111)	18.75 (8062)	18.64 (8016)
Std. Dev.	0.05 (19)	0.05 (20)	0.05 (23)	0.08 (33)
% Std. Dev.	0.29%	0.029%	0.29%	0.41%

$MJ \cdot kg^{-1} = 429.9226\ Btu \cdot lb^{-1}$ was used to calculate the $Btu \cdot lb^{-1}$ values presented in parenthesis.

TABLE 74. SUMMARY OF MEAN CALORIFIC VALUES OBTAINED FOR MEASUREMENTS ON RDF IN BTU·LB^{-1}

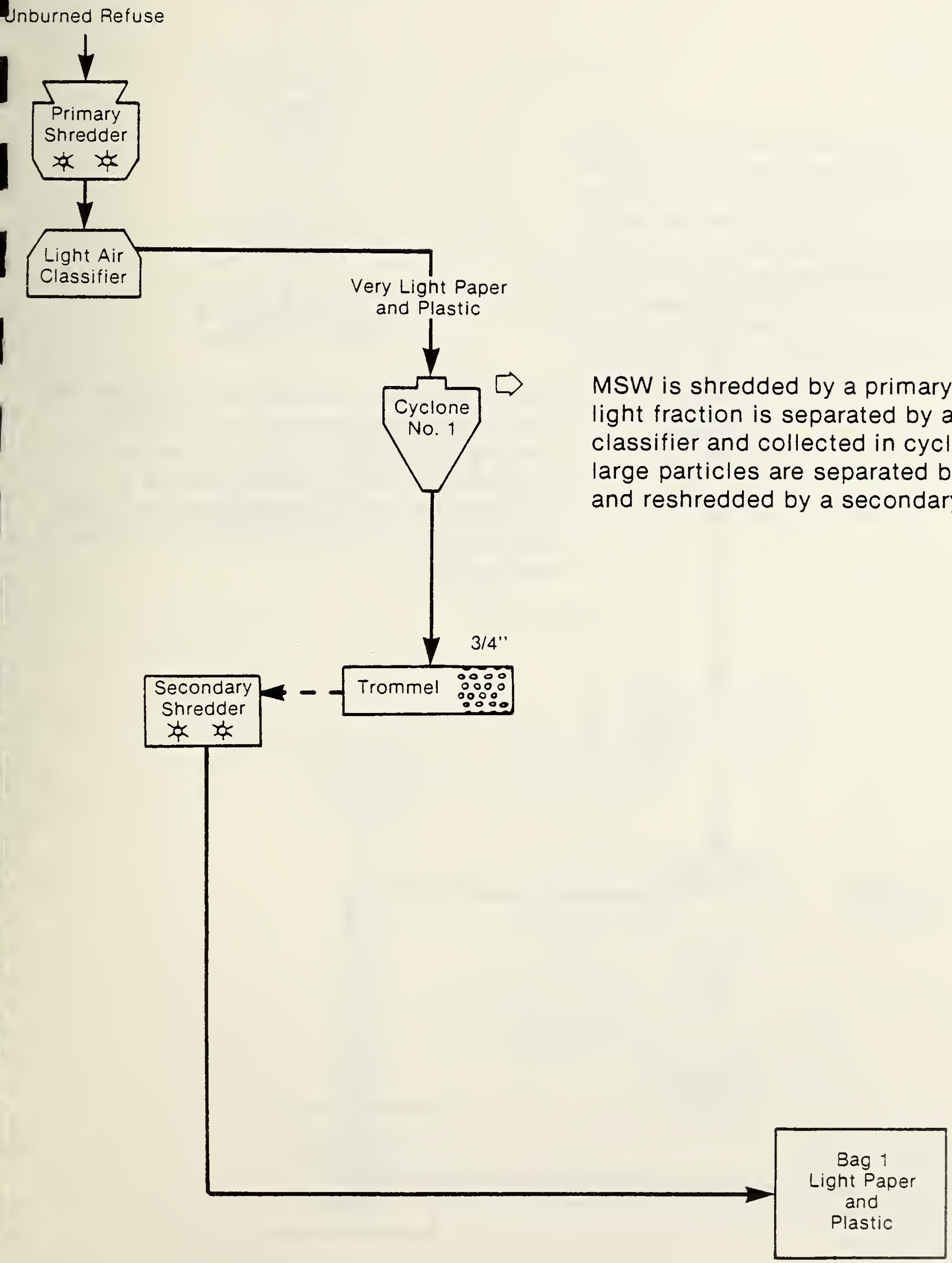
RDF	HHV-AD	HHV2	HHV3-F	HHV3-B
ASTM - RR1	7052	7426	9796	9716
	6947	7256	9570	9582
ASTM - RR2	7419	7900	9253	9126
	7156	7627	9535	9396
ASTM - RR3	6831	7148	9170	8998
	6962	7313	9438	9255
Teledyne Nat.	8352	8797	10526	10391
ECO FUEL II	8072	8205	9450	9304
Americology	6743	7157	9882	9751
Bur. Mines -1	7189	8305	9493	9394
Bur. Mines -2	7444	8598	9379	9282
Bur. Mines -3	7003	8751	9621	9662
Bur. Mines -4	5244	6100	9257	8914
Bur. Mines -5	5359	7026	9127	9116
Bur. Mines -6	8965	9422	10579	----
Bur. Mines -7	8416	8896	10006	----
NCRR - Low Ash	6736	7111	8062	8016
NCRR - Char	6906	7057	10237	9530
Steam Size Red.	7076	7383	8846	8875
Teled. Nat.Sgl.	----	8291	8922	8824
Teled. Nat. Dbl.	7846	8362	9021	8938
Americology Grab	6498	6607	9392	9236
Americology Rep.	6609	6971	9525	9693
Mean	7124	7726	9482	9285
Std.Dev. (% Std. Dev.)	876(12.3%)	841(10.9%)	553(5.3%)	474(5.1%)

Figure 1 U.S. Bureau of Mines, College Park, Md., Resource Recovery Facility Overall Flow-Diagram for Processing MSW



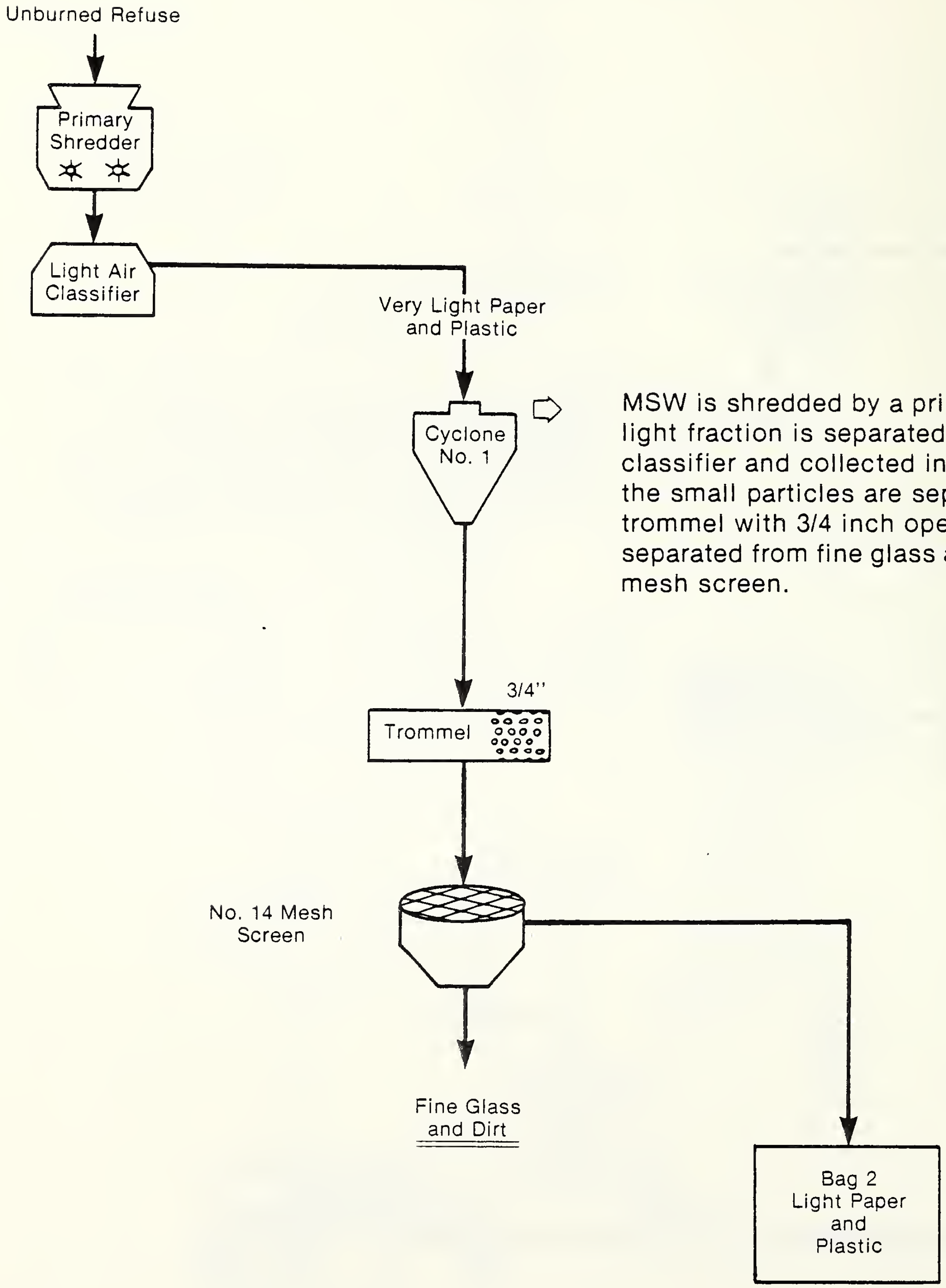
(Circled numbers indicate sample collection points)

Figure 2 U.S. Bureau of Mines, College Park, Md.
Resource Recovery Facility



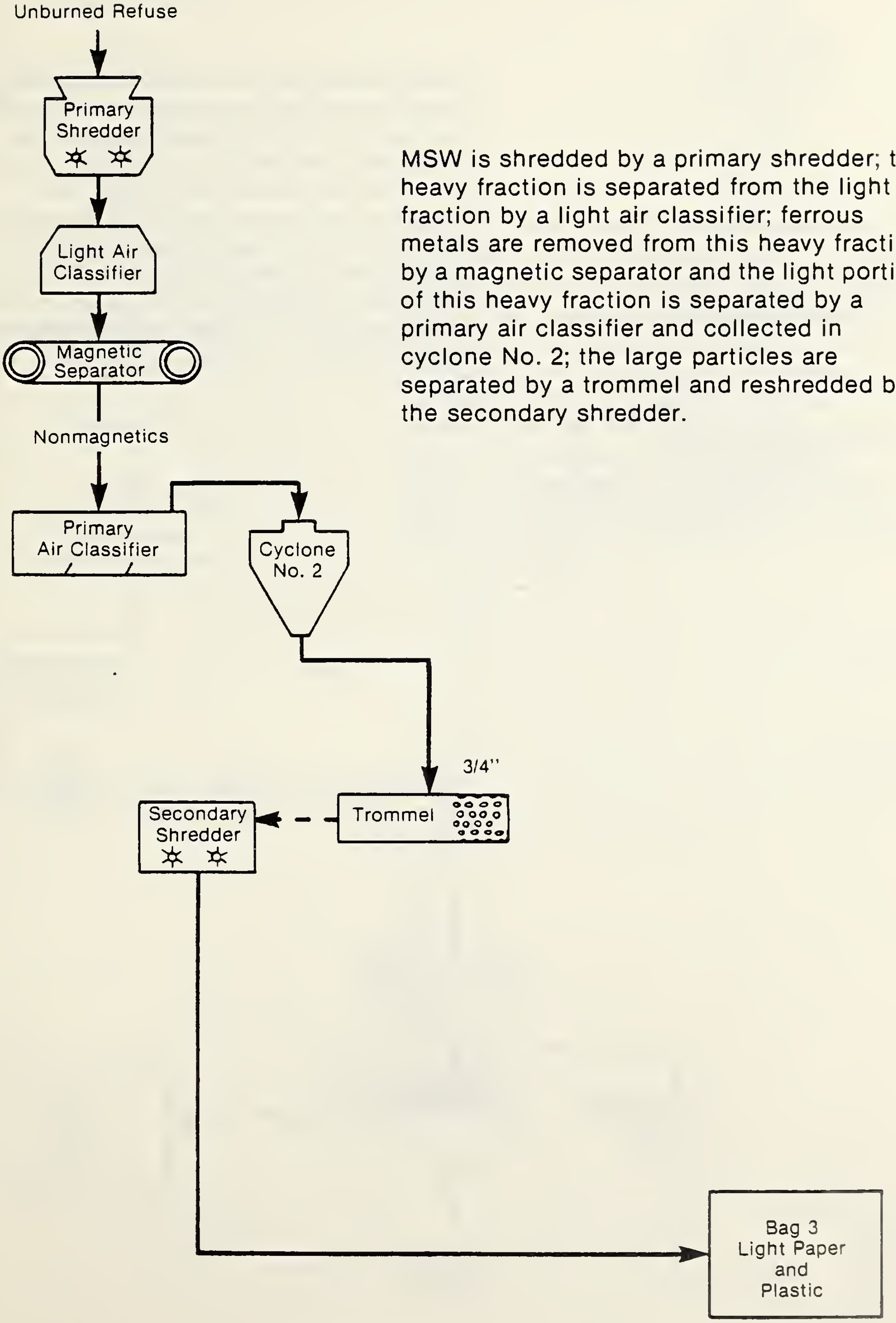
MSW is shredded by a primary shredder; the light fraction is separated by a light air classifier and collected in cyclone No. 1; the large particles are separated by a trommel and reshredded by a secondary shredder.

Figure 3 U.S. Bureau of Mines, College Park, Md. Resource Recovery Facility



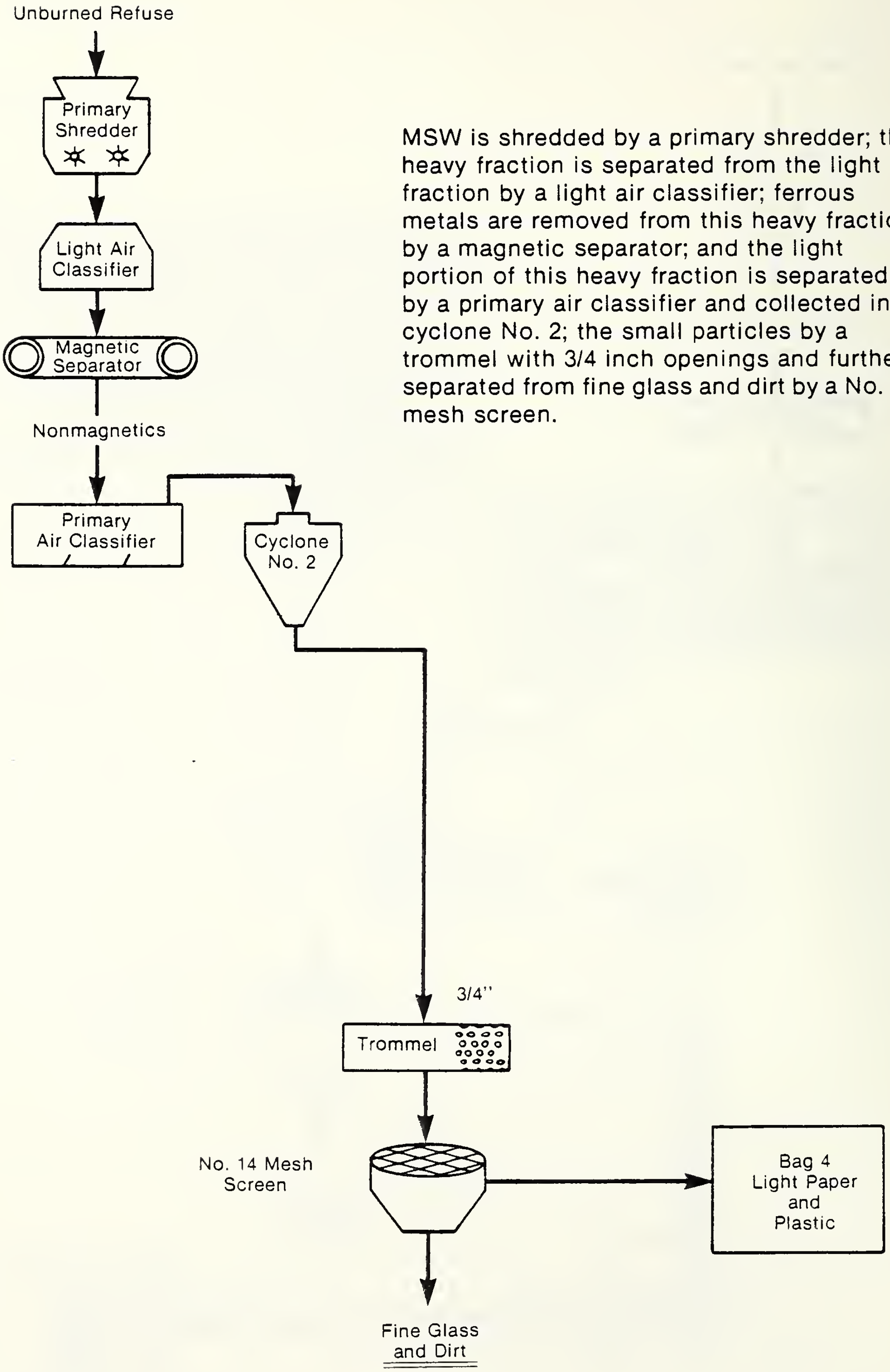
MSW is shredded by a primary shredder; the light fraction is separated by a light air classifier and collected in cyclone No. 1; the small particles are separated by a trommel with 3/4 inch openings and further separated from fine glass and dirt by a No. 14 mesh screen.

Figure 4 U.S. Bureau of Mines, College Park, Md.
Resource Recovery Facility



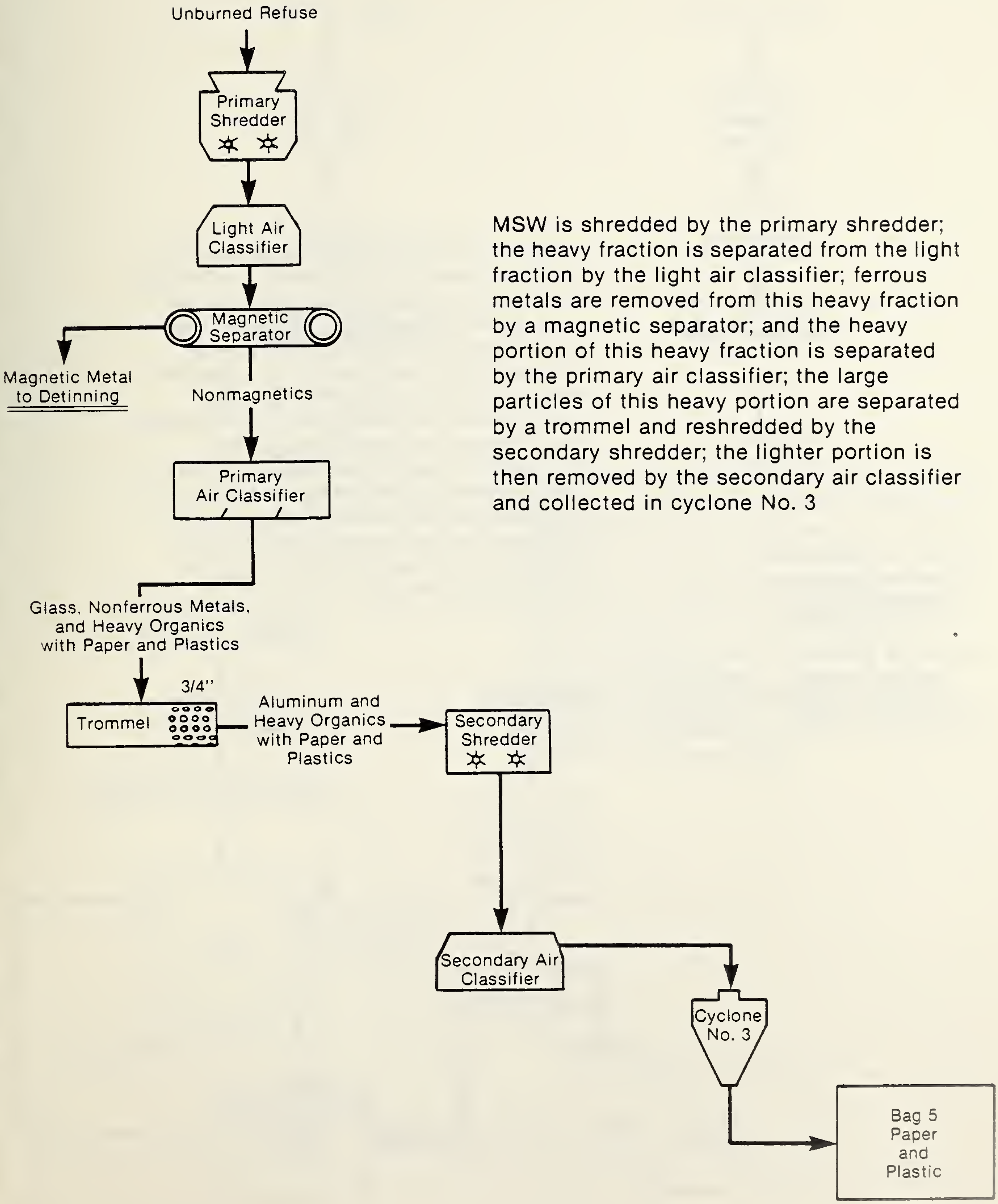
MSW is shredded by a primary shredder; the heavy fraction is separated from the light fraction by a light air classifier; ferrous metals are removed from this heavy fraction by a magnetic separator and the light portion of this heavy fraction is separated by a primary air classifier and collected in cyclone No. 2; the large particles are separated by a trommel and reshredded by the secondary shredder.

Figure 5 U.S. Bureau of Mines, College Park, Md. Resource Recovery Facility



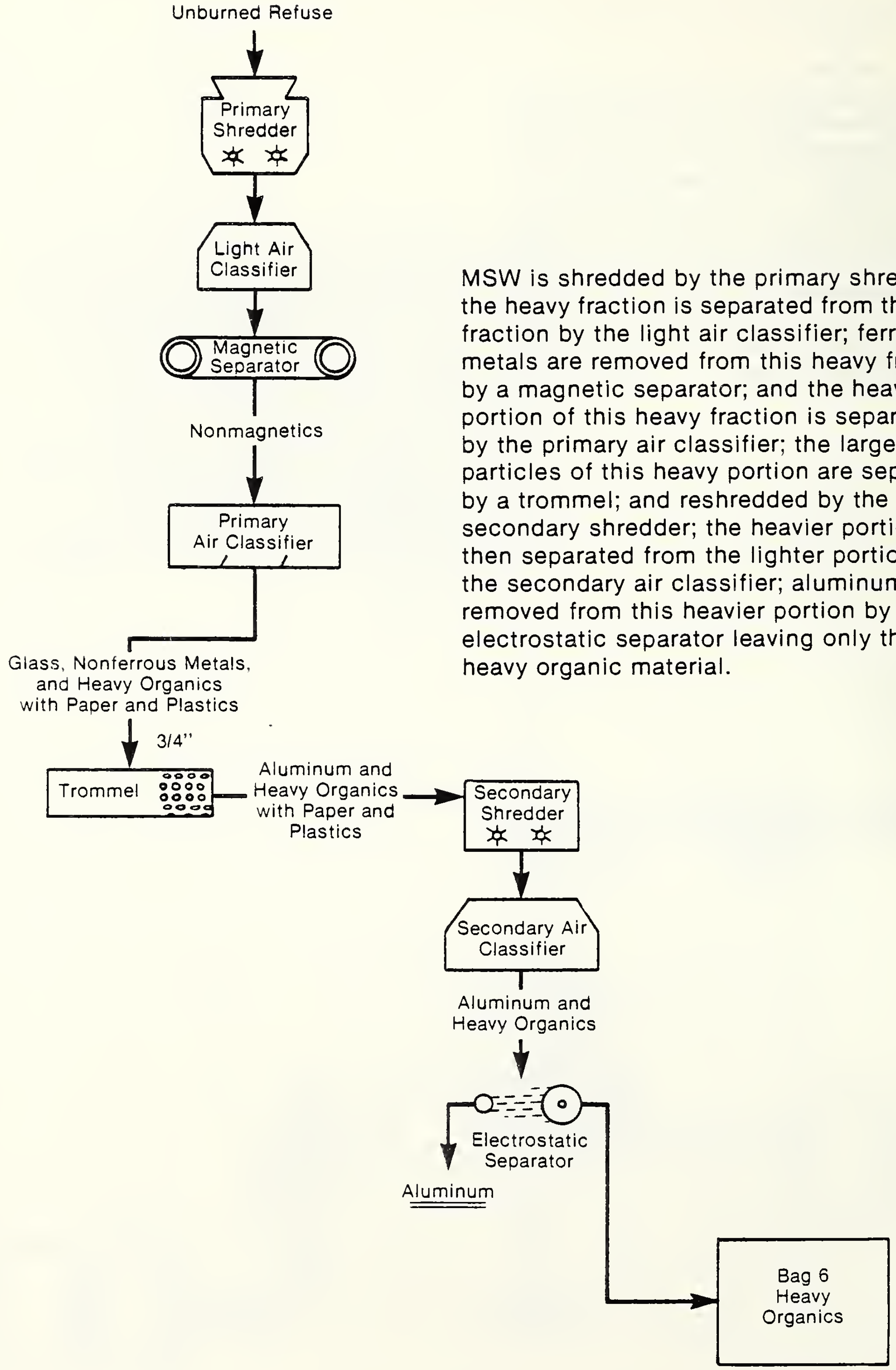
MSW is shredded by a primary shredder; the heavy fraction is separated from the light fraction by a light air classifier; ferrous metals are removed from this heavy fraction by a magnetic separator; and the light portion of this heavy fraction is separated by a primary air classifier and collected in cyclone No. 2; the small particles by a trommel with 3/4 inch openings and further separated from fine glass and dirt by a No. 14 mesh screen.

Figure 6 U.S. Bureau of Mines, College Park, Md.
Resource Recovery Facility



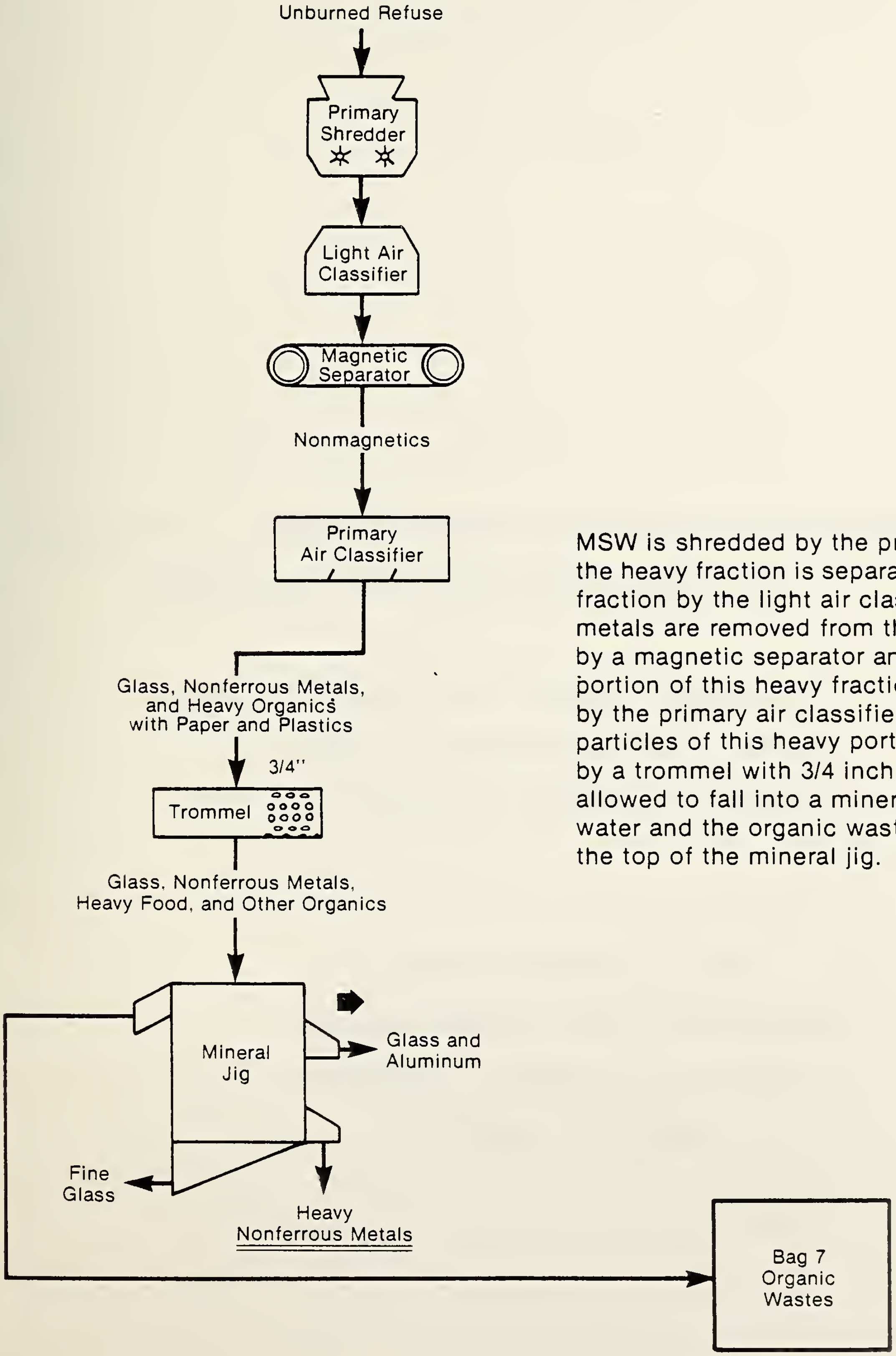
MSW is shredded by the primary shredder; the heavy fraction is separated from the light fraction by the light air classifier; ferrous metals are removed from this heavy fraction by a magnetic separator; and the heavy portion of this heavy fraction is separated by the primary air classifier; the large particles of this heavy portion are separated by a trommel and reshredded by the secondary shredder; the lighter portion is then removed by the secondary air classifier and collected in cyclone No. 3

Figure 7 U.S. Bureau of Mines, College Park, Md.
Resource Recovery Facility



MSW is shredded by the primary shredder; the heavy fraction is separated from the light fraction by the light air classifier; ferrous metals are removed from this heavy fraction by a magnetic separator; and the heavy portion of this heavy fraction is separated by the primary air classifier; the large particles of this heavy portion are separated by a trommel; and reshredded by the secondary shredder; the heavier portion is then separated from the lighter portion by the secondary air classifier; aluminum is removed from this heavier portion by an electrostatic separator leaving only the heavy organic material.

Figure 8 U.S. Bureau of Mines, College Park, Md. Resource Recovery Facility



MSW is shredded by the primary shredder; the heavy fraction is separated from the light fraction by the light air classifier; ferrous metals are removed from this heavy fraction by a magnetic separator and the heavier portion of this heavy fraction is separated by the primary air classifier; the small particles of this heavy portion are separated by a trommel with 3/4 inch openings and allowed to fall into a mineral jig filled with water and the organic wastes are floated off the top of the mineral jig.

APPENDIX A

EVALUATION OF DATA ON HIGHER HEATING VALUES DETERMINED DURING ASTM ROUND ROBIN TESTING OF RDF-3

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Evaluation of Data on Higher Heating Values Determined During ASTM Round Robin Testing of RDF-3

E. S. Domalski and S. Abramowitz

ABSTRACT

The potential application of RDF-3 as an alternative or supplemental fuel is dependent upon its acceptance as an article of commerce. ASTM Committee E-38 on Resource Recovery and its Subcommittee on Energy E-38.01 have been actively engaged in the development of consensus standards for this purpose since April 1974. Standard procedures for the characterization of RDF-3 are being developed. These procedures are based on those ASTM methods used in coal analysis. The procedures developed will insure a meaningful purchase - sales relationship between the buyer and seller. A variety of chemical and physical test procedures were studied by as many as 12 laboratories. Currently 20 editorial draft standards have been prepared and are being studied by the committee membership.

The National Bureau of Standards in cooperation with ASTM subcommittee E38.01 has undertaken a technical review of a selected group of test procedures to determine selected chemical properties of RDF-3. The property of principal interest in this evaluation is the higher heating value. In order to properly characterize this property, critical evaluation of methods to determine total moisture, residual moisture, and ash is also necessary. Intralab and interlab variations in these properties are discussed. A comparison of these results with those on round robin data for coal are also made. The results of this study identify the levels of precision for intralab and interlab agreement.

Introduction

The National Bureau of Standards has been a participant in the development of standard test methods for refuse-derived fuel-three (RDF-3)* with ASTM committee E-38 (Resource Recovery) and its Subcommittee E-38.01 (Energy) since the beginning of the testing program in the summer of 1977. Since that time, two preliminary and three definitive series of round robin RDF-3 samples have been distributed and examined by various testing laboratories. At present, some twenty test protocols are in the form of editorial draft standards. These standards are undergoing evaluation and are under vote for acceptance at either Subcommittee or main Committee levels.

In addition to its role as a participating test laboratory, NBS has also studied the RDF-3 test protocols for the purpose of simplifying procedures, attempting to clarify ambiguities, modifying procedures to improve the precision and accuracy of test data, providing reasons for the apparent dispersion of data, developing new procedures when there appeared to be such a need, and suggesting levels of precision for tests.

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* Refuse-derived fuel-three (RDF-3) is defined as a shredded fuel derived from municipal solid waste (MSW) which has been processed for the removal of metal, glass, and other entrained inorganic materials. Generally, this material has a particle size such that 95 weight percent passes through a 2-inch square mesh screen.

Studies at NBS and elsewhere suggest that the heterogeneity of RDF still poses a serious problem to the extraction of a representative sample of RDF for analysis. Sampling techniques which will provide representative samples need further development. Poor agreement found for data within and between laboratories will not improve until advances in the sampling of RDF have occurred.

The focus of this paper is directed toward the evaluation of data on the higher heating values (HHV) of RDF-3 determined during the round robin testing program. As part of this evaluation of HHV data, procedures for moisture and ash content determinations were also studied because of the need to calculate higher heating values on an as-received basis (HHV1), dry basis (HHV2), and moisture-ash-free basis (HHV3).

The initial path taken by Subcommittee E-38.01 toward the development of standard test methods for RDF-3 was to examine the ASTM procedures already established for coal and coke, and to see what kind of changes were necessary to accommodate the testing of RDF-3. Modifications were introduced as they appeared necessary.

Definitive Round Robin Testing

Arrangements were made to begin a series of round robin tests on RDF-3. Samples were prepared at the National Center for Resource Recovery (NCRR), Washington, D.C., and were distributed to the various participating laboratories for the first two preliminary and first two definitive series of round robin tests. The samples for the third definitive series of testing were prepared and distributed by Americology, Milwaukee, Wisconsin. The RDF-3 laboratory sample was composed of one inch or smaller material. Each laboratory was provided with about 2 kg of sample for chemical analysis. Prior to processing the sample down to 0.5 mm particles for HHV, residual moisture, ash, and other determinations, the sample was air dried in the temperature range from 30 to 40 °C so that its moisture content could be brought near to equilibrium with the atmosphere in the test laboratory. We used 2.5 gram pellets which were prepared from 0.5mm RDF for the HHV, residual moisture, and ash content determinations.

The chronology of the round robin tests is outlined in table 1. Six laboratories participated in the first and second preliminary (zeroth) rounds of testing. The precision of the data did not seem satisfactory from these rounds. It was felt that a period of acclimation was required for laboratory analysts to acquire some experience in processing and analyzing RDF-3 samples.

Eight laboratories participated in the first definitive series of round robin tests which took place in June and July of 1978. The mean and standard deviation of measurements on higher heating values (HHV) as calculated for three different bases are shown in table 2; as-received basis (HHV1), dry basis (HHV2) and moisture-ash-free basis (HHV3). Corresponding data for total moisture, residual moisture, and ash content determinations are given in table 3.

Each mean value, $\bar{x}$, listed in tables 2 through 11 is the average of four determinations; two runs carried out on day one and two runs carried out on day two, each being performed in the same laboratory, by the same analyst, using the same apparatus. The standard deviation, s, of the four replicates is provided in tables 2 through 11. The classification of days and within-days was ignored in calculating s; however, this statistic is still a rough estimate of the variability within labs.

Also provided in tables 2 through 11 are averages of the means for the first round robin sample for the participating laboratories, the corresponding standard deviations, and the average value of the laboratory standard deviations.

Examination of the data on HHV1, HHV2, and HHV3 in table 2 showed that the dispersion of the data between each laboratory appeared higher than what one might expect and, in most cases, was considerably higher than what ASTM protocols for coal and coke (D2015) would allow. The standard deviations of the average of the means for HHV1, HHV2, and HHV3 were five to seven times the dispersion expected. The total moisture, residual moisture, and ash content data tabulated in table 3 also appeared to be disproportionately high.

Doubts about the homogeneity of the milled (particle size, 0.5 mm) RDF-3 sample were being raised at the ASTM E-38 meeting following the first round of testing. Some analysts suggested that sub-division was difficult because segregation of light and heavy particles was sometimes observed in either riffling or coning and quartering of the sample in preparation for analysis. The accountability of non-millable portions of the RDF-3 sample was not easy and required the personal and professional judgment of the analyst. It more than likely varied from one laboratory to another. Unequal distribution of non-millable material in the laboratory samples also contributed to this variation.

Interlaboratory Comparison Samples

In an effort to ascertain whether improved precision could be obtained for higher heating values, moisture, ash content determinations, and other important properties, an interlaboratory reference or comparison sample was prepared for round robin testing in addition to the usual sample of RDF-3 for the second and third rounds. The interlaboratory comparison sample was milled down to 0.5 mm particle size by a single laboratory prior to distribution to all of the participating labs. The comparison sample would eliminate possible biases which might have been introduced into the particle size reduction operation at each of the participating laboratories. Differences of opinion as to how: (1) the RDF-3 sample should be milled, split, blended, (2) non-millable material should be identified and extracted, and (3) sampling should be performed, suggested that the decisions by analysts at the various laboratories were not always equivalent. It was hoped that an interlaboratory comparison sample would remove such biases.

Tables 4 and 5 provide data on the higher heating values, moisture, and ash content for the RDF-3 sample prepared and distributed by Americology to twelve participating laboratories in the second round of testing. A significant improvement in precision was observed except for the results on residual moisture. The standard deviation of the average of the means for the residual moisture had doubled while the actual average of the means itself had decreased from 4.110 to 2.715 wt. percent. An examination of the standard deviations of the average of the means for HHV2 in tables 2 and 4 shows a decrease from 287 Btu/lb to 159 Btu/lb. A more modest improvement is found in tables 3 and 5 for total moisture and ash data when the standard deviations of these averages are inspected. Perhaps the laboratory analysts were becoming more accustomed to handling RDF-3. Results in general were still far from the precisions found for coal and coke.

Tables 6 and 7 show the statistical data for the interlaboratory comparison sample for the second round. The improvement in precision was very encouraging. The standard deviation of the average of the means for HHV2 was 60 Btu/lb; this represents about a 60 percent improvement over the corresponding data for HHV2 which was determined after each lab carried out its own milling of their laboratory samples. Similarly, the standard deviation of the average of the means for the ash content data for the interlaboratory comparison sample in table 7 was about half (0.73 wt. percent) of that found for the corresponding laboratory sample data shown in table 5 (1.46 wt. percent). The standard deviation of the average of the means for the residual moisture of the interlab sample decreased by half in comparison to the regularly processed second round sample. This improvement suggested that fewer biases were interacting.

A third round of testing was carried out during the late Spring of 1979. The statistical data calculated from the results of testing on both the regular RDF-3 laboratory sample and another interlaboratory comparison sample are presented in tables 8 through 11. These results are somewhat disappointing in that they resemble the first round in their level of precision more than the second round. Comparison of HHV1, HHV2, and HHV3 in table 8 with table 2 shows the standard deviation of the average of the means 10 to 20 percent lower in the third round than in the first round for the regular RDF-3 laboratory sample. Comparison of this same statistic in tables 3 and 9 shows that the precision for total moisture, residual moisture, and ash content determination had decreased significantly: from 1.23 to 1.75 wt. percent for the total moisture, from 0.497 to 1.54 wt. percent for residual moisture, and from 1.64 to 2.61 wt. percent for ash, respectively. Precisions were also lower when the standard deviation of the average of the means is examined for the results of the third interlaboratory comparison sample and compared to the corresponding data in the second round of testing. For example, this statistic is 154 Btu/lb for HHV2 for the third round (table 10) as compared with 60 Btu/lb for the second round (table 6). Similarly, the standard deviation of the average of the means for the ash data for the third round is over three times (2.50 wt. percent, table 11) that found for the interlaboratory sample studied in the second round (0.73 wt. percent, table 7). The residual moisture, however, is not too much different in the two interlab rounds of testing; compare this statistic in table 11 (0.643 wt. percent) with the corresponding value in table 7 (0.518 wt. percent).

Bituminous Coal Round Robin

The data obtained for HHV, moisture, and ash content determinations for the three rounds of testing were significantly more variable than had been expected. In order to establish some reassurance that a non-RDF reference sample could be analyzed satisfactorily, a standard bituminous coal sample (SRM-1632a) was distributed to six laboratories by NBS for HHV, residual moisture, and ash determinations. The results of this series of tests is shown in tables 12 and 13. In table 12 one finds that the standard deviations of the average of the means, for the coal sample are about the same for HHV1, HHV2, and HHV3, i.e., ∿ 50 $Btu(lb)^{-1}$. This value is considerably lower than comparable values of the standard deviation of the average of the means found for higher heating values in any of the three rounds of testing. Similarly, values of this statistic for residual moisture and ash content for the coal sample are much lower than comparable values found in earlier rounds of RDF-3 testing; for the residual moisture, the standard deviation of the average of the means is 0.207 wt. percent and for ash is 0.15 wt. percent (table 13).

Hence, this series of tests on bituminous coal showed that the participating laboratories could analyze a coal sample to suitable levels of precision.

Repeatability and Reproducibility

In order to provide some quantitative estimate of where the current state-of-the-art stood with respect to the analysis of RDF-3 samples in contrast to the analysis of coal, we carried out a statistical study of the data accummulated in the first three rounds of testing using a program based upon the theory of nested (hierarchical) designs (see "Statistical Methods in Research and Production" by O.T. Davies, Third Edition, published for Imperial Chemical Industries Ltd., Oliver and Boyd, London and Edinburgh; Hafner Publishing Co., New York). In this case, days were nested within labs and replicates within days. There were two days of testing for each lab and on each day two replicates were carried out.

The data obtained on the NBS bituminous coal sample (SRM 1632a) were analyzed using methods described in ANSI/ASTM E691-79, "Standard Practice for Conducting an Interlaboratory Test Program to Determine the Precision of Test Methods." In this case, each laboratory carried out two replicate determinations, but only on one day.

Two statistics were particularly sought, namely, the repeatability interval, I(r), which is a measure of within laboratory agreement, and the reproducibility interval, I(R), which is a measure of between laboratory agreement. These statistics could easily be compared to existing values listed in documented ASTM Standard Methods of Test for Coal and Coke for HHV, residual moisture and ash. For example, in ASTM D2015 (Standard Test Method for Gross Calorific

Value of Solid Fuel by the Adiabatic Bomb Calorimeter) the precision criteria for judging the acceptability of results at the 95 percent probability level are:

Repeatability - duplicate results by the same laboratory on different days, using the same operator and equipment should not be considered suspect unless they differ by more than 50 Btu/lb, dry basis.

Reproducibility - results submitted by two or more laboratories (different equipment, operators, date of test, and different portions of the same gross sample) should not be considered suspect unless two results differ by more than 100 Btu/lb, dry basis.

Equations required to calculate the repeatability interval, I(r), are:

$$I(r) = 2.83\ s(r);$$

where s(r) is the pooled within-laboratory standard deviation and 2.83 is a factor which converts the repeatability standard deviation to a 95 percent repeatability interval.

$$s(r) = [s^2(\mathrm{repl}) + s^2(\mathrm{days})]^{1/2}$$

where s(repl) is the standard deviation of replicate measurements and s(days) is the standard deviation due to variations in measurements observed between days.

Similarly, equations needed to calculate the reproducibility interval, I(R), are:

$$I(R) = 2.83\ s(R)$$

where s(R) is the between-laboratory estimate of precision and 2.83 is defined as above.

$$s(R) = [(s^2(\text{repl}) + s^2(\text{days}) + s^2(\text{labs})]^{1/2}$$

where s(labs) is the standard deviation due to variations in measurements observed between laboratories and s(repl) and s(days) are as defined above.

Tables 14 through 17 summarize the repeatability and reproducibility intervals calculated for HHV2, total moisture, residual moisture, and ash, respectively. Data on all three RDF-3 rounds of testing are tabulated, including the results from testing of the bituminous coal sample, SRM 1632a. The first row of tables 14, 16, and 17 gives the acceptability limits according to the appropriate ASTM protocol for coal and coke, residual moisture, and ash content, respectively. No acceptability limits are available for total moisture in coal and coke protocols.

Examination of the HHV2 data in table 14 shows that the repeatability interval for the SRM coal round of testing, 69 Btu/lb, is close to the acceptable limit of 50 Btu/lb. The reproducibility interval obtained for the SRM coal is 159 Btu/lb; this departs somewhat from the acceptable limit of 100 Btu/lb, yielding a ratio of I(R) to I(r) of 2.30 rather than 2.00.

The repeatability interval for the first three rounds of testing of RDF-3 are between 3 and 6 times the 50 Btu/lb limit; this includes the interlab comparison samples tested in rounds two and three. The values of I(r) for the three rounds of testing give an average value of 226 Btu/lb. The corresponding average value for I(R) is 694 Btu/lb. It may be observed that the I(R)/I(r) ratio for HHV2 for coal and coke is approximately 2.00, and for RDF-3 is close to 3.00. For practical purposes, one may adopt for RDF-3 repeatability and reproducibility intervals for HHV2 of 250 and 750 Btu/lb respectively. The latter values are much larger than those given for coal and coke, but we feel that they represent the current state-of-the-art in the repeatability and reproducibility of HHV2 for RDF-3.

Heterogeneity of RDF-3

The heterogeneity of RDF-3 is still present at 0.5 mm particle size.

Figures 1 and 2 show analysis samples of 0.5 mm RDF-3 magnified 50 times under a microscope. The particles are heterogeneous even at this level of sub-division. Some are spherical, some are elongated rods while others are irregularly shaped. Differences in density are suggested by the black and white contrast shown in the photos.

Figure 3 is the bituminous coal sample (SRM 1632a) magnified 50 times under a microscope. The particles are 0.25 mm (-60 mesh) and have a generally homogeneous appearance in comparison to the RDF-3 at the same magnification. Differences in the densities of 0.5 mm plastic, cellulose, iron, aluminum, sand, etc. help to defy the blending process and extraction of representative samples for various tests.

The values of I(r) and I(R) for the interlaboratory comparison samples of rounds two and three show that the second round sample gave better results than the third round. The reason for this is not immediately clear, but the I(R)/I(r) ratio does illustrate the benefits of having only one laboratory perform the processing of one inch nominal RDF-3 down to 0.5 mm. Biases in the reduction of the size of RDF-3 are an inherent part of the reproducibility interval values and cause I(R) to be so much larger than I(r) for the three regular rounds of testing.

Summary of Results

The summary of I(r) and I(R) data for total moisture is given in table 15. A trend is observed between the average of the means and the repeatability interval I(r). Samples increased in total moisture as testing went from the first, to the second round, and to the third rounds; compare 13.82, 18.91, and 30.09 wt. percent with I(r) values of 0.37, 0.57, and 0.76 wt. percent, respectively. Within-lab repeatability lies between 0.4 and 0.8 wt. percent while between-lab reproducibility lies between 3 and 5 wt. percent. The ratio of I(R)/I(r) is poorest for the first round, 9.21.

In table 16, we find I(r) and I(R) values for residual moisture. The limits imposed on coal and coke samples (ASTM D3173) are related to the moisture content of the samples and are given in the table. For example, if less than 5 wt. percent moisture is present in the coal or coke sample, then I(r) and I(R) values are not considered suspect unless they are above 0.2 and 0.3 wt. percent, respectively. The data obtained by the six participating laboratories for the SRM 1632a sample fall within the above acceptability limits since the sample contained 1.43 wt. percent residual moisture. There appears to be a trend in the I(r) values for the three rounds of testing similar to that observed in table 15, related to the average of the means of the total moisture. Whether there is a relationship or whether this is merely chance is not clear. The averages of the means of the residual moisture do not parallel the I(r) trend. The ratio of I(R)/I(r) ranges from 3.5 to 4.5. Again, for practical consideration, we have adopted the ratio 4.00 with 0.75 and 3.00 wt. percent as values for I(r) and I(r), respectively. No limits on the amount of residual moisture in the sample have been set as with coal and coke.

In table 17, we find the summary of values for I(r) and I(R) for the ash content data. The limits for acceptable data with regard to coal and coke samples (ASTM D31.74) which contain (or do not contain) carbonates or more than 12 wt. percent moisture are given in the first three rows of table 17. The results of testing the SRM 1632a coal sample show that the participating laboratories have determined the ash content well within the levels of acceptability. Values of 0.08 and 0.16 wt. percent for I(r) and I(R), respectively, are quite satisfactory. The repeatability and reproducibility intervals for the first three rounds are quite high in comparison to the coal and coke acceptability limits. The same is true for the I(r) and I(R) values obtained for the interlab comparison sample. Although the second round data for I(r) and I(R) are better for both the regular and interlab samples, the absolute value of the average of the means is significantly different; compare 14.17 wt. percent for the regular RDF-3 sample with 19.76 wt. percent for the interlab sample. This difference may be due to the overall heterogeneity of RDF-3. Other biases may be involved but are not apparent. The suggested values for I(r) and I(R) are 2.5 and 7.5 wt. percent, respectively. These values are based on an adopted I(R)/I(r) ratio of 3.00, and are much higher than expected, but this is what is observed.

Final Summary and Conclusions

(1) The data from higher heating value, total moisture, residual moisture, and ash content determinations were examined and evaluated for three rounds of RDF-3 sample testing.

(2) Repeatability and reproducibility intervals were calculated as a result of carrying out a statistical analysis of the data as described in ANSI/ASTM E691-79 for the bituminous coal round robin, and in "Statistical Methods in Research and Production" by O. T. Davies (loc. cit.) for the RDF-3 round robin tests.

(3) Repeatability intervals for HHV2 (dry basis), residual moisture, and ash content are 250 Btu/lb, 0.75 wt. percent, and 2.5 wt. percent, respectively. These adopted values are significantly higher than the acceptable limits for coal and coke. Compare the above with 50 Btu/lb, 0.3 wt. percent, and 0.5 wt. percent, respectively, which are the acceptable limits for coal and coke.

(4) Reproducibility intervals for HHV2 (dry basis), residual moisture, and ash content are 750 Btu/lb, 3.00 wt. percent and 7.5 wt. percent, respectively. As with the repeatability intervals, these adopted values are significantly higher by six to eight times the acceptable limits for coal and coke. Compare the above with 100 Btu/lb, 0.5 wt. percent and 1.0 wt. percent, respectively, which are the acceptable limits for coal and coke.

(5) The heterogeneity of RDF-3 and RDF milled to 0.5 mm particles makes extraction of a representative sample difficult. Further development of sampling techniques for RDF-3 and size reduction to smaller particles must be pursued before improvement in the levels of precision of round robin tests can be expected.

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Table 1
RDF-3 Testing Program

Round Robin Test	Period of Study
Zeroth round robin (first)	Sept., Oct. 1977
Zeroth round robin (second)	Dec., 1977, Jan., 1978
First round robin	June, July 1978
Second round robin	Dec., 1978, Jan., 1979
Third round robin	May, June 1979
Bituminous coal round robin	Feb., Mar., 1980

Table 2
ASTM Round Robin Testing of RDF-3 (First Round) Statistical Data on Higher Heating Values RDF-3 Sample Prepared by NCRR

Laboratory No.	Mean $\overline{X}$ (Btu/lb)			Standard Deviation S (Btu/lb)		
	HHV1	HHV2	HHV3	HHV1	HHV2	HHV3
Lab 1	6473	7519	9579	23	24	58
Lab 2	6709	7842	10214	37	46	66
Lab 3	6527	7585	9476	65	75	189
Lab 4	6095	7426	9795	17	22	105
Lab 5	6151	7187	9896	52	60	30
Lab 6	6830	7687	9619	13	15	19
Lab 7	5859	6929	8951	175	194	310
Lab 8	6384	7387	9434	91	103	195
Average	6378	7445	9621	54	67	122
Std. Dev.	306	287	357			

Table 3
ASTM Round Robin Testing of RDF-3 (First Round) Statistical Data on Total Moisture, Residual Moisture, and Ash RDF-3 Sample Prepared by NCRR

Laboratory No.	Mean $\overline{X}$ (wt. percent)			Standard Deviation S (wt. percent)		
	Total Moist.	Res. Moist.	Ash	Total Moist.	Res. Moist.	Ash
Lab 1	13.91	4.845	21.51	0.03	0.034	0.24
Lab 2	14.44	3.865	23.23	0.11	0.122	0.28
Lab 3	13.94	3.853	19.94	0.07	0.080	1.67
Lab 4	13.59	4.478	24.18	0.05	0.056	0.97
Lab 5	14.41	(1.525)*	24.06	0.05	(0.054)*	0.74
Lab 6	11.15	4.475	20.09	0.01	0.180	0.50
Lab 7	15.45	3.830	22.57	0.19	0.216	1.31
Lab 8	13.58	3.425	21.69	0.10	0.108	0.91
Average	13.81	4.110	22.16	0.08	0.114	0.83
Std. Dev.	1.23	0.497	1.64			

(*)Data from Lab 5 not used in calculations

Table 4

ASTM Round Robin Testing of RDF-3 (Second Round) Statistical Data on Higher Heating Values RDF-3 Sample Prepared by NCRR

Laboratory	Mean $\overline{X}$ (Btu/lb)			Standard Deviation S (Btu/lb)		
	HHV1	HHV2	HHV3	HHV1	HHV2	HHV3
Lab 1	6450	7759	9301	9	11	24
Lab 2	6622	8167	9397	28	51	23
Lab 3	6438	7973	9301	23	26	37
Lab 4	6294	7901	9233	34	45	73
Lab 5	6324	7735	8792	17	22	26
Lab 6	6517	8012	9229	42	51	64
Lab 7	6473	8041	9356	38	44	27
Lab 8	6413	7836	9165	65	90	146
Lab 9	—	—	—	—	—	—
Lab 10	6183	7781	9305	15	15	33
Lab 11	7103	7899	9289	3	5	31
Lab 12	6717	8209	9373	71	102	120
Average	6503	7938	9249	31	42	55
Std. Dev.	248	159	166			

Table 5
ASTM Round Robin Testing of RDF-3 (Second Round) Statistical Data on Total Moisture, Residual Moisture, and Ash RDF-3 Sample Prepared by NCRR

Laboratory	Mean $\overline{X}$ (wt. percent)			Standard Deviation S (wt. percent)		
	Total Moist.	Res. Moist.	Ash	Total Moist.	Res. Moist.	Ash
Lab 1	16.87	2.185	16.58	0.03	0.436	0.22
Lab 2	18.89	2.845	13.09	0.35	0.451	0.48
Lab 3	19.26	2.998	14.28	0.15	0.175	0.09
Lab 4	20.34	2.358	14.43	0.07	0.079	0.31
Lab 5	18.24	2.413	12.03	0.06	0.071	0.33
Lab 6	18.66	3.135	13.18	0.07	0.081	0.16
Lab 7	19.48	2.613	14.04	0.04	0.048	0.38
Lab 8	18.34	2.158	14.50	0.07	0.288	0.45
Lab 9	—	—	—	—	—	—
Lab 10	20.53	2.190	16.41	0.06	0.070	0.38
Lab 11	(10.17)*	1.568	14.88	(0.14)*	0.173	0.20
Lab 12	18.17	5.405	12.41	0.17	0.205	0.93
Average	18.91	2.715	14.17	0.11	0.189	0.36
Std. Dev.	1.08	0.996	1.46			

(*)Data from Lab 11 not used in calculations

Table 6
ASTM Round Robin Testing of RDF-3 (Second Round) Statistical Data on Higher Heating Values RDF-3 Interlaboratory Comparison Sample Prepared by Wisconsin Electric

Laboratory	Mean $\overline{X}$ (Btu/lb)			Standard Deviation S (Btu/lb)		
	HHV1	HHV2	HHV3	HHV1	HHV2	HHV3
Lab 1	—	7622	9553	—	40	59
Lab 2	—	7619	9606	—	63	203
Lab 3	—	7571	9385	—	58	174
Lab 4	—	7627	9531	—	30	58
Lab 5	—	7545	9187	—	54	126
Lab 6	—	7611	9566	—	28	61
Lab 7	—	(6713)*	(8399)*	—	(626)*	(802)*
Lab 8	—	7552	9396	—	205	172
Lab 9	—	7655	9578	—	29	78
Lab 10	—	7651	9470	—	27	50
Lab 11	—	7755	9702	—	38	67
Lab 12	—	7681	9524	—	109	73
Average	—	7627	9500	—	62	102
Std. Dev.	—	60	138			

(*)Data from Lab 7 not used in calculations

Table 7
ASTM Round Robin Testing of RDF-3 (Second Round)
Statistical Data on Residual Moisture and Ash
RDF-3 Interlaboratory Comparison Sample Prepared by Wisconsin Electric

Laboratory	Mean $\overline{X}$ (wt. percent)		Standard Deviation S (wt. percent)	
	Res. Moist.	Ash	Res. Moist.	Ash
Lab 1	3.360	20.22	0.070	0.43
Lab 2	3.335	20.67	0.484	1.32
Lab 3	3.278	19.32	0.182	0.90
Lab 4	3.353	19.97	0.079	0.40
Lab 5	2.570	17.86	0.061	1.42
Lab 6	3.415	20.44	0.785	0.76
Lab 7	2.788	20.05	0.120	1.23
Lab 8	3.323	19.63	0.239	1.56
Lab 9	2.645	20.08	0.065	0.41
Lab 10	1.683	19.49	0.052	0.22
Lab 11	3.070	20.07	0.224	0.17
Lab 12	3.378	19.34	0.303	1.33
Average	3.017	19.76	0.22	0.85
Std. Dev.	0.518	0.73		

Table 8

ASTM Round Robin Testing of RDF-3 (Third Round) Statistical Data on Higher Heating Values RDF-3 Sample Prepared by Americology

Laboratory	Mean $\bar{X}$ (Btu/lb)			Standard Deviation S (Btu/lb)		
	HHV1	HHV2	HHV3	HHV1	HHV2	HHV3
Lab 1	5397	7718	9736	22	30	25
Lab 2	5426	7847	9851	21	17	38
Lab 3	5158	7436	9539	126	175	152
Lab 4	5128	7313	9438	28	37	44
Lab 5	5420	7466	9619	11	16	19
Lab 6	5178	7345	9922	39	46	222
Lab 7	6658	7392	9664	30	84	168
Lab 8	5899	7616	10283	373	48	79
Lab 9	5147	7391	9103	18	26	33
Lab 10	4956	7260	9501	17	26	45
Lab 11	4703	7094	9224	6	7	14
Lab 12	5676	8015	9682	178	216	444
Average	5395	7491	9630	72	61	107
Std. Dev.	249	262	313			

Table 9
ASTM Round Robin Testing of RDF-3 (Third Round) Statistical Data on Total Moisture, Residual Moisture, and Ash RDF-3 Sample Prepared by Americology

Laboratory	Mean $\overline{X}$ (wt. percent)			Standard Deviation S (wt. percent)		
	Total Moist.	Res. Moist.	Ash	Total Moist.	Res. Moist.	Ash
Lab 1	30.08	4.470	20.73	0.10	0.134	0.20
Lab 2	30.84	3.485	20.33	0.18	0.249	0.19
Lab 3	30.64	3.573	22.05	0.07	0.097	0.70
Lab 4	29.88	4.796	22.52	0.04	0.048	0.31
Lab 5	27.59	4.063	22.38	0.09	0.119	0.26
Lab 6	29.51	4.960	25.95	0.10	0.132	1.20
Lab 7	(9.93)*	—	23.50	(0.67)*	—	0.57
Lab 8	27.54	7.410	25.94	0.62	0.798	0.51
Lab 9	30.35	4.523	18.81	0.02	0.340	0.16
Lab 10	31.74	3.163	23.58	0.06	0.062	0.29
Lab 11	33.70	3.780	23.10	0.08	0.117	0.10
Lab 12	29.17	7.843	17.17	0.35	0.457	1.57
Average	30.09	4.733	22.17	0.16	0.232	0.51
Std. Dev.	1.75	1.54	2.61			

(*)Data from Lab 7 was not used in calculations

Table 10
ASTM Round Robin Testing of RDF-3 (Third Round) Statistical Data on Higher Heating Values RDF-3 Inter-lab Comparison Sample Prepared by ORNL

Laboratory	Mean $\overline{X}$ (Btu/lb)			Standard Deviation S (Btu/lb)		
	HHV1	HHV2	HHV3	HHV1	HHV2	HHV3
Lab 1	—	7530	9282	—	30	62
Lab 2	—	7370	9528	—	28	98
Lab 3	—	7342	9240	—	100	143
Lab 4	—	7148	9170	—	71	111
Lab 5	—	7383	9470	—	5	7
Lab 6	—	7177	10098	—	42	264
Lab 7	—	7277	9422	—	64	140
Lab 8	—	7338	9648	—	127	94
Lab 9	—	7217	9446	—	27	46
Lab 10	—	7355	9213	—	23	130
Lab 11	—	7693	9758	—	45	99
Lab 12	—	7215	9190	—	284	382
Average	—	7337	9455	—	71	131
Std. Dev.	—	154	276			

Table 11
ASTM Round Robin Testing of RDF-3 (Third Round) Statistical Data on Residual Moisture and Ash RDF-3 Interlaboratory Comparison Sample Prepared at ORNL

Laboratory	Mean $\overline{X}$ (wt. percent)		Standard Deviation S (wt. percent)	
	Res. Moist.	Ash	Res. Moist.	Ash
Lab 1	4.605	18.87	0.03	0.35
Lab 2	4.373	22.64	0.18	0.66
Lab 3	3.775	20.54	0.11	0.54
Lab 4	4.434	22.05	0.07	0.54
Lab 5	4.135	21.71	0.09	0.07
Lab 6	3.088	28.91	0.64	1.52
Lab 7	5.418	22.75	0.41	1.56
Lab 8	4.298	23.94	0.22	0.64
Lab 9	3.868	23.60	0.02	0.37
Lab 10	4.128	20.27	0.16	0.82
Lab 11	5.328	21.44	0.37	0.39
Lab 12	4.698	21.49	0.07	0.52
Average	4.346	22.35	0.64	0.67
Std. Dev.	0.643	2.50		

Table 12
ASTM Round Robin Testing of RDF-3 Statistical Data on Higher Heating Values Coal Sample (SRM 1632a) Distributed by NBS

Laboratory	Mean $\overline{X}$ of Duplicate Runs (Btu/lb)			Standard Deviation S (Btu/lb)		
	HHV1	HHV2	HHV3	HHV1	HHV2	HHV3
Lab 1	11155	11335	14546	0	1	32
Lab 3	11266	11437	14636	24	57	72
Lab 4	11184	11314	14534	4	5	14
Lab 5	11220	11378	14612	16	16	21
Lab 7	11137	11284	14503	15	8	12
Lab 8	11164	11361	14584	1	3	2
Average	11188	11352	14569	10	15	26
Std. Dev.	48	54	50			

Table 13
ASTM Round Robin Testing of RDF-3 Statistical Data on Residual Moisture and Ash Coal Sample (SRM 1632a) Distributed by NBS

Laboratory	Mean $\overline{X}$ of Duplicate Runs (wt. percent)		Standard Deviation S (wt. percent)	
	Res. Moist.	Ash	Res. Moist.	Ash
Lab 1	1.590	22.21	0.014	0.007
Lab 3	1.485	21.80	0.276	0.000
Lab 4	1.145	22.16	0.007	0.042
Lab 5	1.355	22.12	0.007	0.050
Lab 7	1.305	22.20	0.064	0.007
Lab 8	1.720	22.10	0.042	0.007
Average	1.433	22.10	0.068	0.019
Std. Dev.	0.207	0.15		

Table 14
Summary of Repeatability and Reproducibility Intervals for Higher Heating Value HHV2, Dry Basis in ASTM Round Robin Testing of RDF-3

Method/Test	Lab Average HHV2, Btu/lb	Repeat. Interval I(r) (95% Probab.) HHV2, Btu/lb	Reprod. Interval I(R) (95% Probab.) HHV2, Btu/lb	Reprod./ Repeat. I(R)/I(r)
ASTM D 2015, Coal and Coke	—	50	100	2.00
SRM Coal 1632a	11352	69	159	2.30
First Round Robin	7445	284	841	2.96
Second Round Robin	7938	146	469	3.21
Third Round Robin	7491	249	772	3.10
Second Round Interlab Comparison Sample	7626	238	260	1.09
Third Round Interlab Comparison Sample	7337	296	502	1.70
Adopted Values	—	250	750	3.00

Table 15
Summary of Repeatability and Reproducibility of Total Moisture in ASTM Round Robin Testing of RDF-3

Test	Lab Average Total Moist. (wt. percent)	Repeat. Interval I(r) (95% Probab.) Total Moist. (wt. percent)	Reprod. Interval I(R) (95% Probab.) Total Moist. (wt. percent)	Reprod./Repeat. I(R)/I(r)
First Round Robin	13.82	0.37	3.41	9.21
Second Round Robin	18.91	0.57	3.24	5.68
Third Round Robin	30.09	0.76	4.99	6.57

Table 16
Summary of Repeatability and Reproducibility for Residual Moisture in ASTM Round Robin Testing of RDF-3

Method/Test	Lab Average Res. Moist., (wt. percent)	Repeat. Interval I(r) (95% Probab.) Res. Moist., (wt. percent)	Reprod. Interval I(R) (95% Probab.) Res. Moist., (wt. percent)	Reprod./ Repeat. I(R)/I(r)
ASTM D 3173, Coal and Coke				
less than 5% moisture	—	0.2	0.3	1.50
more than 5% moisture	—	0.3	0.5	1.67
SRM Coal 1632a	1.43	0.12	0.23	1.92
First Round Robin	4.11	0.41	1.44	3.51
Second Round Robin	2.72	0.63	2.86	4.54
Third Round Robin	4.73	0.99	4.42	4.46
Second Round Interlab Comparison Sample	3.02	0.58	1.55	2.67
Third Round Interlab Comparison Sample	4.35	0.84	1.93	2.30
Adopted Values	—	0.75	3.00	4.00

Table 17
Summary of Repeatability and Reproducibility Intervals for Ash Content in ASTM Round Robin Testing of RDF-3

Method/Test	Lab Average Ash, Dry Basis, (wt. percent)	Repeat. Interval I(r) (95% Probab.) Ash, Dry Basis, (wt. percent)	Reprod. Interval I(R) (95% Probab.) Ash, Dry Basis, (wt. percent)	Reprod./ Repeat. I(R)/I(r)
ASTM D 3174, Coal and Coke				
no carbonates	—	0.2	0.3	1.50
carbonates	—	0.3	0.5	1.67
more than 12% ash	—	0.5	1.0	2.00
SRM Coal 1632a	22.10	0.08	0.16	2.10
First Round Robin	22.16	2.73	5.20	1.90
Second Round Robin	14.17	1.31	4.26	3.25
Third Round Robin	22.17	1.88	7.57	4.03
Second Round Interlab Comparison Sample	19.76	3.09	3.16	1.02
Third Round Interlab Comparison Sample	22.35	2.54	7.35	2.89
Adopted Values	—	2.5	7.5	3.00

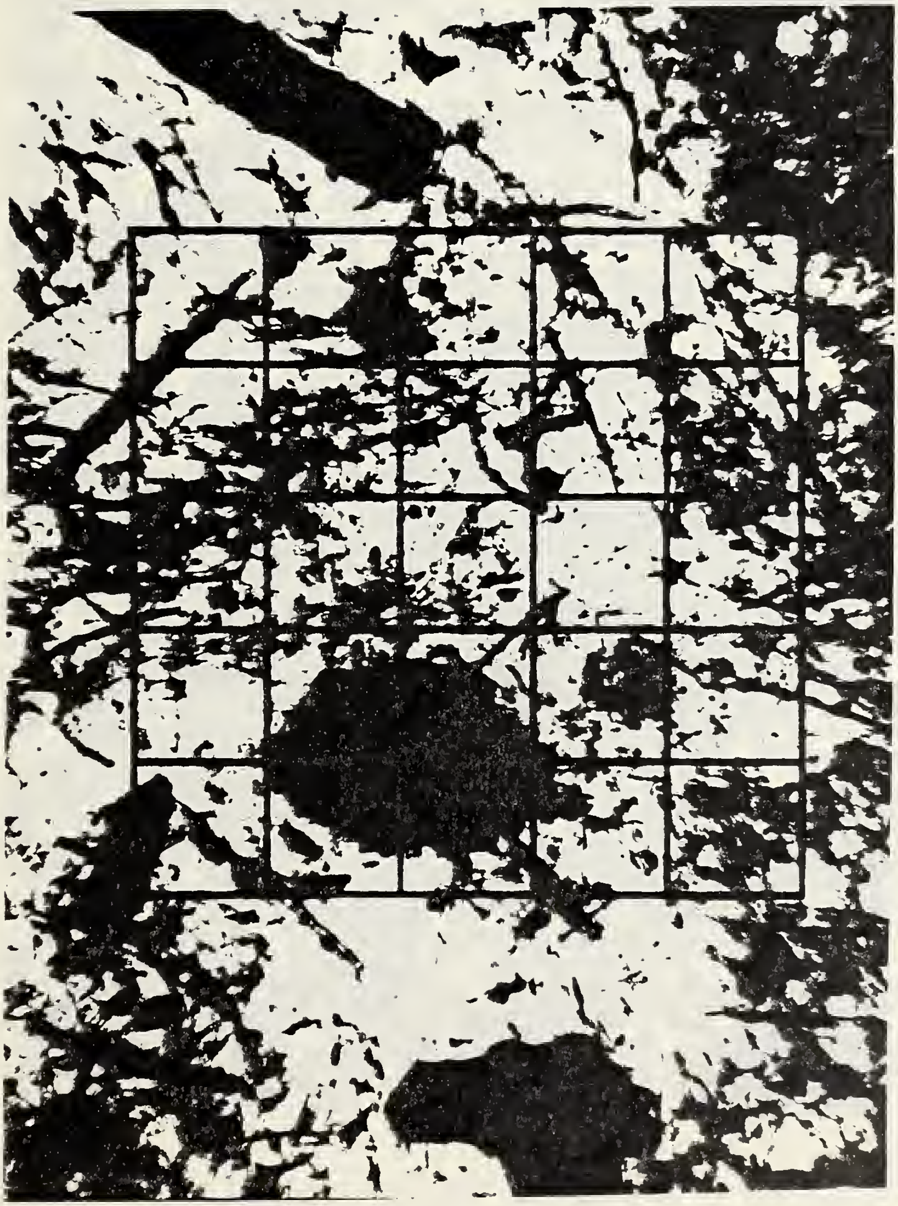


Figure 1 RDF-3 Reduced to 0.5 mm

Magnified 50 Times

(Each square is 0.25 mm on a side)

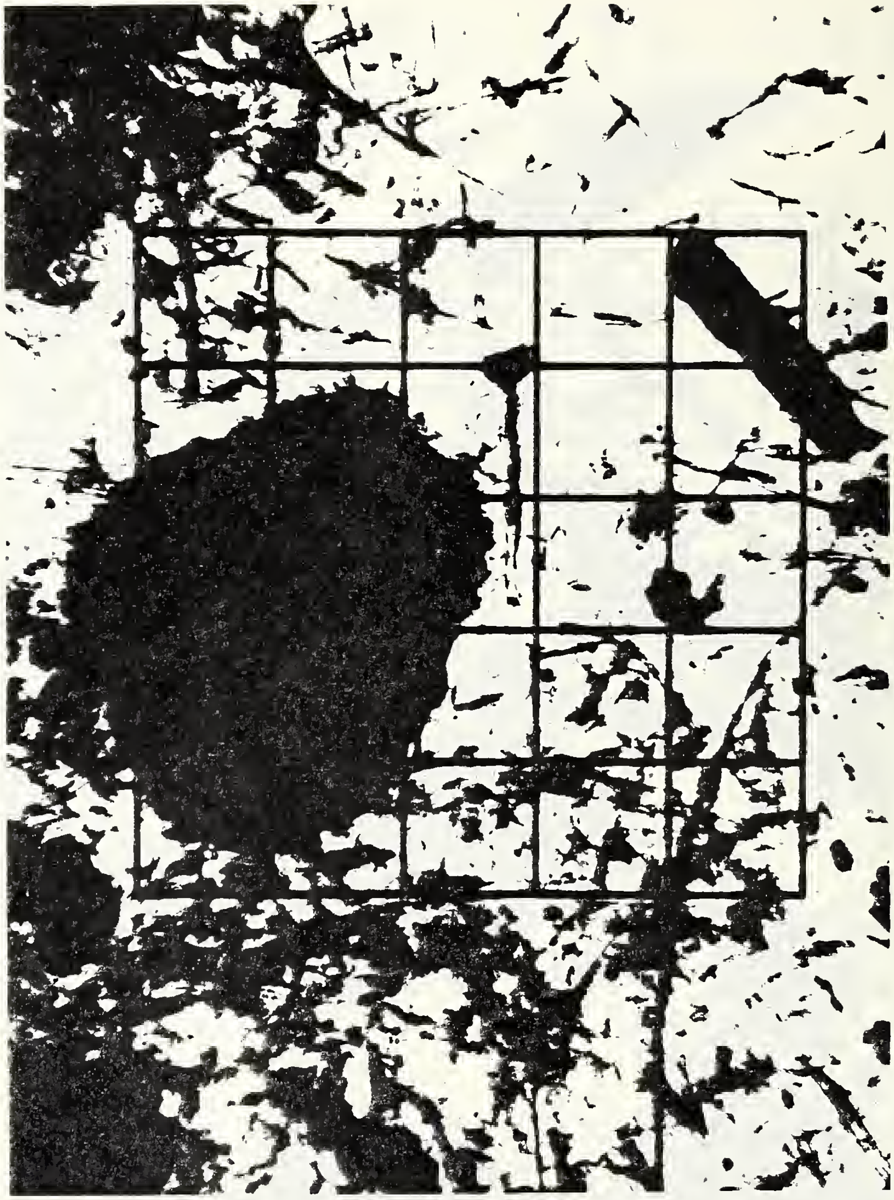


Figure 2 RDF-3 Reduced to 0.5 mm

Magnified 50 Times

(Each square is 0.25 mm on a side)

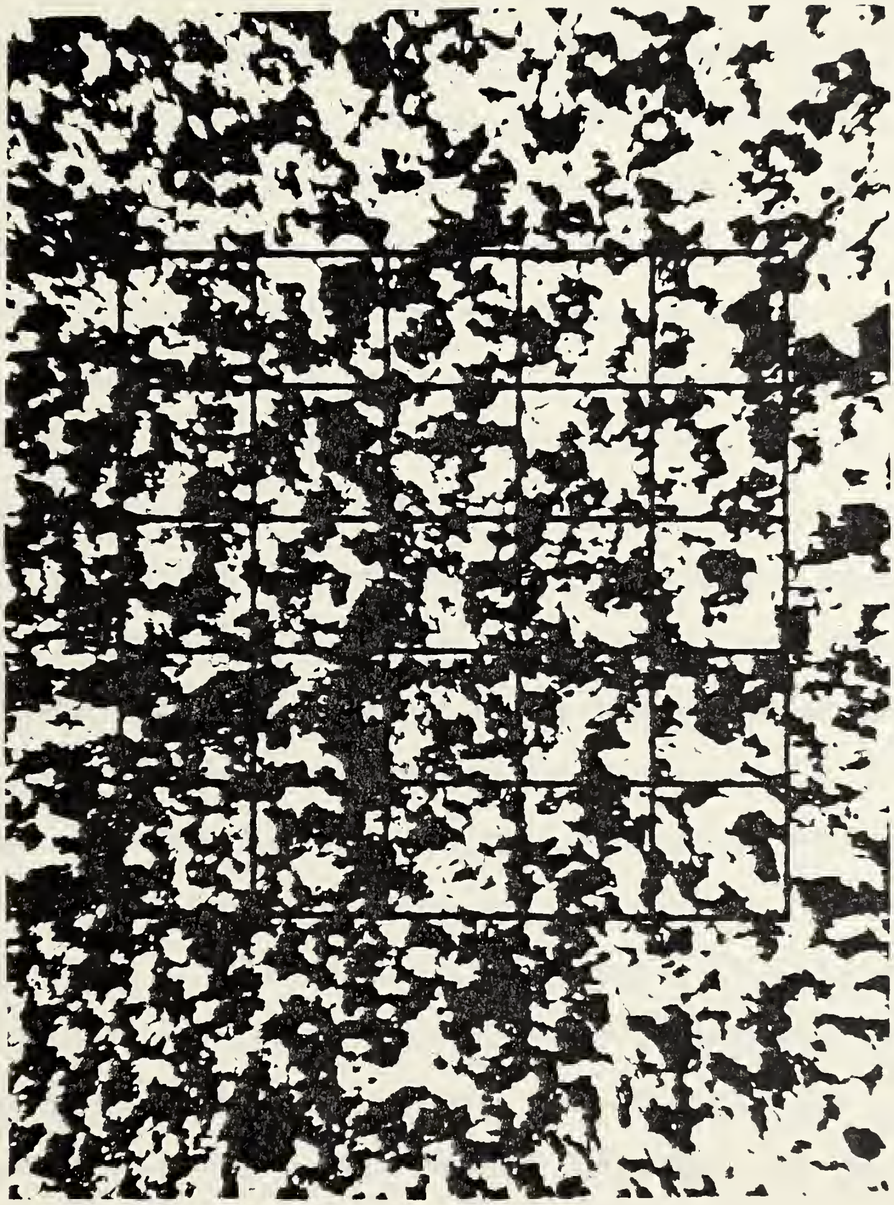


Figure 3 SRM 1632a Bituminous Coal Sample

Magnified 50 Times

(Each square is 0.25 mm on a side)

APPENDIX B

1. Large Isoperibol Bomb Calorimeter

The design of the large isoperibol (isothermal-jacket) calorimeter which will accommodate the large combustion bomb, described in the following section, is similar to that of Coops, van Nes, Kentee and Dienske, Rec. Trav. Chim. 66, 113-130 (1947), Coops and van Nes, Rec. Trav. Chim. 66, 131-141 (1947), and Gundry, Harrop, Head, and Lewis, J. Chem. Thermodynam. 1, 321-332 (1969) and is shown in Figure 1 of this appendix. The calorimeter is constructed entirely of stainless steel. It consists of a cylindrical calorimeter vessel 25.4 cm diam., 45.7 cm height (10 inch diam., 18 inch height) in which three rods support a concentric cylindrical shield 20.3 cm diam., 30.5 cm height (8 inch diam., 12 inch height). The calorimeter lid supports a stirrer assembly and a shield cover. The shield, shield cover, and stirrer assembly facilitate the flow of water in the calorimeter so that water is moving downward in the space between the bomb and shield, and upward between the shield and wall of the calorimeter vessel. The combustion bomb is supported by a foot which has been welded to the bottom of the vessel to insure that the bomb will be positioned in the same manner in the calorimeter for each experiment. The calorimeter vessel contains 19 liters of water for each calorimetric measurement and is housed in a submarine vessel which has a cover. The submarine cover is fastened to the vessel with six bolts and has four vertical ports for: (1) fuse circuit leads, (2) a calorimeter heater, (3) a central stirrer assembly and, (4) quartz thermometer leads. The frequency of oscillation of a temperature-sensitive quartz crystal is used to determine the calorimeter temperature. The NBS standard frequencies of 10 and 100 kHz are utilized. The entire calorimeter system (bomb, calorimeter vessel, and submarine compartment) is immersed in a constant temperature water bath maintained at 30 ± 0.03°C. The overall volume of the bath is 280 liters and holds 235 liters of water with the calorimeter system immersed in it.

2. Large Combustion Bomb

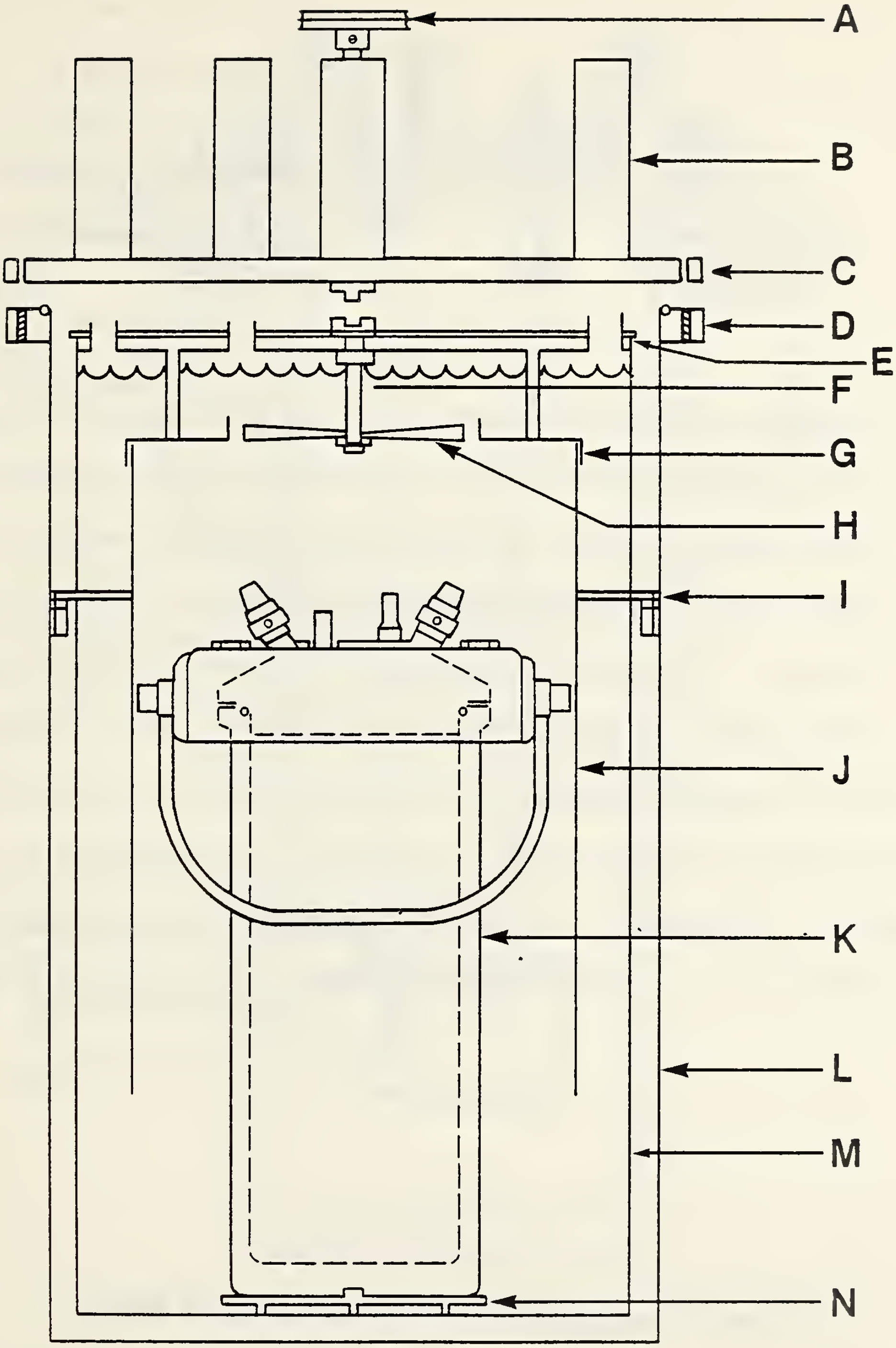
The combustion bomb which accommodates a 25 gram sample of RDF was purchased from the Parr Instrument Company and is shown schematically in Figure 2 of this appendix. The bomb has an overall height of 35 cm (13.75 in) and has a mass of 13.8 kg (30.5 lb) when assembled; the internal and external volumes of the bomb are 1.85 and 3.62 liters, respectively. A platinum crucible (~50 cm^3) was used in the calibration experiments with benzoic acid and a stainless steel crucible (~50 cm^3) was used in the combustion experiments with RDF. The bomb body has a wall thickness of 0.953 cm (0.375 in) and an outer diameter of 11.4 cm (4.5 in).

3. Calorimetric Procedures

Stoichiometrically, 2 grams of oxygen are required for the combustion of 1 gram of benzoic acid. Normally, 10 grams of oxygen are used for each gram of benzoic acid to insure complete combustion in the conventional-size bomb. It has been found in practice that 6.5 g of oxygen per gram of benzoic acid is the minimum requirement for complete combustion in the present design of bomb calorimeters. The large bomb utilizes a 16.3 gram benzoic acid sample and contains about 100 grams of oxygen under a pressure of 4.1 MPa (40 atm). This is on the borderline of the minimum amount of oxygen necessary for complete combustion in bomb calorimetry. Due to the limited quantity of oxygen in the large bomb, combustion is incomplete and a few tenths of a milligram of carbon residue is found in the combustion crucible. The energy of combustion of benzoic acid assumes complete combustion and therefore a small correction for unburned carbon, q-carbon, is included in the energy summation. In our experiments, the total correction for unburned carbon is usually less than 40 joules for a 400 000 joule experiment (1 part/10 000; hence, even without this correction, the error introduced would be below the 1 part in 10 000 level, and acceptable for precision combustion calorimetry of benzoic acid.

The corrected temperature rise of the calorimeter, ΔT-corr, is calculated and divided into the total energy supplied to the calorimeter, Q-total, to yield the energy equivalent, E-cal, of the calorimeter. The mass of water introduced into the bomb, the crucible, and the sample may change from experiment to experiment. The product of the mass of these materials and their specific heats is subtracted from the energy equivalent of the calorimeter, and the energy equivalent of the "empty" calorimeter, E-si, is obtained.

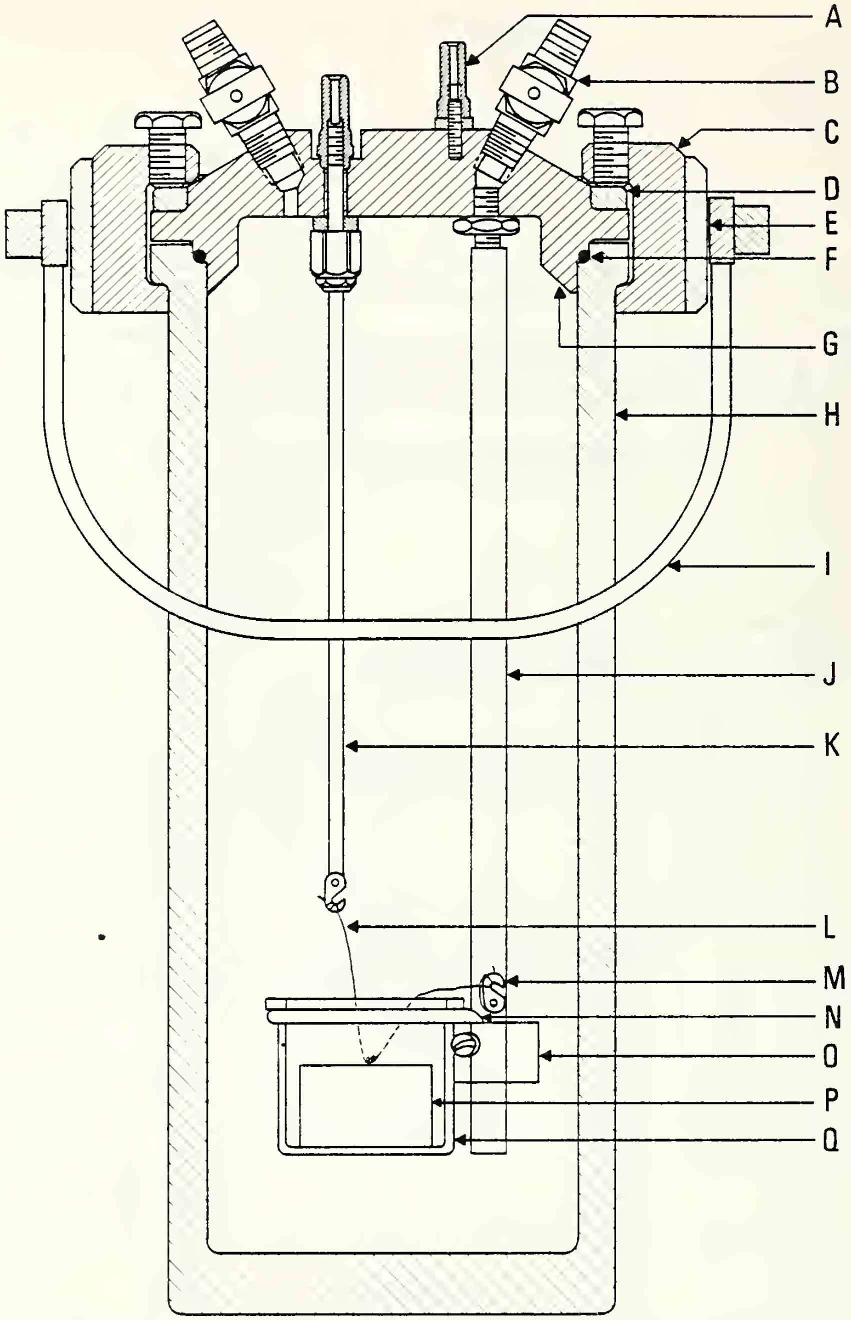
The important parameters to compare between the two calorimeters are the energy equivalents and the total energy capacities. The energy equivalent of the small calorimeter is 14 555 joules per Kelvin while that of the large calorimeter is 86 162 joules per Kelvin. The product of the energy equivalent and the specified temperature rise gives the total energy capacity. The large calorimeter has a total energy capacity of 430 810 joules, as compared to 43 665 joules of the small calorimeter. Therefore, a sample which has a mass ten times larger and less particle size reduction can be used in the large calorimeter. This is extremely important since the calorific value of an RDF sample can now be determined with only minimal processing, and the possibility of chemical changes imposed upon the RDF sample as a result of size reduction can now be investigated quantitatively.



25-gram-capacity Bomb Calorimeter

A, pulley to stirrer motor; B, one of four stacks attached to the submarine lid to accommodate (1) fuse circuit, (2) heater, (3) temperature sensor, and (4) stirrer, C, submarine lid; D, submarine flange with "O" ring; E, calorimeter vessel lid; F, stirrer shaft; G, shield cover; H, stirrer; I, calorimeter vessel supports; J, shield; K, combustion bomb; L, submarine vessel; M, calorimeter vessel; N, bomb foot.

FIGURE 1



Inner Arrangement of Large Combustion Bomb

A Grounded Electrode
B Valve
C Split Ring
D Compression Ring
E Drop Band
F Buna-N O-Ring
G Bomb Head
H Bomb Body
I Handle
J Gas Inlet Tube
K Ungrounded Electrode
L Fuse
M Fuse Attachment Hook
N Ring Holder
O Ring Support
P Sample Pellet
Q Crucible

FIGURE 2

Appendix C

Emission Spectrochemical Analyses of Bomb Residues

1. Teledyne National
2. ECO FUEL-II
3. Americology

Emission spectrochemical analyses were performed on bomb residues which were collected after bomb calorimetric measurements were performed on RDF samples. The RDF samples for which emission spectrochemical analyses are available are: Teledyne National, ECO FUEL-II, and Americology. Semi-quantitative results which were obtained for the RDF residues are provided in tables 1, 2, and 4 of this appendix. The elements are listed in order of decreasing abundance in the residue with the approximate percentages adjacent to the elemental symbol in parenthesis. In addition, similar data are given for ECO FUEL-II and the Americology RDF samples prior to their combustion in a bomb calorimeter. The latter data are shown in tables 3 and 5 of this appendix.

Table 1. Emission Spectrochemical Analysis of Teledyne National RDF Bomb Residue [1]

% $\geq$ 1	1 > % $\geq$ .1	.1 > % $\geq$ .01	.01 > % $\geq$ .001
Si (50)	Mn (.5)	Li (.05)	Ag (.002)
Ca (15)	Ba (.2)	Cu (.04)	Be (.001)
Na (10)	Sr (.2)	Cd (.02)	
Al (5)	Cr (.1)	Ni (.02)	
Fe (2)	Pb (.1)	V (.02)	
Zn (2)	Sn (.1)	Zr (.02)	
K (2)		B (.01)	
Mg (1)		Mo (.01)	
Ti (.1)		Rb (.01)	

Table 2. Emission Spectrochemical Analysis of ECO FUEL-II RDF Bomb Residue

$\% \geq 1$	$1 > \% \geq .1$	$.1 > \% \geq .01$	$.01 > \% .001$
Si (>>15)	Ti (.5)	Cr (.05)	Ag (.001)
Al (5)	Mn (.3)	Cu (.05)	
Ca (3)	Ba (.2)	Ni (.02)	
Fe (2.5)	Sr (.1)	Su (< .02)	
Na (.2)		V (< .02)	
Mg (1.5)		B (.01)	
K (1)			

Table 3. Emission Spectrochemical Analysis of ECO FUEL-II RDF (as-received)

$\% \geq 1$	$1 > \% \geq .1$	$.1 > \% \geq .01$	$.01 > \% \geq .001$
Si (2)	Al (.5)	Mn (.05)	Ag (< .001)
	Fe (.5)	Ti (.05)	
	Ba (.5)	Ba (.02)	
	Ca (.2)	Cu (.02)	
	K (.2)	Cr (< .02)	
	Mg (.2)	Ni (< .02)	
		Su (< .02)	
		Sr (< .02)	

Table 4. Emission Spectrochemical Analysis of Americology RDF Bomb Residue

$\% \geq 1$	$1 > \% \geq .1$	$.1 > \% \geq .01$	$.01 > \% \geq .001$
Si (>>15)	Ti (.5)	Cr (.05)	Ag (.002)
Na (10)	Ba (.2)	Cu (.05)	
Ca (5)	Mn (.2)	Sr (.05)	
Mg (5)	Pb (.2)	Ni (.02)	
Al (2.5)		Sn (.02)	
Fe (1.5)		V (<.02)	
K (1)		B (.01)	

Table 5. Emission Spectrochemical Analysis of Americology RDF processed to 0.5mm Particle Size

$\% \geq 1$	$1 > \% \geq .1$	$.1 > \% \geq .01$	$.01 > \% \geq .001$
Si (5)	Al (.5)	Mn (.05)	Ag (< .001)
Na (1)	Ca (.5)	Ti (.05)	
Mg (1)	Fe (.3)	Ba (.02)	
	K (.2)	Cu (.01)	
	Pb (.2)	Ni (<.02)	
		Sn (<.02)	
		Sr (<.02)	
		V (<.02)	
		Cr (<.02)	
		B (< .01	

APPENDIX D

Conversion Table for the Calculation of Data to Different Bases

Conversion Formula Table

	Wanted			
Given	As Determined (ad)	As Received (ar)	Dry (d)	Dry Ash-Free (daf)
As Determined (ad)	———	$\frac{100 - M_{ar}}{100 - M_{ad}}$	$\frac{100}{100 - M_{ad}}$	$\frac{100}{100 - M_{ad} - A_{ad}}$
As Received (ar)	$\frac{100 - M_{ad}}{100 - M_{ar}}$	———	$\frac{100}{100 - M_{ar}}$	$\frac{100}{100 - M_{ar} - A_{ar}}$
Dry (d)	$\frac{100 - M_{ad}}{100}$	$\frac{100 - M_{ar}}{100}$	———	$\frac{100}{100 - A_d}$
Dry, Ash-Free (daf)	$\frac{100 - M_{ad} - A_{ad}}{100}$	$\frac{100 - M_{ar} - A_{ar}}{100}$	$\frac{100 - A_d}{100}$	———

To convert any of the analysis values for the parameters listed above from one basis to another, multiply the given value by the value shown in the appropriate wanted column; M_{ar} is the as-received or total moisture, M_{ad} is the as-determined or residual moisture, A_{ad} is the as-determined ash content, and A_d is the ash content on a dry basis. Moisture and ash content data should be in units of wt. percent.

APPENDIX E

Calculation of Total Moisture in an RDF Sample

A. Definitions

1. Total Moisture

A determination of the water contained in a sample in the as-received state. This determination is made by drying the sample in an atmosphere under rigidly controlled conditions of temperature, time, and air flow. This determination may consist of a single-step or a two-step drying process.

2. Air Drying

A process of partially drying RDF to bring its moisture content to near equilibrium with the ambient atmosphere in which further reduction, division, and characterization of the sample are to take place. In order to bring about this equilibrium, the RDF is usually subjected to drying under controlled temperature conditions ranging from 30 to 40°C.

3. Air Dry Loss (A.D.L.)

The decrease in mass due to air drying. This decrease is presumed to be moisture in the sample.

4. Residual Moisture

The moisture content remaining in an RDF sample after it has been milled to small particle sizes such as 2 mm or 0.5 mm. A certain amount of the residual moisture may be lost or picked up by the sample during the equilibration with the ambient atmosphere. Prior to milling, the RDF sample should have been subjected to either a total moisture determination (single-step), or an air drying procedure.

B. Calculation of Total Moisture

$M\ (\text{total}) = M\ (\text{residual}) \times (100-\text{A.D.L.})/100 + \text{A.D.L.}$ where M (total) is the total moisture in the sample, M (residual) is the residual moisture, and A.D.L. is the air dry loss; units should be expressed in wt. percent.

Acknowledgement

We wish to acknowledge the assistance of Mr. David J. Mitchell from the NBS Thermal Processes Division in the preparation of some of the RDF samples for calorimetric measurement, and in some of the determinations of moisture and ash content.

NBS-114A (REV. 2-8C)

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5. AUTHOR(S)

D.R. Kirklin, J. Colbert, P. Decker, S. Abramowitz, and E.S. Domalski

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☐ Document describes a computer program; SF-185, FIPS Software Summary, is attached.

11. ABSTRACT *(A 200-word or less factual summary of most significant information. If document includes a significant bibliography or literature survey, mention it here)*

This report provides the results of investigations during fiscal year 1979 to develop test procedures for the determination of the calorific value of refuse and refuse-derived-fuel (RDF) by means of oxygen bomb calorimetry. The results of 138 calorimetric measurements are discussed, along with 32 calorimeter calibration measurements, for 20 different RDF samples. In addition, determinations have been carried out on these RDF samples for air dry loss, residual moisture, furnace ash, bomb ash, and sulfur content to correct the calorimetric data for their presence; the latter group of measurements amounts to 283 experiments. The calorimetric (and related) measurements can be categorized into five classes: (1) measurements carried out in conjunction with ASTM round robin testing of RDF-3, (2) measurements on an Americology RDF extracted using both selective and non-selective sampling methods, (3) measurements required to compare data obtained from the small (2.5 gram capacity) and large (25 gram capacity) bomb calorimeters, (4) measurements carried out in the large bomb calorimeter on seven processed samples of different particle size obtained from New Castle County Delaware MSW, and (5) measurements on some RDF's of special interest. Five appendixes include additional information on: the evaluation of data on higher heating values obtained during ASTM round robin testing of RDF-3, the large bomb calorimeter, spectrochemical analysis of bomb residues, conversion tables, and calculation of total moisture in an RDF sample.

12. KEY WORDS *(Six to twelve entries; alphabetical order; capitalize only proper names; and separate key words by semicolons)*

ASTM round robin testing; bomb calorimetry; 2.5 and 25 gram capacity bomb calorimeters; higher heating value; refused-derived-fuel; sample characterization; sample processing effects

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An evaluation of data on higher heating values determined during the ASTM round robin testing of RDF-3 showed that the repeatability (within-lab agreement) and reproducibility (between-lab agreemtn) are 250 and 750 Btu/lb when values are converted to a dry basis. These variabilities are 5 and 7.5 times greater, respectively, than the precision levels accepted in ASTM protocols for determining the higher heating values of coal or coke on a dry basis (see Appendix A).

Measurements on three RDF samples in both small (2.5 gram-capacity) and large (25 gram-capacity) bomb calorimeters showed that equivalent high precision can be obtained in both calorimeters. The large bomb calorimeter has the advantage that it can accommodate RDF samples whose particle size is as large as 25 mm (1 inch).

The mean and standard deviation of the calorific values obtained for 20 different RDF samples give 9482 and 553 Btu/lb, respectively, on a moisture-ash free basis using the furnace ash method to determine the ash content. Thse results support the generally accepted value of 9500 Btu/lb as the higher heating value for RDF on a moisture-ash free basis.

DOA 204-1

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7. ITINERARY *(Point of origin and places to be visited)*

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8. PERIOD OF TRAVEL	a. BEGIN ON OR ABOUT 7-28-81	b. END ON OR ABOUT 7-29-81	9. ESTIMATED COST *(See 11, 12, 13 below)* 340.00

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Maximum prevailing rate & time of travel.

Note: Travelers authorized to operate a Government - owned or leased vehicle must possess SF-46, "U.S. Government Motor Vehicle Operator's Identification Card," in accordance with DAO 209-2, unless covered by special exception therein.

13. SPECIAL EXPENSES AUTHORIZED *(Check applicable blocks)*

a. ☐ Meeting registration fees

b. ☐ Hire of taxis between lodging and/or place(s) of business

c. ☐ Excess baggage, not to exceed________________pounds

d. ☐ Other *(Specify)*

14. SPECIAL PROVISIONS/REMARKS *(No. of Transportation Requests needed, justification of Air First Class, exception to Requirement for SF-46, etc.)*

I certify that the travel approved herein has been reviewed and determined to be essential for the accomplishment of agency programs and missions.

Travel voucher must be submitted within ______ days after completion of travel, and travel advances must be refunded at that time unless you have a standing travel advance.

15. CHECK APPLICABLE BLOCK → ☐ CHANGE OF OFFICIAL STATION ☐ REPORT TO FIRST PERMANENT DUTY STATION

It is hereby certified that this action is not primarily for the convenience or benefit of the employee, or at his request. Items b, c, d, and e, when completed, are authorized in accordance with Office of Management and Budget Circular, A-56.

a. TO *(Travelers new/first permanent station)*	b. TRANSPORTATION OF IMMEDIATE FAMILY →	NUMBER
c. SHIP HOUSEHOLD EFFECTS FROM *(Location)*	d. TO *(Location)*	e. EST. WEIGHT *(Pounds)*

16. SIGNATURE OF REQUESTING/APPROVING OFFICER Eugene Domalski	TITLE Acting Program Manager for Resource Recovery

17. Certificate of Authorization By Designated Authorizing Officer — You are hereby authorized to travel at Government expense under the conditions noted on this Order, and in accordance with the Standardized Government Travel Regulations. The number and approval date of this Order must appear on each voucher claiming reimbursement for expenses incurred consequent to this Order.

a. SIGNATURE OF AUTHORIZING OFFICER

b. TITLE *(Please type)* Chief, CCT	c. DATE ORDER APPROVED

July 28

LV National @ 5:30 PM via Eastern 199
AR Jacksonville @ 7:09 PM

July 29

LV Jacksonville @ 5:55 PM via PA 66
AR National @ 7:35 PM

Reservation made on beltway limo for return trip
(Call dispatcher 622-0700)

Holiday Inn - Orange Park
U.S. 17 @ I295
$25.00/night
904-264-9513

Meeting with Bill Roche - 904-772-2114. Meet him @ gate @ 9:30 AM