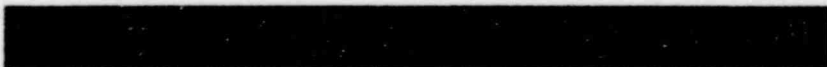


EVALUATION OF MATERIALS USED
IN FIRE-RESISTANT PHENOLIC FOAM

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ABSTRACT

Materials to be used in the fire-resistant phenolic foam are outlined in the U. S. Atomic Energy Commission (Department of Energy) Material and Equipment Specification SP-9. Many of the specified materials are no longer available, or are difficult to obtain, due to specified vendor, grade, or mesh size. Compatibility evaluations of materials that are now being substituted for those outlined in the specification have been made, and the results are presented in this report.

A number of tests were run on the substituted materials, including mesh evaluation, thermal gravimetric analyses, free-foam density, fire tests, and moisture tests. The tests indicated that the substituted materials did not affect the quality of the final foam product. It is recommended that the required properties of the final foam product be detailed in the material specification, along with suggested component materials that are known to yield an acceptable foam. Specification of component materials to be used should be avoided because material availability, material variations between vendors or between lots from the same vendor, and other differences would necessitate obtaining formal deviations from the specification or issuing frequent specification revisions.

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EVALUATION OF MATERIALS USED IN FIRE-RESISTANT PHENOLIC FOAM

INTRODUCTION

Many of the materials outlined in the U. S. Atomic Energy Commission [Department of Energy (DOE)] Material and Equipment Specification SP-9 for fire-resistant phenolic foam are no longer available, and in some cases, the specific grade, mesh size, and/or vendor have presented purchasing problems to contractors. As a result, other materials have been substituted for the originals.

The original specification contained the following materials:

<u>Material</u>	<u>Vendor - Trade Name</u>	<u>Component Weight Per 100 lb of Material, lb</u>
<u>Liquid Components:</u>		
1. Phenolic Resin BRL-2760	UCC Plastics Division	65.8 ± 0.2
2. Surfactant	UCC Plastics Division Silicone Surfactant L-530; Dow Corning Surfactant No. 202, or equal	2.0 ± 0.1
3. Refrigerant-113	DuPont Freon 113, or equal	6.6 ± 0.1
<u>Powder Components:</u>		
4. Boric Anhydride (B ₂ O ₃) -100 +200 Mesh	Var-Lac-OiD Chemical Company	4.1 ± 0.1
Boric Anhydride (B ₂ O ₃) -200 Mesh	Var-Lac-OiD Chemical Company	4.1 ± 0.1
5. Oxalic Acid, H ₂ C ₂ O ₄ , Anhydrous Powder, Reagent Grade	Baker and Adamson No. 1135, or equal	8.2 ± 0.1
<u>Reinforcing Component:</u>		
6. Fiberglas Rovings No. 805 (HSI) 1/4-in. Chopped Lengths	Owens-Corning Fiberglas Corporation	9.6 ± 0.4

Materials 2 and 6 are no longer available from the manufacturers. Union Carbide Corporation (UCC) Y-6663 has been substituted for UCC L-530 and Owens-Corning Fiberglas No. 833 has been substituted for Fiberglas No. 805. Both of these substitutions were made based on recommendations from the manufacturers. Because it is difficult to obtain, practical grade boric

anhydride PO-2685 from Eastman has been substituted for the compound manufactured by Var-Lac-Oid. Since reagent-grade anhydrous oxalic acid is also difficult to obtain, technical grade has been substituted for Material 5. Materials 1 and 3 are still available and remain unchanged. Tests have been run on the substituted boric anhydride and oxalic acid to ensure that the properties of the final phenolic foam are comparable to the foam produced according to the specifications.

EXPERIMENTAL

The materials analyzed are listed in Table 1.

Table 1

MATERIAL ANALYZED

Boric Anhydride (B_2O_3)	Reagent grade, -100 +200 mesh, Var-Lac-Oid Chemical Company Practical grade, -100 mesh, PO-2685, Eastman Chemical Company
Oxalic Acid ($H_2C_2O_4$)	Hydrated, (a) reagent grade, Baker and Adamson Anhydrous, technical grade, Browning Chemical Company

(a) This compound was previously thought to be the anhydrous form, as indicated in the specification, but was later determined to contain two waters of hydration ($H_2C_2O_4 \cdot 2H_2O$).

Thermal analyses, mesh evaluation, and free-foam tests were run on the compounds. Thermal gravimetric and differential thermal analyses were run on the compounds which indicated no significant differences in chemical properties, except for the oxalic acid from Baker and Adamson which was found to be hydrated. The two compounds of boric anhydride were sieved to evaluate the difference in mesh size. The results are given in Table 2.

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Table 2

EVALUATION OF MESH SIZES FOR BORIC ANHYDRIDE COMPOUNDS

Sieve No.	B ₂ O ₃ , Reagent Grade, Var-Lac-OiD -100 +200 Mesh (% Retained)	B ₂ O ₃ , Practical Grade, Eastman PO-2685 -100 Mesh (% Retained)
40	0.41	0.25
70	0.75	62.30
100	14.12	18.48
<100	84.72	18.97

The difference in mesh size did not affect mixing or reaction rate during the foam tests described in the following paragraphs.

Free-foam tests were run on mixtures of the chemicals to evaluate reaction properties. Figure 1 is a diagram of the foaming apparatus. The tests were performed in a round, 95-liter (25-gal) galvanized steel container which was approximately 60 cm (24 in.) high and 42 cm (16-1/2 in) in diameter. Affixed to a vertical aluminum rod in the center of the container were three thermocouples which measured the temperature of the components during the foaming process. A fourth thermocouple was suspended approximately 5 cm (2 in.) above the top of the foam layer and was moved upward, to maintain this 5-cm position constant as the foam expanded. A fifth thermocouple recorded room temperature during the process. The container had 0.5-cm (3/16-in.) weep holes spaced 7.5-10 cm (3-4 in.) apart. The container was heated at a constant rate during the foaming process, with the temperature at the beginning of the process recorded at 40°C.

Two tests were run, using approximately 4.5 kg (10 lb) of material in each. In the first test, the materials used were:

1. Phenolic resin BRL-2760, UCC Plastics Division.
2. Surfactant Y-663, UCC Plastics Division.
3. Freon 113, DuPont.
4. Boric anhydride, reagent grade, -100 +200 mesh, Var-Lac-OiD Chemical Company.

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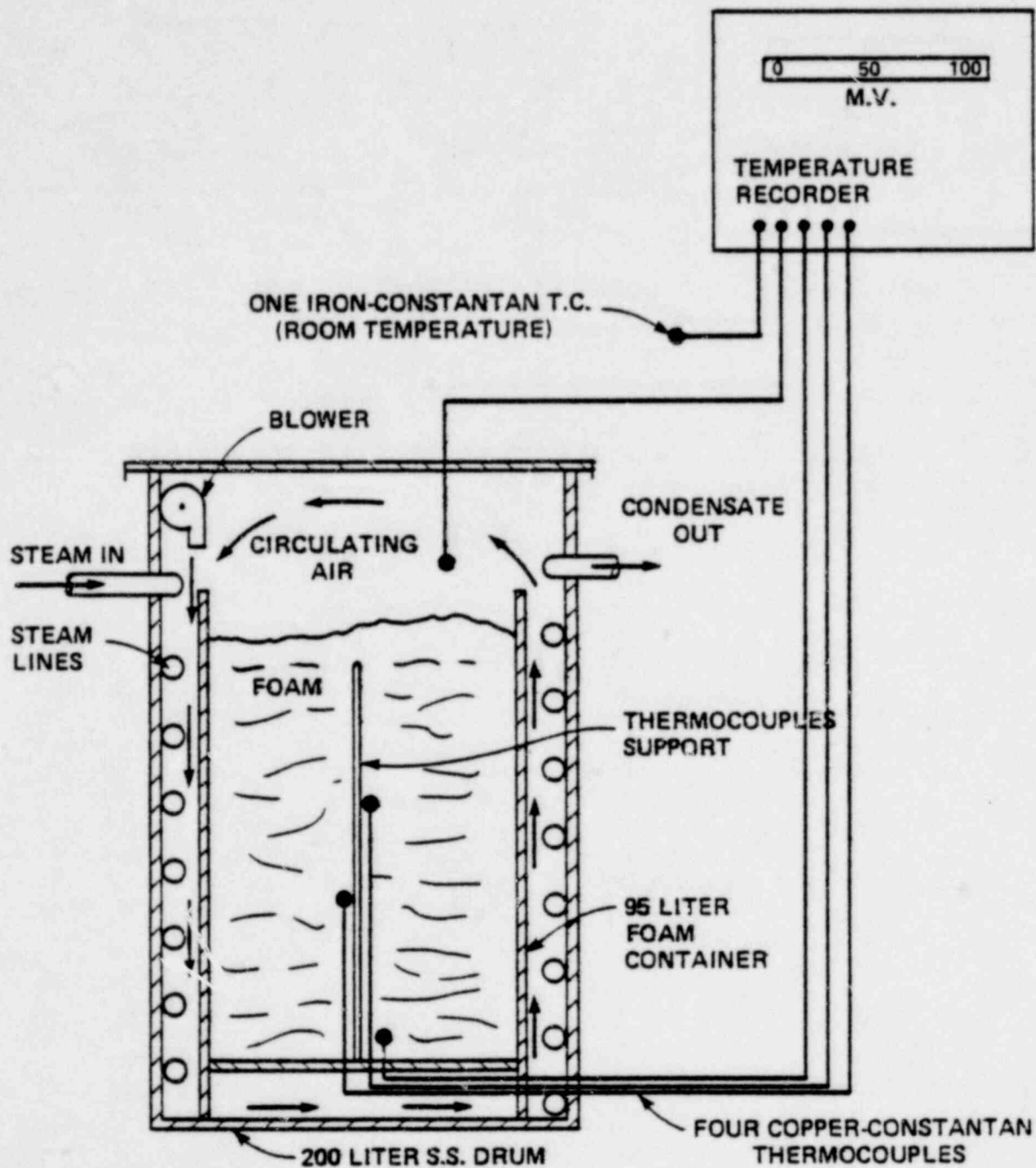


Figure 1

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DIAGRAM OF FOAMING APPARATUS

5. Hydrated oxalic acid ($\text{H}_2\text{C}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$), reagent grade, Baker and Adamson.
6. Fiberglas Rovings No. 883, 0.63 cm (1/4 in.) lengths, Owens-Corning Fiberglas Corporation.

The same materials were used in the second test, except for Items 4 and 5 for which were substituted:

4. Boric anhydride, practical grade, -100 mesh, PO-2685, Eastman Chemical Company.
5. Anhydrous oxalic acid ($\text{H}_2\text{C}_2\text{O}_4$), technical grade, Browning Chemical Company.

It should be noted that no effort was made to compensate for the additional water present in the hydrated oxalic acid. The same weights of hydrated and anhydrous oxalic acid were used in both tests.

For each test, the ingredients were mixed in the same manner. The resin, surfactant, and refrigerant were mixed until homogeneous. Fiberglass was added separately and mixed, followed by addition of the oxalic acid and boric anhydride. After a total mix time of 5 min, the mixture was poured into the container and allowed to free-foam. Heats of reaction during the foaming process for each test are shown in Figures 2 and 3. No significant difference was determined between the two exothermic reactions. Cross sections of the foams after 24 hr are shown in Figure 4. Except for a slight difference in color, the two samples were similar. Both samples reached a height of 50 cm (20 in.) with a foam density of 0.05 g/cm^3 (3.2 lb/ft^3). The irregular shape of the sample from the second test was a result of uneven distribution of the material before foaming.

To compare their properties after burning, the samples were exposed to a 10-min fire test at 760°C . Cross sections of samples after burning are pictured in Figure 5. The samples behaved in the same manner; there was no reduction in size after burning, and the insulating properties adjudged by the depth of charring appeared to be equal.

Portions of each of the two samples were soaked in water for 72 hr to evaluate the absorbent properties of the foam. The pH of each solution was measured after the 72-hr period to give some indication of the corrosive nature of wet, uncoated foam. The results are given below:

<u>Test No.</u>	<u>Water Absorbed</u>	<u>Solution pH</u>
1	3.1 x sample wt	2.5
2	4.6 x sample wt	2.4

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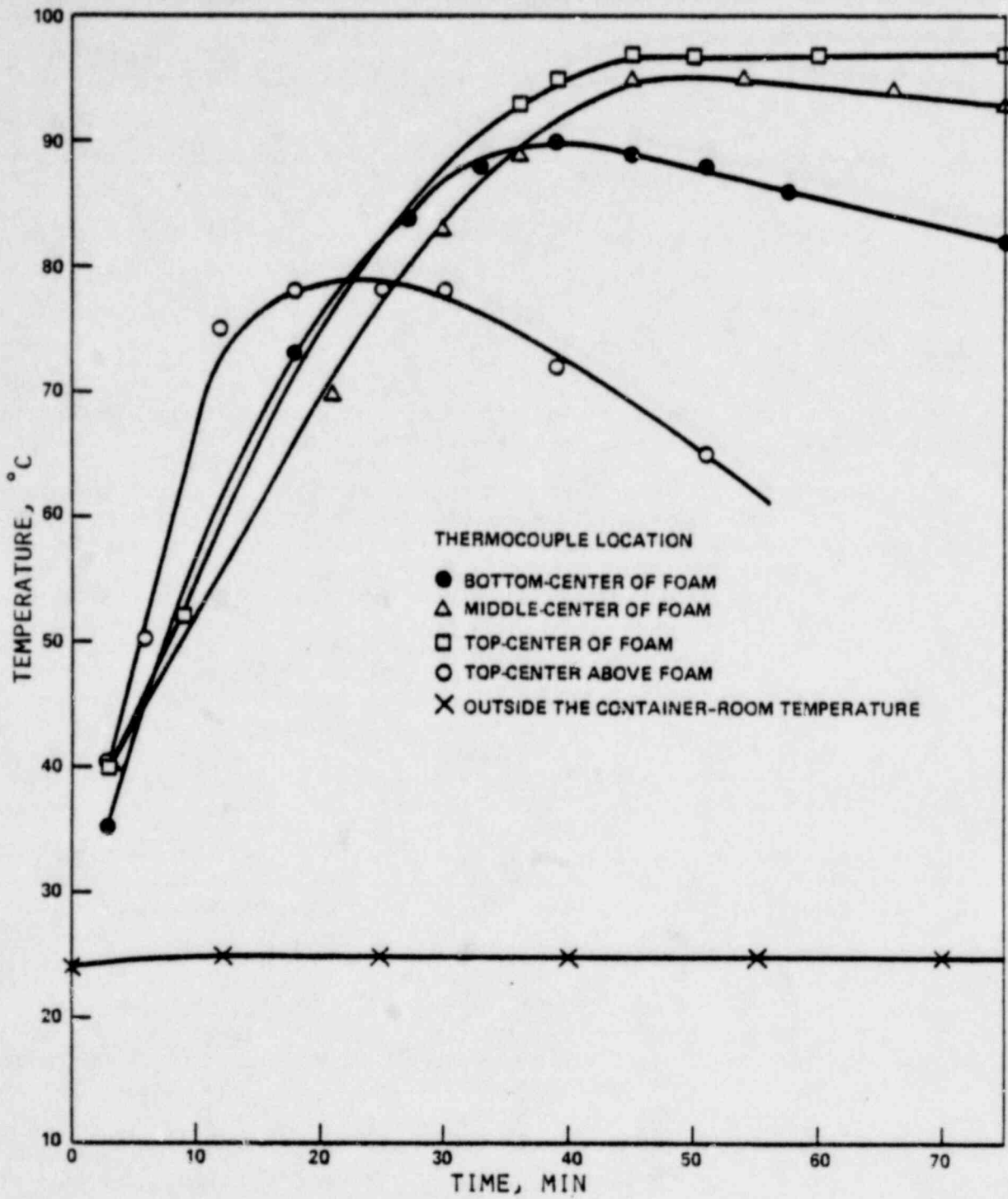


Figure 2

TEST 1: HEAT OF REACTION DURING FOAMING PROCESS

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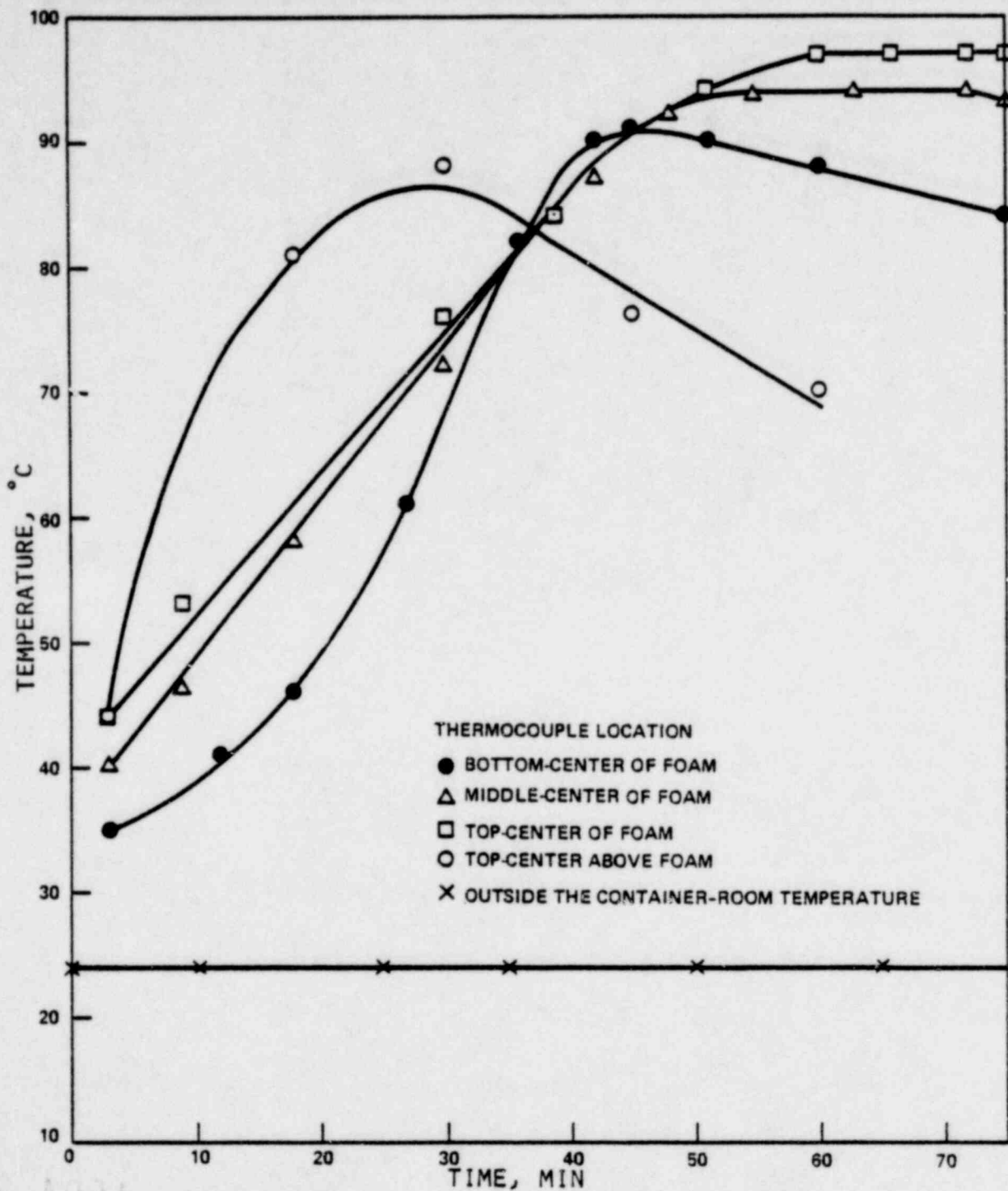


Figure 3

TEST 2: HEAT OF REACTION DURING FOAMING PROCESS

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Test 1



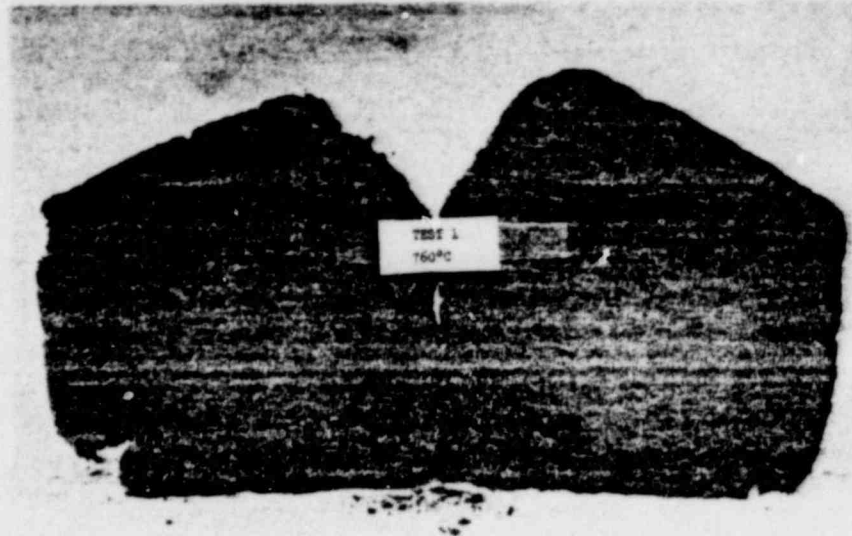
Test 2

Samples are approximately 50 cm (20 in.) high.

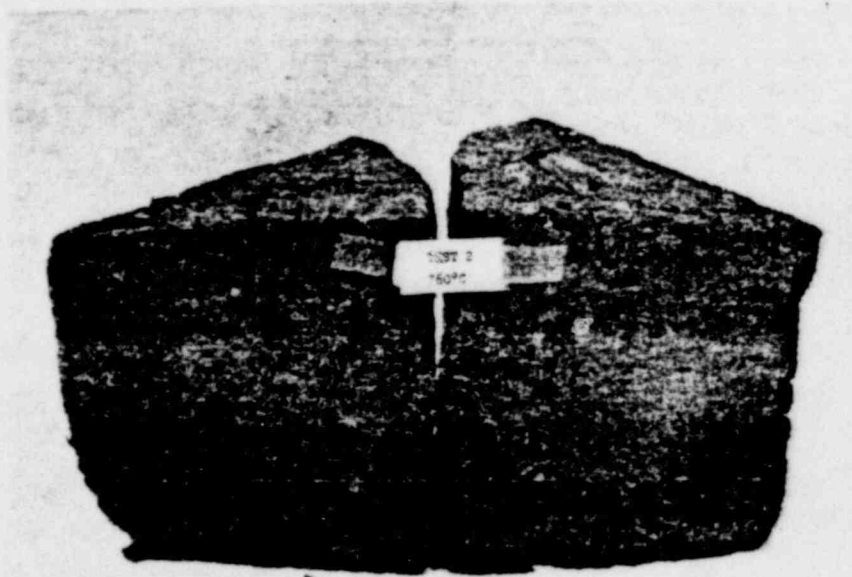
Figure 4

CROSS SECTION OF FOAM

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Test 1



Test 2

Figure 5

SAMPLES OF FOAM AFTER BURNING 10 MIN AT 760°C

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Test 2 was determined to be more absorbent than Test 1; however, the pH values of both solutions were approximately the same. The low pH in both tests indicated that wet, uncoated foam would be corrosive.

CONCLUSIONS

Since substituted and specified materials behaved in a similar manner during the foaming, fire, and water tests, it is evident that the quality of the final product is not entirely dependent on grade, mesh size, or product vendor. An unexpected discovery was that the hydrated form of oxalic acid ($H_2C_2O_4 \cdot 2H_2O$), accidentally substituted for the anhydrous form ($H_2C_2O_4$), did not affect the quality of the foam, indicating that water content in materials can be varied.

It is recommended that the required properties of the final foam product be detailed in the material specification along with suggested component materials that are known to yield an acceptable foam. Specification of component materials to be used should be avoided because material availability, material variations between vendors or between lots from the same vendor, and other differences would necessitate obtaining formal deviations from the specification or issuing frequent specification revisions.

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