NATIONAL BUREAU OF STANDARDS REPORT

10 329

PRELIMINARY TESTS OF NON-COMBUSTIBILITY OF MATERIALS USING CRUCIBLE METHOD

U.S. DEPARTMENT OF COMMERCE
NATIONAL BUREAU OF STANDARDS

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The Bureau comprises the Institute for Basic Standards, the Institute for Materials Research, the Institute for Applied Technology, the Center for Radiation Research, the Center for Computer Sciences and Technology, and the Office for Information Programs.

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> Office of Standard Reference Data—Clearinghouse for Federal Scientific and Technical Information [3]—Office of Technical Information and Publications—Library—Office of Public Information—Office of International Relations.

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[1] Headquarters and Laboratories at Gaithersburg, Maryland, unless otherwise noted; mailing address Washington, D.C. 20234.
[2] Located at Boulder, Colorado 80302.
[3] Located at 5285 Port Royal Road, Springfield, Virginia 22151.

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PRELIMINARY TESTS OF NON-COMBUSTIBILITY OF MATERIALS USING CRUCIBLE METHOD

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U.S. DEPARTMENT OF COMMERCE

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Preliminary Tests of Non-Combustibility of Materials Using Crucible Method

by J. B. Fang

1.0 INTRODUCTION

The crucible method for determining non-combustibility was developed by CSTB. This procedure uses a special apparatus which measures the heat content of the volatile components of a material. A duplicate of the apparatus was given to NBS to study the use of the procedure for determining non-combustibility.

2.0 TEST METHOD

The apparatus consists of a crucible, having a lid fitted with a small diameter pipe. As material in the crucible is heated the volatiles evolve from the pipe and are burned by a pilot light. The heat content of the volatiles is then determined by measuring the temperature use of a container of water. The apparatus is described in detail in ISO/TC92 WG2 (France 8).

All the samples used were crushed, mixed, conditioned for about 20 hours in a well ventilated oven maintained at 60 ± 5 °C. They were then placed in a desiccator prior to the test. A weighed sample was mixed homogeneously with furnace-dried and pulverized vermiculite, which served as an inert filler, and then placed in the crucible. The amount of sample used was varied according to its calorific value and was limited to ensure complete combustion without condensation of water on the hollow bottom of the water container.

The crucible was placed in the furnace and heated by a premixed propane/air flame from the gas burner. A diffusion propane flame was employed as a pilot to initiate flaming of combustible volatiles issuing from the mixture in the crucible. The temperature rise of the water container of known water equivalent was measured by means of a chromel/alumel thermocouple whose output emf as a function of time was continuously monitored by a potentiometric recorder. For each sample, four tests were performed: three tests were made with the pilot flame; and one without the pilot flame.

3.0 TEST RESULTS AND OBSERVATIONS

Table 1 presents the results of volatile calorific potential measurements made on five samples of materials which were being used in the current ISO program on studying reaction to fire. In general, the variability in volatile calorific value for repeat tests was noted to be of the order of ± 5 to ± 10%. The amount of fine vermiculite used for each test was about 6 g due to the limitation of available volume in the crucible.

Examination of the table shows that with the exception of red oak, the amount of heat transferred to the container by the volatiles evolved from the sample undergoing pyrolysis when no pilot was used was less than 5% of the total measured when the pilot was used. This low value of heat content may be attributed to a drop in the temperature of the volatiles due to endothermic chemical reactions for phase changes, and a lower convection heat transfer coefficient because of the physical and thermal properties of the volatile products.

An attempt was made to compare the results of with and without using the vermiculite filler to mix with the sample. Tests conducted for red oak, shown in Table 1, indicate that the volatile calorific potentials were approximately 25% less than those for samples mixed with vermiculite. This may be due to incomplete combustion caused by a rapid evolution of a large amount of volatile products and an insufficient air entrainment for stoichiometric combustion.

The volumetric flow rates of propane supplied to the pilot under a pressure of 0.52 mm water and to the Meker burner at 370 mm water were found to be 2.52 cc/min and 996 cc/min (based on 21°C and 1 atm.) respectively by means of a soap bubble tube and a gas flow meter.

A blank test was made by determining the temperature rise of the water container due to the pilot flame, the crucible filled with about 6 g of vermiculite and the burner flame. The increase in its temperature was found to be 2.5°C in 20 minutes with a container of 372.8 g equivalent in water. This corresponds to a rate of heat addition of 47 cal/min.

In order to estimate calorific efficiency of the apparatus, a metaldehyde/vermiculite mixture with calorific values ranging from 0 to 380 cal/g was used as a standard specimen. Different calorific values of the mixture were obtained by varying the mass of Metaldehyde (heat of combustion 6300 cal/g), using a constant weight of vermiculite. The experimental results are shown in Figure 1 where the calorific potential of volatiles evolved from the mixture is plotted against the calorific value of the mixture . The details are summarized in Table 2. The figure indicates that the volatile calorific potential increases with an increase in the mixture calorific value, and at the calorific value greater than about 120 cal/g the data tend to lie along a straight line. The data for the calorific efficiency of the apparatus, which is defined as the ratio of the volatile calorific potential to the mixture calorific value, are also plotted in terms of the mixture calorific value in Figure 1. As the mixture calorific potential increases, the calorific efficiency raises rapidly up to about 60% and then increases at a relatively moderate rate to approximately 71% over the range examined.

As would be expected the radiation emitted from the combustion of the distilled volatiles, for the case of the mixture with lower calorific value, because of its low flaming temperature and small flame, becomes important. However by mixing with small amounts of Metaldehyde, at the pilot with a large portion of the entrained air, most of the heat liberated by combustion are lost to the surroundings. The present results are inconsistent with those presented in the CSTB report (CR No. 990 R 234) where the calorific efficiency was constant throughout the range of the mixture calorific power investigated, and of the order of 73 ± 3%. This discrepancy may be the result of NBS using different amounts of metaldehyde and vermiculite for each test although the calorific value of the two materials was maintained in the same range.

4.0 COMMENTS

4.1 A unified procedure to determine the water equivalent of the container is desirable to be included in the test method.

4.2 In order to let the volatile outlet pipe of the crucible lid pass right through the opening in the water screen without hitting the asbestos around it, a mechanical improvement for the crucible support can be made.

4.3 The appropriate range for operation of the apparatus should be that in which the calorific efficiency is almost constant with respect to the calorific value of the mixture in the crucible. This value should be determined by using a standard specimen.

4.4 The amounts of propane supplied to the pilot and to the burner should be expressed in terms of volumetric flow rates instead of static pressures in the gaseous fuel lines.

4.5 A study is needed of the effects of variables, such as size and amount of the sample, proportion of the inert filler and porosity of the mixture in the crucible on the volatile calorific value to provide a basis for determining their influences on the calorific efficiency of the apparatus.

4.6 Some modifications of the apparatus to increase its calorific efficiency may be possible since the test is primarily concerned with the materials which have low calorific potential or consist of small amounts of the combustibles.

5.0 CONCLUSIONS

On the basis of the preliminary tests conducted to date, it can be concluded that :

1. The calorific efficiency of the apparatus varies with the calorific value of the metaldehyde/vermiculite mixture.

2. Red oak samples which were mixed with an inert filler had higher values of volatile calorific potential than those without it.

Table 1 Data for Heat Released by the Volatiles Evolved from Five ISO Specimens

Material	Test No.	Weight of Sample (g)	Weight of Vermiculate (g)	Weight Loss (%)	Heat Released (cal/g)	Volatile Calorific Potential (cal/g)
Danish Hardboard	1	0.4610	6.45	87.2	1383	1278
	2	0.5039	6.25	86.6	1295	
	3	0.4966	6.23	91.6	1314	
	Ave.				1331	
	*W/OP	0.5008	6.30	79.0	53	
Melamine Chip Board	1	0.5549	6.18	86.1	1259	1163
	2	0.5268	6.23	89.8	1238	
	3	0.5025	6.34	82.0	1113	
	Ave.				1203	
	*W/OP	0.5028	5.73	87.5	40	
Red Oak	1	0.4977	6.25	93.3	1220	1142
	2	0.5030	6.25	87.9	1445	
	3	0.5039	6.25	92.4	1258	
	Ave.				1308	
	*W/OP	0.5157	6.23	96.1	166	
Styrofoam (Fire Retardant Treated)	1	0.1446	6.41	95.2	1340	1305
	2	0.1509	6.19	89.9	1235	
	3	0.1496	6.26	85.2	1420	
	Ave.				1332	
	*W/OP	0.3704	5.60	91.8	27	

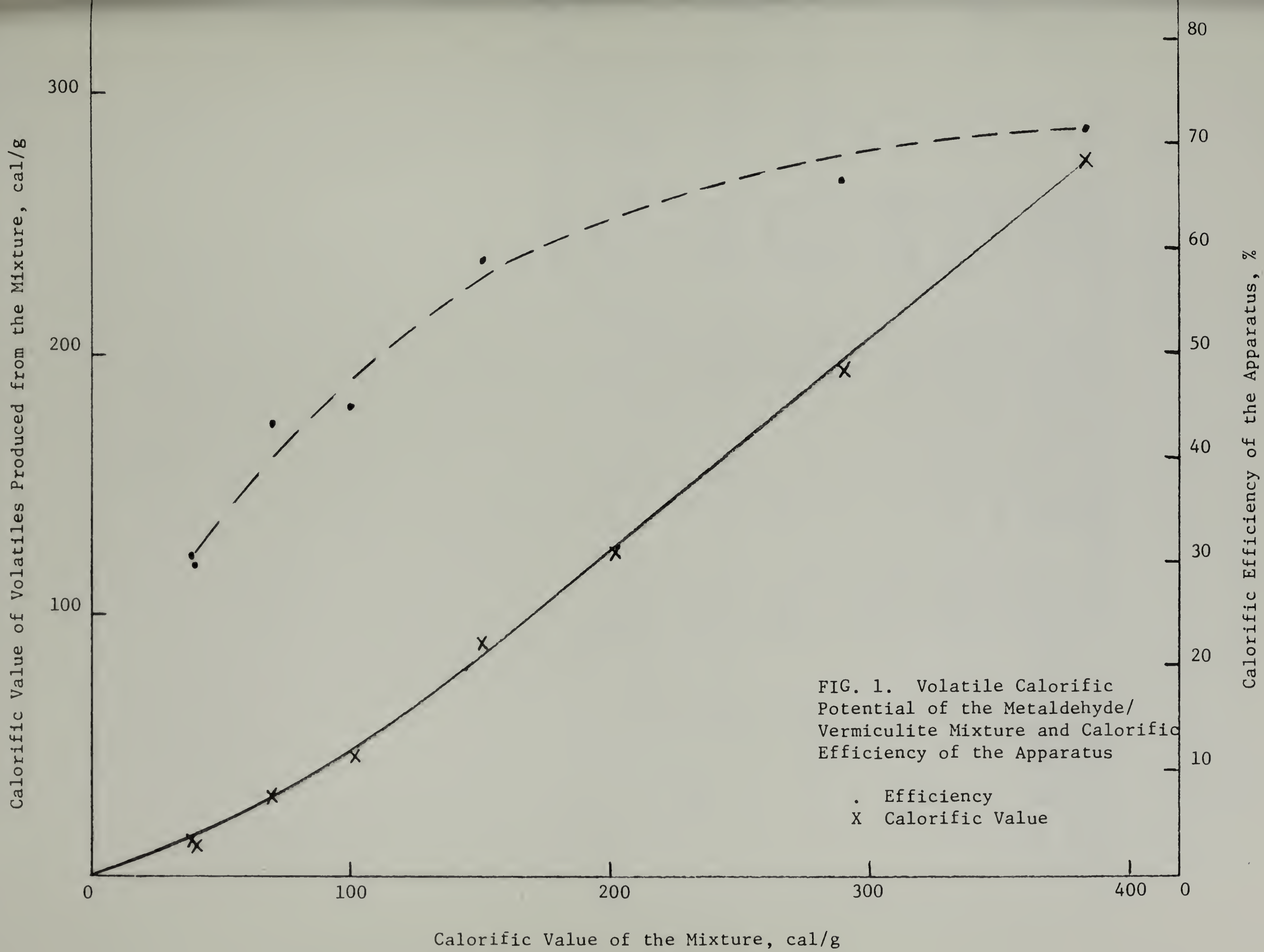

FIG. 1. Volatile Calorific Potential of the Metaldehyde/Vermiculite Mixture and Calorific Efficiency of the Apparatus

Table 1 (Continued)

Material	Test No.	Weight of Sample (g)	Weight of Vermiculate (g)	Weight Loss (%)	Heat Released (cal/g)	Volatile Calorific Potential (cal/g)
Polyurethane	1	0.5035	6.10	77.3	2254	
	2	0.5109	5.60	76.9	2028	
	3	0.4992	5.58	74.9	2075	2102
	Ave.				2119	
	*W/OP	0.5041	5.98	79.6	17	
Red Oak (No filler)	1	6.80	0	79.4	1200	
	2	6.80	0	80.9	1180	
	3	6.80	0	79.4	1040	839
	Ave.				1140	
	*W/OP	6.80	0	80.9	301	

*W/OP = with pilot flame.

Table 2 Data for Calorific Efficiency of the Apparatus

Test No.	Weight of Metaldehyde (g)	Weight of Vermiculite (g)	Calorific Value of the Mixture (cal/g)	Volatile Calorific Potential (cal/g)	Calorific Efficiency (%)
1	0.0399	6.2054	40.3	12.0	29.9
2	0.0391	6.1987	39.5	12.0	30.5
3	0.0699	6.1981	70.3	30.3	43.1
4	0.1005	6.1947	100.6	44.4	44.6
5	0.1521	6.2061	150.7	88.6	58.8
6	0.2052	6.1963	202.0	123.5	61.1
7	0.2993	6.1952	290.3	193.3	66.6
8	0.4016	6.2101	382.7	272.5	71.2

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