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INTERLABORATORY COMPARISON OF THE POTENTIAL HEAT METHOD

by

D. Gross

NBS

U.S. DEPARTMENT OF COMMERCE
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ABSTRACT

Quantitative measurements of the total heat release from eight selected building materials were made during an interlaboratory study of the Potential Heat Test Method. Statistical analysis of the results indicates that a higher degree of variability existed among the nine participating laboratories than would be expected of a single laboratory performing standardized oxygen bomb calorimeter determinations of the heat of combustion. It was noted that the majority of laboratories tended to rank the materials in the same order, or in an order which could be made the same by a ranking change of a single material.

A discussion is presented of the effects of material sampling, of the amount of combustion promoter used, and of specific details of the experimental procedure. Suggestions are made for evaluating the causes of interlaboratory differences and reducing their magnitude in a follow-up study.

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Introduction

In March 1965, an interlaboratory comparison study of the potential heat test method was initiated by a Task Group of Subcommittee V (Definitions and Nomenclature) of ASTM E-5 (Fire Tests of Materials and Constructions). One goal of the Task Group was to evaluate the suitability of the test method for measuring and classifying materials according to their degree of combustibility.

The test method had been developed several years earlier [1] to measure the total heat released under typical fire exposure conditions, but without regard to the rate at which the heat was released. The method makes use of standard calorimetric techniques in which the burning of small quantities of combustible in an otherwise inert material is assured by use of a combustion promoter which is added prior to test. By measuring heat of combustion in an oxygen bomb calorimeter both before and after exposure to a "standardized fire" (2 hr in air at 750 C), the difference may be considered as the potential heat of the material.

The Task Group consisted of the following members:

Mr. I. A. Benjamin, Granco Steel Products Co., Chairman
Mr. P. E. Baseler, Building Officials Conference of America
Mr. D. Gross, National Bureau of Standards
Dr. J. R. Jutras, National Research Council of Canada
Mr. J. A. Wilson, Factory Mutual Engineering Division.

The NBS Fire Research Section undertook the task of mailing specimens to the participants in May 1965, and the test results were returned in the period July to December 1965.

Participants

A total of nine laboratories, including the National Bureau of Standards, participated in the study. These were:

Ohio State University
Underwriters Laboratories, Inc.
U. S. Forest Products Laboratory
Fire Research Section, National Research Council of Canada
Johns-Manville Fiber Glass Division
Owens-Corning Fiberglas Corporation
Factory Mutual Engineering Division
U. S. Bureau of Mines

Test Procedure

Details of the test procedure were described in a published article [1] and in a tentative test method standard (see Appendix A) distributed to the participants. Prior to initiating the study, the ASTM Manual on interlaboratory studies [2] was consulted and advice was solicited from the NBS Statistical Engineering Section.

To obtain a broad base of experience with a minimum of participation time, it was suggested that the pilot study would provide satisfactory estimates of precision without the need for duplicate determinations. Except for preliminary experimentation for orientation in techniques of bomb calorimetry, therefore, a single determination on each of eight materials was requested, with additional testing (to provide duplicate data) to be undertaken only if necessary.

The following were also distributed to the participating laboratories:

(1) a reference material (for practice and test method familiarization;
(2) a set of supplementary notes and instructions (see Appendix B);
(3) a summary data sheet;
(4) a questionnaire
(5) packages containing the samples, a specimen container and cap, and a wire specimen holder.

Materials

A list of the eight materials selected is given in Table 1. These were considered typical composite materials used in buildings at the present time. The test materials were designated by code letter only, and no producer identification was intended or implied. Asbestos millboard, designated M and conforming to Federal Specification HH-M-351 b, was the reference material. All samples were cut from larger sheets or blocks in a pre-arranged manner and a record kept of the sampling location.

Results

Table 2 is a summary of the replies to the questionnaire. There were three requests for code letter identification of participating laboratories, and, by prior agreement, only one was necessary to carry. The code letter S was given to this laboratory (NBS). As to whether the individual lab's values and the ranking order of the materials was realistic, four indicated yes and five were undecided. Comments on the basic theory and application, difficulties encountered, and suggested revisions, are also summarized. With regard to theory and application, several laboratories commented that the method does not evaluate the rate at which heat is released. As to experimental difficulties, several laboratories noted instances of incomplete combustion, and there were questions as to preparation of representative samples.

Table 3 summarizes individual values of the heat of combustion of the specimen (direct), the heat of combustion of the residue (after simulated fire exposure), and the potential heat. For these materials, the heat of combustion of the residue was very significant in the determination of potential heat. Vacant spaces represent the absence of data. In several instances, as noted, arithmetic or typographical errors were found or assumed, and values adjusted.

Table 4 summarizes the percent residue values for all materials and laboratories, and illustrates, with few exceptions, the good reproducibility of this determination. For materials C, D, and E, the percent residue values agreed to within ±0.5 % for all laboratories.

Table 5 lists, for each laboratory, average (or typical) values of test parameters which affect the direct measurement of the heat of combustion. From this list, and the individual values on which they were based, certain points may be noted:

1. It appeared that the incremental temperature readout varied from 0.005 to 0.02 F. For an average calorimeter water equivalent of nearly 2500 Btu/deg F (g/lb);* a change of 0.02 F corresponds to a change of 50 Btu/lb.

2. The fuse wire correction varied from 20 to 182 Btu-g/lb.* (The latter value greatly exceeds the total calorific value of the prescribed length, usually 10 cm, of No. 34 B & S gage Chromel C or iron fuse wire).

3. The acid titration correction for material C varied from 0 to 94 Btu-g/lb. Since material C contained approximately 16% sulfur, an appreciable correction was to be expected.

4. The temperature of the calorimeter water ranged from 3.1 F below to nearly 12 F above the jacket at the start of the test.

5. The individual percentage values of combustion promoter used varied from 0 to 91. However, only laboratory Z used a consistently different (higher) proportion of combustion promoter than called for. This was done to prevent incomplete combustion of specimens containing the prescribed amount (50%) of promoter.

One laboratory (V) separated composite materials C, E, and H into their two layered components and tested each component separately. The contributions of heat were then combined in proportion to their original weight fractions.

*For explanation of units, see footnote, Table 5

Statistical Analysis

Because the results were variable and the materials so different, it was decided to present the results as deviations from the median value for each material rather than in terms of a conventional analysis of variance. The median value is the value such that half the results are smaller than, and half the results are larger than the median. The median was used rather than the arithmetic mean because it is less influenced by outlying values.

Table 6 summarizes the results of the direct and residue heat of combustion measurements, showing the median value for each material and the deviation of each laboratory from this median value. The first line below the tabulated values gives the sum of such signed deviations for each laboratory. This sum (or the equivalent average) is some measure of the apparent bias of a particular laboratory - i.e., its sign and its relative magnitude show the level of a laboratory with respect to the rest of the group. The next line below the tabulation gives the sum of the absolute deviations for each laboratory, and is a comparative measure of the spread of a laboratory's results.

The deviations listed in Table 6 are plotted in Figure 1 for each material and laboratory. The materials have been arranged along the horizontal axis in increasing order of the median value of the material. It may be noted that the dispersion does not seem to depend upon the magnitude of the median heat of combustion value.

The magnitudes of the interlaboratory deviations suggests that differences between duplicate determinations by an individual laboratory may be higher than 100 Btu/lb, the value derived from the initial test method evaluation [1]. A conventional heat of combustion measurement is expected to duplicate within 0.3 percent within the same laboratory, and within 0.5 percent at different laboratories [3]. For judging the acceptability of test results obtained with a bomb calorimeter (95 percent probability), the following limits are specified [4]:

> "Repeatability - Duplicate results by the same operator shall be considered suspect if they differ by more than 55 Btu/lb.
>
> "Reproducibility - Results submitted by two or more laboratories should be considered suspect if they differ by more than 175 Btu/lb."

Since this test method requires the mixing of a combustion promoter with the specimen, as well as reporting the difference of direct and residue bomb calorimeter measurements and accounting for weight changes during the muffle furnace exposure, errors of somewhat greater magnitude are to be expected. It appears highly desirable that duplicate determinations be included in the next phase of interlaboratory comparisons.

A simplified overall summary of the interlaboratory comparison may be obtained by the comparison of material rankings listed in Table 7. Three of the nine laboratories ranked the eight materials in exactly the same order, corresponding to the overall consensus. Considering materials E and F to be "identical" for the purposes of the consensus (overall average values 1123 and 1093 Btu/lb), a ranking change of a single material would bring 3 more laboratories into agreement with the consensus.

Discussion

1. Sampling

Several laboratories expressed concern about the preparation of representative samples from the heterogeneous engineering materials used and the fact that the direct bomb calorimeter specimens weighed only one gram. A measure of the extent of this problem might be gaged by (a) variability in the direct bomb heat of combustion, and (b) variability in the percent residue values for the muffle furnace specimens, since this is a measure of the combustible and volatile content. For materials B (mineralized wood) and G (cinder aggregate concrete), there was considerable variability in the direct bomb heats of combustion. For material B, five of the laboratories cluster very close to the median value, while four laboratories are extreme "outliers." This characteristic variability for material B may possibly be associated with a differential separation of the components during specimen preparation by some laboratories and suggests that details of the specimen preparation need to be defined more closely.

For material B, 8 of 9 laboratories measured the residue within fairly close limits, 44.2 to 46.4 percent. Only for material G was there an appreciable variation in the percent residue values, ranging from 58.7 to 82.4. For all other materials there was very good agreement in the weight loss during the muffle furnace exposure (See Table 4).

It is also interesting and informative to note that the separation of composite materials C, E, and H into their two (or three) layered components, and their separate testing by laboratory V, yielded consistent and meaningful heat of combustion values, when their heat contributions were combined in proportion to their original weight fractions.

With one exception, there did not appear to be any systematic correlation between the high (or low) heat of combustion values measured by some laboratories and the location of the sample as cut from its original large sheet or block. For material G (cinder aggregate concrete),however, there was a significant difference between the direct heat of combustion values as obtained from the two 6-5/8-by 16-in. blocks from which the samples were cut. Figure 2 illustrates how the individual samples were distributed among the nine laboratories, and the resultant direct heat of combustion values measured. The average for laboratories Q, R, S, T, and U from one block was 1735 Btu/lb, compared to 2578 Btu per lb for laboratories V, X, Y and Z from the other block.

For most engineering materials and products, the use of a prescribed sampling procedure is probably not necessary provided the 1 gram (or 1/2 g) direct bomb specimen is obtained from the intimately-mixed powder of a pulverized piece of minimum size 1/2 in. by 3 in. by nominal thickness. For typical building materials with densities ranging up to 120 lb per cu ft, this piece would weigh between 5 and 20 grams. A change in the sampling procedure is therefore suggested (see Appendix C) to require this minimum sample size to be pulverized for the direct heat of combustion measurement. For grossly heterogeneous materials, e.g. cinder aggregate concrete, bulk sampling procedures are obviously necessary if more consistent interlaboratory results are to be achieved.

2. Combustion Promoter

Several laboratories noted instances of incomplete combustion. Laboratory Z used a higher proportion of combustion promoter than that prescribed (50 percent) in order to avoid incomplete combustion. The use of an equal weight of benzoic acid promoter was based on a prior investigation in which less than 30 percent benzoic acid mixed with aluminum oxide was found to result in incomplete combustion [1].

In Figure 3 are plotted the specimen and residue heats of combustion for the one material (E, protected steel) for which benzoic acid promoter in a very wide range had been used. This data suggests the possibility that 50 percent benzoic acid may not be quite sufficient to ensure complete combustion of some materials.

3. Experimental Procedures

It is evident from the comments provided, as well as from the summary of typical test parameters shown in Table 5, that certain procedures and measurements must be closely controlled, while others are of much less importance.

In addition to representative sampling and the use of a minimum of 50% of combustion promoter, the following items require particular care and accuracy:

(a) mechanical or manual pulverization by grinding, filing, etc. and intimate mixing, so as to avoid segregation or separation of components (see Appendix C);

(b) accurate weighing and careful accounting for losses during the preparation of pellets;

(c) temperature measurement to 0.005 F, or less;

(d) proper fuse wire and acid titration corrections.

The (plain) bomb calorimeter test procedure recommends that the temperature of the water be initially 3 to 3.5 deg F below that of the calorimeter jacket. Assuming the jacket to be at room temperature, the initial water temperature ranged from 3.1 deg F below to nearly 12 deg F above that of the jacket, but no systematic effect on the results could be noted.

Only one laboratory (T) made allowance for the quantity of sulfur (approx. 16 percent) in material C. However, since allowances of similar magnitude occur for both the direct and residue tests, the difference in the potential heat was not significant.

Future Work

A second phase of the potential heat interlaboratory study, which would incorporate the specified changes in the test procedure, is desirable. For 5 selected materials, and a minimum of 4 laboratories, a test plan is listed in Table 8 which would permit evaluation of the percent combustion promoter as well as repeatability within each laboratory and reproducibility between laboratories. Each participant would be requested to perform a total of 12 tests. In all such testing programs, comments and feedback from the individual participants is very important.

Summary and Conclusions

Valuable experience has been gained from this initial interlaboratory comparison of the Potential Heat Test Method. Of the 9 participating laboratories, some were experienced in oxygen bomb calorimetry, while others were novices. Major and minor variations were made by individual laboratories in many aspects of the testing procedure. In addition, the majority of the 8 test materials, selected as typical of composites used in buildings at the present time, were of generally low potential heat as a critical test of the method.

A higher degree of variability existed among the participating laboratories than would be expected of a single laboratory performing standardized oxygen bomb calorimeter determinations of the heat of combustion. However, the majority of laboratories tended to rank materials in the same order, or in an order which could be made the same by a ranking change of a single material.

Based on a study of the interlaboratory differences, and the comments provided by the participating laboratories, it is suggested that more specific test instructions and evaluations be provided in future test programs. These include material sampling, the proportion of combustion promoter, close control of pulverization, weighing and temperature measurement, and the requirement for duplicate dterminations. Specific suggestions were made in the "Discussion" and "Future Work" sections, and by revisions in the Tentative Test Method (Appendix C).

Acknowledgments

This study was made possible through the excellent and prompt cooperation of the laboratory and research workers in each of the participating laboratories. The generous help of Mrs. M. G. Natrella and Dr. W. J. Youden of the NBS Statistical Engineering Section in the statistical analysis and experimental design aspects is gratefully acknowledged.

References

[1] Loftus, J. J., Gross, D., and Robertson, A. F., Potential Heat - A Method for Measuring the Heat Release of Materials in Building Fires, ASTM Proc. 61, 1336-48 (1961).

[2] ASTM Manual for Conducting an Interlaboratory Study of a Test Method, ASTM STP No. 335 (1963).

[3] Standard Methods of Laboratory Sampling and Analysis of Coal and Coke, ASTM Designation D 271-64, 1966 Book of ASTM Standards, Part 19.

[4] Standard Method of Test for Heat of Combustion of Liquid Hydrocarbon Fuels by Bomb Calorimeter, ASTM Designation D 240-64, 1966 Book of ASTM Standards, Part 17.

Appendix A. Tentative Method of Test For

POTENTIAL HEAT RELEASE OF MATERIALS IN BUILDING FIRES

Scope

1. This method of test provides a means of determining the potential release of heat of materials involved in building fires. The method is applicable to a variety of materials including metals, and especially materials of low combustible content. Determinations may be made on simple materials, or on composite assemblies of materials from which a representative sample can be taken and pulverized into a homogeneous mixture.

Definition

2. Potential heat of a material is the difference between the heat of combustion of a representative sample of the material and the heat of combustion of any residue remaining after exposure to a simulated standard fire, using combustion calorimetric techniques.

Summary of Method*

3. One of two specimens removed from the material to be tested is pulverized, pelleted, and burned in a high-pressure oxygen atmosphere. The process is generally as described in ASTM D 271-58 (Laboratory Sampling and Analysis of Coal and Coke), but with certain modifications or permissible exceptions, to be noted in the test procedure. This determines the gross heat of combustion of the material. The second specimen is heated in air for 2 hr at a temperature of 1382 F (750 C), conditions adopted as representing a standard fire exposure. A portion of the resulting residue of this specimen, if any, corresponding to a predetermined weight of original material, is ground or pulverized, mixed with a combustion promoter, and pelleted for burning as was the first specimen. After correcting for the heat produced by the combustion promoter, the difference in heating values of the two specimens is the potential heat, as defined in par. 2. The test procedure is illustrated schematically in Fig.1A.

Apparatus and Materials for Test

4. The apparatus and materials required for the test are listed below.

(a) Oxygen bomb calorimeter, including firing circuit and fuse wire.

(b) Muffle furnace (having small opening or port for passage of air supply tube).

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* See Reference [1].

(c) Hand mill (or ball mill).

(d) Pelleting press.

(e) Microbalance, weighing to 0.1 mg.

(f) Oxygen cylinder and accessory equipment.

(g) Compressed air supply.

(h) Standard alkali solution.

(i) Combustion promoter, National Bureau of Standards standard material, sample 39 h, benzoic acid (calorimetric standard).

(j) Parts associated with muffle furnace firing (illustrated in Fig. 2).

(1) Specimen container for use in the muffle furnace; a suitable part may consist of a fused silica or ceramic tube, 1-1/4 in. inside diameter by 4 in. long, closed at one end.

(2) Cap to fit open end of specimen container; cap to be provided with an opening to pass an air supply tube with loose fit.

(3) Air Supply tube; may be of porcelain, fused silica, or corrosion-resistant metal; inside diameter 3/16 in. minimum; length to extend beyond port of muffle furnace.

(4) Wire specimen holder; of corrosion-resistant metal; formed to support the specimen away from the walls of the specimen container, for circulation of air about specimen.

(5) Support of fire brick or similar material shaped to hold the specimen container and its cap in alignment with the muffle furnace port, so that the air supply tube may be positioned in the specimen container.

Test Specimens

5. Two air-dry test specimens representative of the material or assembly involved are required for each determination. A specimen is considered "air-dry" when it has reached constant weight in an atmosphere maintained at 73 ± 2 F (23 ± 1 C) and 50 ± 5 percent relative humidity. If the test subject is an assembly or composite material, it is essential that the several elements of the subject are contained in the test specimen in the same proportions that they occur in the subject. The two specimens are subjected to separate test procedures.

Procedure for Direct Bomb Test

6. (a) The specimen shall be pulverized, using a hand mill or other means as may be necessary, into a form suitable to pass a No. 60 sieve.

(b) A pellet, weighing approximately 1 g., shall be prepared from a representative sample of the powder, and then weighed.

Note 1: All weight measurements shall be to the nearest 0.1 mg.

Note 2: Pellets shall be made in accordance with the method for the particular pelleting press in use and of a size convenient for the specimen cup. The pellets shall be no harder than necessary to prevent their disintegration when fired.

(c) The prepared pellet is the test specimen for the procedure for determination of the heat of combustion in accordance with ASTM D271-58, par. 51-55 (oxygen bomb test). Also see ASTM E-144, Recommended Practice for Safe Use of Oxygen Combustion Bombs.

Note 3: Modifications and exceptions to the requirements of ASTM D271-58 are listed in the Addendum.

(d) If, after being fired in the oxygen bomb, the pellet is found to have burned completely, or to have left no significant amount of residue or ash, the heat of combustion on an air-dry basis may be computed, and the following three steps (e), (f), (g), shall be omitted.

(e) If the pellet does not burn, or a residue remains after the firing, another 1-g pellet is prepared, this time using an intimate mixture of the powdered sample and a standard sample of benzoic acid combustion promoter, in equal weight proportions.

(f) The pellet prepared with the added benzoic acid is used as the test specimen following the same procedures as for the original specimen.

(g) A correction for the heat of combustion of the benzoic acid present in the pellet is applied to the measured heat released by the specimen. The heat of combustion of the sample material, on an air-dry basis, is then computed.

Procedure for Muffle Furnace and Bomb Test

7. (a) An air-dry specimen representative of the test material or assembly shall be cut in the form of a rectangular prism 1/2-by 3/4-by 3-in. Sheet materials may be folded or laminated to these dimensions.

(b) The muffle furnace is preheated to 1382 ± 18 F (750 ± 10 C). The specimen is weighed, and placed on the wire support 4. (j) (4) in the specimen container 4. (j) (1). The container is closed with its cap 4. (j) (2), and placed in the firebrick base 4. (j) (5) in the muffle furnace in such position as to align the muffle furnace port and the opening in the specimen container cap. The external air supply tube 4. (j) (3) is passed through the port into the container in proximity to the specimen. Firing is continued for 2 hr with a regulated air flow of 0.1 ft^3 per min, referred to 60 F and 30.0 in. Hg, supplied to the specimen. If ignition should occur immediately upon placing the specimen in the furnace, application of air shall be delayed until the initial flaming has stopped.

(c) The container with the specimen shall be cooled in a desiccator, after which the weight of the residue is determined.

(d) If the residue from the muffle firing procedure is less than 5 percent of the initial weight of the specimen, the following steps (e) and (f) are omitted, and the heat of combustion previously determined under the direct bomb test, par. 6 (d), shall be reported as the potential heat of the material.

(e) If the residue after the muffle firing is in excess of 5 percent of the original specimen weight, the residue shall be pulverized, mixed with an equal weight of benzoic acid and treated as specified in the procedure for direct bomb test to determine the heat of combustion (of the residue).

(f) To determine the heat of combustion of the residue per unit weight of original specimen, multiply the heat of combustion determined in par. 7. (e) above by the ratio of residue weight, 7. (c) to the original specimen weight 7. (b).

Potential Heat

8. The potential heat of a material is determined by subtracting the heat of combustion of the residue remaining from the muffle furnace firing, par. 7. (f), from the heat of combustion of the material established in the direct bomb test, par. 6 (g). The potential heat is thus a measure of the heat released by a material in the muffle furnace firing, the conditions of which are considered to simulate a standard fire. For most materials,

potential heat may be reported in heat units per unit weight, or where appropriate to the material and its use, it may be expressed on the basis of volume or surface area of the material. For materials such as metals where the combustion process is relatively slow, and is a function of surface area, potential heat should appropriately be reported on a surface area basis only.

ADDENDUM

Modifications and exceptions to the requirements of ASTM D 271-58 are as follows:

(paragraph references apply to the standard as presented in the 1961 Book of ASTM Standards).

par. 52 (3)	Fuse wire may be that provided for the bomb in use; a suitable correction factor for the wire shall be applied.
par. 53 (b)	Benzoic acid is a suitable substance for standardization procedures (see 4. (i) of this standard).
par. 54 (a)	Alternate method of Note 32 for materials of high ash content not used; in cases of incomplete burning, a combustion promoter is added (see 6.(e)).
par. 54 (f)	An ignition system supplied for the bomb may be substituted.
par. 54 (h)	Where materials leave a residue, remove the cup containing the residue, then proceed to rinse out the bomb and titrate as described in this paragraph.
par. 55 (Note 40)	The method of this test for potential heat release of materials gives the gross heat of combustion of a material in an air-dry condition; net calorific value (net heat of combustion) calculations are not normally a part of this procedure. (see footnote*).

Appendix B. Supplementary Notes and Instructions

1. To permit statistical interpretation of variations in test results, the samples have been cut from the larger sheets in a pre-arranged manner. It is requested that the following directions be followed:

   For the Muffle Furnace Test, cut a test specimen from the end opposite to the label location. Remove sufficient material from the newly exposed edge for the Direct Bomb Test. This may be done using a clean file, rasp or other device and the filings pulverized, if necessary, using a mortar and pestle, ball mill, etc. Use the edge closest to the label for trial runs, if desired. The remaining sample should be retained for possible future comparisons or for use as reference materials.

2. Please note the following change in test procedure [Par. 7(a)]:

   The specimen for the Muffle Furnace Test should be 1/2" wide, 3" long and the <u>thickness as supplied</u>, but not greater than 1 in. Sheet materials should be folded (or laminated) to these dimensions (1/2" by 1" by 3").

3. Standard grade benzoic acid for use as a combustion promoter and for standardizing the oxygen bomb calorimeter is available for purchase from the oxygen bomb manufacturers or from chemical supply companies. A primary standard benzoic acid powder may be purchased from the Standard Materials Unit, Attn: Mrs. Pauline Michael, National Bureau of Standards, Washington, D. C. 20234. This is currently identified as Calorimetric Standard Sample No. 39i, and is priced at $6.00 for a 30 gram sample.

4. Caution should be observed when performing a direct bomb calorimeter test on a specimen which is predominantly metallic (One material in this series). It is possible that high reaction temperatures will occur which could involve portions of the capsule and bomb, or that an electrical short in the ignition system may occur through the conventional steel capsule. For such tests, the use of a silica combustion capsule is recommended (c.f. A.H. Thomas Co., Cat. No. 3879-C).

5. A suitable air supply tube could be one of the following:

   (a) Vycor 7mm i.d. standard tubing (Corning No. 743172, or equivalent)
   (b) Porcelain 7mm o.d. protection tubing (McDanel No. PT5M, or "   )

6. Please use the accompanying data sheet, which was designed to facilitate the recording of data. It is desirable, in addition, that each participant make a personal, permanent record of all the data and calculations, properly dated and labeled for future reference. A spare data sheet is included.

7. If a Fahrenheit thermometer is used, compute heats of combustion directly in Btu/lb, using a calorimeter water equivalent expressed in Btu/°F (g/lb), and the heat of combustion of benzoic acid as 11,373 Btu/lb.

   If a Centigrade thermometer is used, compute heats of combustion in cal/g using a water equivalent expressed in cal/°C, and the heat of combustion of benzoic acid as 6318 cal/g. Multiply by 1.80 to convert heats of combustion to Btu/lb.

8. The appropriate oxygen bomb instruction manual, e.g. Parr Manual No. 130, Oxygen Bomb Calorimetry and Combustion Methods, should be studied.

Appendix C. Test Method Revisions

Replace existing sections in Tentative Test Method (Appendix A) with the following:

6. (a) The specimen shall be pulverized into a form suitable to pass a No. 60 sieve. Grinding, filing or milling operations, whether manual or mechanical, should be effected on the cross-section which is normal to the grain, fiber or other process-induced orientation. Particular care should be taken to avoid segregation or separation of components. The representative specimen shall not be smaller than 1/2 in. by 3 in. by the thickness as supplied, nor shall the resultant powder weigh less than 10 grams.

Note: While many materials may be suitably reduced using a clean carbide double bastard file and/or mortar and pestle, it may sometimes be useful to (dry-ice) freeze materials containing asphaltic, mastic or plastic components prior to filing, or to use mechanical blendors, ball or hammer mills, grinders, milling or lathe cutters, etc. For laminated materials, it may be preferable to separate into component layers and to grind, file or pulverize each component separately. The powdered components may then be intimately mixed in proportion to their original weight fractions and the mixture tested, or, alternately, each component may be tested separately and the contributions of heat combined in proportion to their original weight fractions.

6. (b) A pellet, weighing approximately 1 g, shall be prepared from an intimate mixture of the powder, and then weighed.

6. (e) If the pellet does not burn, or a residue remains after the firing, another 1-g pellet shall be prepared using approximately 1/2-g portions of the powdered sample and a standard sample of benzoic acid combustion promoter. Weigh each portion accurately to 0.1 mg, mix together thoroughly and pelletize. Record the weight of the pellet to 0.1 mg. Any loss in weight after mixing and pelletizing should be subtracted from the sample and the combustion promoter in proportion to their original weight fractions, and the corrected weights used in the heat of combustion calculations.

Add the following after Note 3, Section 6.(c)

Note 4: For tests on specimens which are predominantly metallic, the use of a silica combustion capsule is recommended. The water equivalent of the calorimeter using the silica capsule should be measured and used.

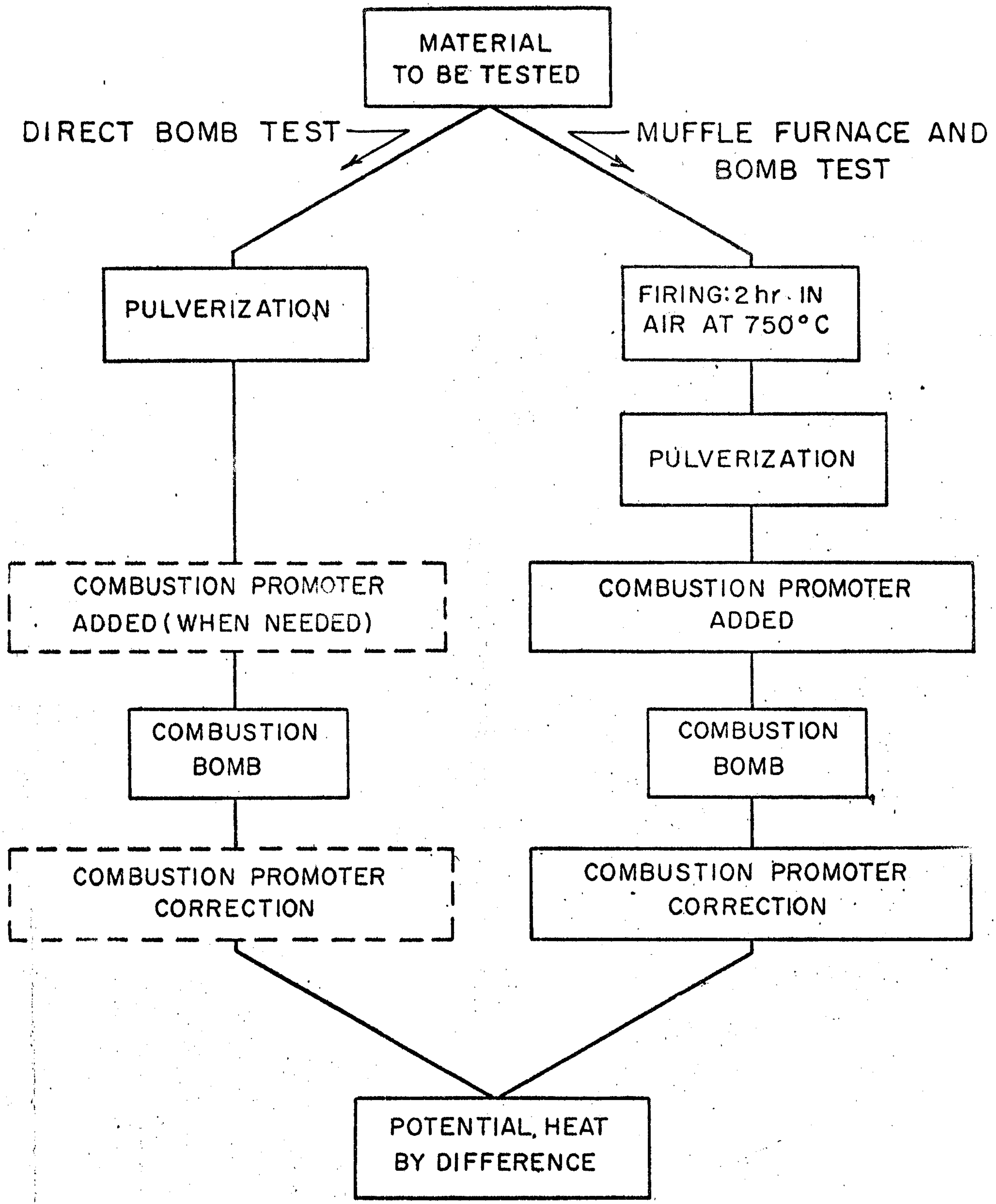

Figure 1A. Schematic Diagram of Test Procedure for Potential Heat Measurements

TABLE 1. LIST OF MATERIALS

Designation	Material	Nominal Thickness in.	Measured Density lb per cu ft
A	Asbestos-cement board	1/4	121
B	Mineralized wood	7/8	24
C	Gypsum board	3/8	49
D	Mineral acoustic tile	3/4	24
E	Protected steel	1/16	281
F	Glass fiber insulation	1	2.2
G	Cinder aggregate concrete	1 5/8	120
H	Aluminum sandwich panel	1	28
M	Asbestos millboard	1/2	55