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U.S. DEPARTMENT OF COMMERCE
National Bureau of Standards
Chemical Thermodynamics Division
and the
Temperature and Pressure Measurements and Standards Division
Washington, DC 20234

June 1983

Prepared for
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Forrestal Building
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KILOGRAM-SIZE SAMPLES OF
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U.S. DEPARTMENT OF COMMERCE, Malcolm Baldrige, *Secretary*
NATIONAL BUREAU OF STANDARDS, Ernest Ambler, *Director*

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Abstract

A new calorimeter is being developed at the National Bureau of Standards to determine the enthalpies of combustion of kilogram-size samples of municipal solid waste (MSW) in flowing oxygen near atmospheric pressure. Experiments were carried out to develop a prototype combustor in which pellets of relatively unprocessed MSW can be rapidly and completely burned with minimal scattering of ash. Pellets of up to 2.2 kg mass with ash contents between 20 and 35 percent have been successfully burned at a rate of 15 minutes per kilogram initial mass with CO/CO₂ ratios not greater than 0.1 percent.

Keywords: combustor; municipal solid waste; oxygen combustion; refuse derived fuel.

Introduction

The Resource Conservation and Recovery Act of 1976 mandates the National Bureau of Standards (NBS) to publish guidelines for the development of specifications for the classification of materials which can be recovered and would otherwise be destined for disposal. Of particular national interest is the desire to produce useable energy from such sources as refuse and refuse-derived-fuel (RDF).

At present, the calorific value of RDF is determined at commercial laboratories with conventional combustion bomb calorimetric techniques using gram-size samples. Such samples are prepared by reducing the bulk RDF to a powder of 0.5 mm particle size (RDF-4). Substantial processing is required to obtain the gram-size sample and there is concern among some combustion engineers that this processing may significantly alter its calorific value. There are also questions about sampling procedures and how representative such a gram-size sample can be of the waste stream.

To address these doubts, NBS has developed a calorimeter to determine the calorific value of kilogram-size samples of dried RDF-2. RDF-2 is municipal solid waste which has been processed to reduce the particle size so that 95 mass-percent passes through a 15 cm square mesh screen. Kilogram-size samples of RDF-2 should represent the properties of the heterogeneous bulk material more accurately and reliably than the highly processed gram-size samples currently used in bomb calorimetric determinations.

For safety considerations, we chose to build a calorimeter for the com-

bustion of samples in flowing oxygen near atmospheric pressure rather than to scale up the conventional high pressure oxygen combustion bomb. Unfortunately, after the 1880's the development of the flow technique to determine the enthalpy of combustion of solids was discontinued in favor of the simpler procedures and more quantitative results which could be obtained with the bomb calorimeter. As a consequence, the first goal of the NBS project was to demonstrate that the oxygen flow technique could be used to obtain complete combustion of municipal solid waste (MSW).

A small combustion flow calorimeter has been successfully used to burn 25 gram pellets of the highly processed MSW prepared for combustion bomb experiments [1,2]. The amount of carbon in the ash and the uncertainty in the amount of CO in the combustion products contributed errors of less than 0.1% to the enthalpy of combustion.

Before the design of a large-scale combustion flow calorimeter could be undertaken, it was necessary to determine the burning characteristics of the kilogram-size RDF-2 pellets in oxygen. The composition of the kilogram-size pellet is more variable and the physical heterogeneity measured against the sample size is greater than the 25 gram pellet of highly processed RDF-4. As a consequence its combustion properties were expected to be different.

The goal of the trial experiments was to develop a method for controlled burning of the sample pellets at a rate of 15 minutes per kilogram or less. This time limit was estimated to be the maximum time which would guarantee that the imprecision contributed by the calorimetric measurements would be less than one percent.

Apparatus and Samples

Trial combustions of samples up to 2.2 kg of RDF-2 were carried out in a burner cooled by convective and radiant heat losses to ambient temperature. The burner was mounted in a large exhaust hood. The burner design was similar to that used in the 25 gram experiments in that thermal shields were employed to keep the hot, reacting, combustion product gases from being cooled by contact with the outer burner walls. The combustor differed from that used in the 25 gram experiments in that all of the oxygen was supplied locally to the sample; no oxygen flowed between the thermal shield and the cool combustor walls. Oxygen was supplied in the form of high velocity jets which were directed either at the top of or the side of the sample or both, depending on the experiment. A diffuse, slower, flow of oxygen was directed upward at the bottom of the sample.

The combustor, as it appeared in the first of the trial burns, is shown in cross section in Fig. 1. The unit consists of two chambers. The lower chamber, which was the cylindrical region enclosed by the lower burner jacket (E of Fig. 1), contained the sample and two oxygen supply inlets. The sample was supported on vertical stainless steel pins which fit into the holes of a perforated stainless steel plate (H). Primary oxygen was supplied to the bottom of the sample from the inlet I. Secondary oxygen was directed at the top of the sample by three jets aimed radially and horizontally by the inlet F. The intent of the sample-oxygen supply arrangement was to mimic as far as possible the 25 gram flow experiments while maintaining an unobstructed view of the combustion. Hence, no crucible was used.

The thermal shield (B) of the upper chamber and upper burner jacket (C) sat on a annular steel plate (D) which was supported by three rods (G) that projected through the lower chamber to the concrete floor. The annular plate also supported the lower burner jacket which was bolted to the plate around two-thirds of its circumference.

For access to the lower chamber, one-third of the circumference of the lower burner jacket was a semi-cylindrical door. Two Pyrex glass viewing ports (not shown in Fig. 1) were installed in the door so that the course of a combustion could be observed.

Oxygen was supplied to the primary and secondary inlets in the lower burner jacket by independent sources, each consisting of three standard 6,200 liter (STP) oxygen tanks equipped with reducing valves and connected in parallel. Flow rates were measured with variable orifice flowmeters. A product gas analysis train similar to that used in the 25 gram experiments was used to monitor the CO and CO₂ production. The product gases were continuously sampled at a constant flow rate of 5 L/min through a stainless line whose entry port was centrally located in the top of the upper combustion chamber. The gases were cooled to room temperature by passing them through a helical heat exchanger immersed in cold water and then dried by passing them through a drying tube containing anhydrous CaSO₄. CO and CO₂ concentrations were measured using two dedicated infrared detectors which were equipped with appropriate interference filters. The detectors were calibrated with standard gas mixtures of 0.1, 0.3, 1.6, 1.0 and 2.0 mol% CO in nitrogen and 10 and 20 mol% CO₂ in oxygen respectively. The pressure in the product gas line at the CO and CO₂ detectors was measured with a digital capacitance manometer.

Temperatures were monitored at as many as twelve different locations using 18 gauge Type K (Chromel P - Alumel) thermocouples.

The RDF-2 from which the sample pellets were made was obtained from the Teledyne National Resource Recovery Facility in Cockeysville, Maryland where municipal solid waste of Baltimore County is processed. Lots were withdrawn at random from the conveyer belt leaving the primary shredder. At NBS, the large noncombustibles were removed and the remainder was dried in air at 105°C for 12 to 16 hours. Sample pellets were made by compressing the dried RDF-2 in a cylindrical die piece with a force ranging from 265 to 625 kN (30 to 70 tons). A single compression yielded a pellet with reasonably good adhesion of the various heterogeneous horizontal layers. This adhesion was not improved by wetting the material with water prior to pressing. The finished pellet had a diameter of 22 cm and a height of about 6 cm/kg of sample mass. To test the effect of increased surface area, three or seven vertical holes were drilled in some of the pellets using a metal drill and jig to hold the sample. One cellulose pellet was made by pressing pure cellulose fluff using the same technique.

In all, eighteen experiments were carried out to test the effectiveness of: (1) various arrangements of primary and secondary oxygen inlets, (2) pre-heating the oxygen, and (3) reducing heat losses from the sample by the use of a crucible and a radiation shield. Thirteen experiments were run with RDF-2 pellets. Five experiments were run with pure cellulose or its equivalent (a stack of unglazed paper plates).

Changes in the apparatus made as a result of these tests are illustrated

by Fig. 2 which shows the configuration of the lower chamber of the combustor used in the final two experiments. An RDF-2 sample with seven vertical 2.5 cm diameter holes sat on a horizontal lattice of alumina rods which rested on a stainless steel support (D of Fig. 2). Two tiers of secondary oxygen inlets (B and C) were aimed radially inward and horizontally at the side of the pellet. The lower tier of six inlets was supplied with oxygen that was preheated by passing it through coiled tubing (F) which was wound on the outside of the crucible. The upper tier of three inlets was supplied with oxygen that was preheated by passing it through the coil (I) which was inside of the crucible. Oxygen was supplied to the bottom of the sample from the primary oxygen inlet (E). Most of the ash falls through the center of the inlet to the bottom of the crucible. A radiation shield which just fits the inside diameter of the annular steel plate (D of Fig. 2) has been placed around the crucible to reduce heat losses.

Back flow of the product gases between the crucible side and the lower thermal shield was prevented by a low upward flow of diffuse oxygen from the multiport ring inlet (G of Fig. 2). Rectangular openings were cut in the crucible and thermal shield in order to observe the combustion. A Pyrex window covered the opening in the thermal shield.

Description of a Typical Experiment

In a typical experiment, the combustor was flushed with oxygen for ten minutes. The sample was then ignited by passing electrical current through an iron fuse wire (H of Fig. 2) touching the top of the sample. The flow rates were adjusted to preselected initial values for the experiment. Primary and

secondary oxygen flows were varied to study the effectiveness of different rates and inlet arrangements. The ratio of diffuse to directed flow was of the order of 3:1 or greater and the total flow rate ranged between 3.5 and 11 moles per minute. At the end of the burn, the combustor was flushed with oxygen and allowed to cool. The ash was collected and weighed. The ash contents of RDF-2 pellets ranged from 15 to 32% of the initial mass and no unburned organic material was identified in the ash.

In general, the most rapid burning of the sample occurred on the areas where the secondary jets of oxygen struck the sample. As the secondary flow velocity was increased, these areas became white hot and the flame became ever more turbulent. Above a critical flow rate, the sample began to fragment vigorously with significant scattering of burning matter and ash. As the temperature of the preheated secondary oxygen was increased, the flow rate at which these changes occurred was lowered. Intense burning occurred in the vertical holes, when present. Bright columns of flame were observed above the holes.

CO production tended to be larger during the combustion of the last quarter of the sample. The fraction combusted at any time was assumed to be the ratio of CO₂ produced up to that time divided by the total CO₂ production. The ratio of total CO to CO₂ ranged from 2 to less than 0.1 mol%. The time required to burn the last quarter of all RDF-2 samples was always longer than that required to burn the first three quarters of the sample. This was due to the presence of noncombustibles which tended to inhibit the combustion of the last quarter of the unburned material. Peak temperatures of the exhaust gases at the exit stack ranged from 365 to 500°C, depending upon the experiment, and

occurred before half of sample had been combusted. The flame temperature above the sample was determined to be in excess of 1400°C. The peak temperature of the combustor components nearest the sample ranged from 500 to 1200°C for the top of the primary oxygen supply and 440 to 500°C for various parts of the crucible and lower thermal shield. For the most part, only minor surface corrosion of these components occurred. Actual ignition of the combustor components only took place when the burning sample fell from its support and came into contact with the oxygen inlets.

Summary of Results

The total burn time of RDF-2 pellets was reduced from 77 minutes per kilogram initial mass in the early runs with the experimental arrangement illustrated in Fig. 1 to an acceptable 15 min/kg using the arrangement of Fig. 2. As more than one change in sample and/or combustor configuration was made in each trial experiment, interpretation of the effects due to the individual changes tends to be somewhat ambiguous. However, we draw the following conclusions: (1) The introduction of a crucible (having a side wall one half the height of that shown in Fig. 2) caused a reduction in burn times for RDF-2 pellets of 33%. (2) The introduction of vertical holes reduced burn times between 10 and 50%. (3) Burn times for a single tier of lower secondary oxygen inlets (equivalent to C of Fig. 2) were about 50% less than those obtained using a single tier of upper secondary inlets (which were directed downward at a 45° angle toward the top edge of the pellet). (4) We found that cellulose pellets were easily ignited and burned smoothly leaving negligible ash. Burn times for cellulose samples were up to 50% shorter than those for RDF-2. Cellulose appears to be a satisfactory potential calibrant.

Using the results of this and earlier work [1,2], the design of the final kilogram-capacity calorimeter has been completed. Assembly started in January, 1982.

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Fig. 1. The 2.5 Kilogram Combustor. A denotes the stack, B the upper thermal shield, C the upper burner jacket, D the annular plate, E the lower burner jacket, F the secondary oxygen inlets, G the support rod, H the sample support and I the primary oxygen inlet.

Fig. 2. The Configuration of the Lower Chamber of the Combustor for Experiments 17 and 18. A denotes the lower thermal shield, B the upper tier of secondary oxygen inlets, C the lower tier of secondary oxygen inlets, D the sample support, E the primary oxygen inlet, F the lower tier preheat coil, G the multiport ring oxygen inlet, H the iron fuse, I the upper tier preheat coil and J the crucible lid.

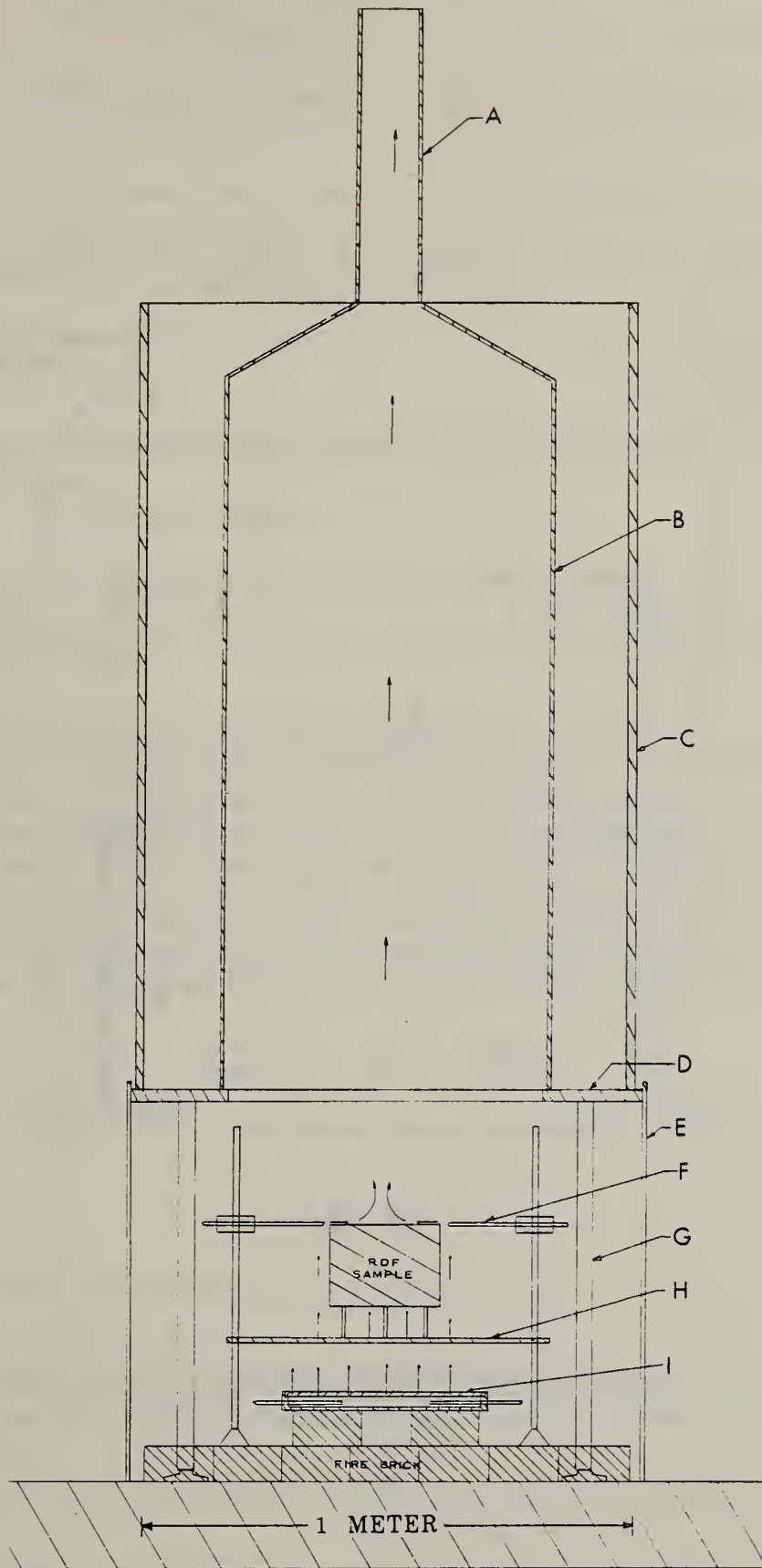
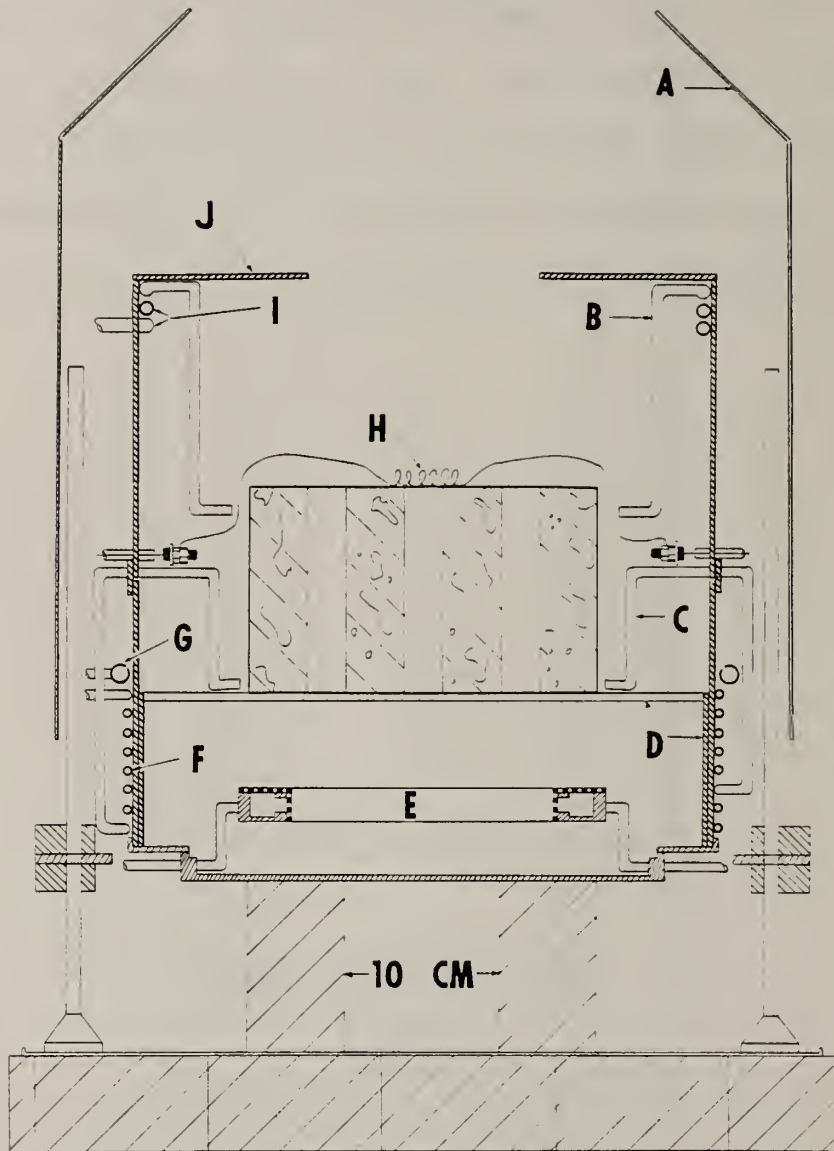


Fig. 1.



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