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THERMAL EVALUATION OF WATER-EXTENDED POLYESTER

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THERMAL EVALUATION OF WATER-EXTENDED POLYESTER

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ABSTRACT

Cast water-extended polyester, containing ethylene glycol and neutralized boric acid, was not damaged by a temperature of -40°C. Exposure of one surface of a steel-encased casting to 800°C for 30 minutes caused damage to a depth of 0.6 cm.

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INTRODUCTION

Radioactive sources shipped by common carrier require containment that will attenuate radiation and withstand possible weather and accident conditions. A solid polyester-based radiation shield for neutron-emitting sources,[1] such as californium-252, was evaluated at -40°C and 800°C and was found acceptable.

THE SHIELDING MATERIAL

A water-extenxed polyester (WEP) that is resistant to both cold and heat extremes was developed from Ashland Chemical's type 661-P polyester. To produce a solid neutron shield from 661-P polyester, 3 parts of the following solution is added to 2 parts of the polyester:

	Content, wt %
Ethylene glycol	43.4
Water	40
Boric acid	13.3
Sodium hydroxide	3 (to pH 7.5)
"Lupersol DSW"* (hardener)	0.3

When this solution is added to the polyester, a stable emulsion forms in which water and glycol are dispersed as very small droplets throughout a matrix of polyester. The emulsion hardens within a few hours, with heat evolution, to produce a mass resembling plaster of paris.

FREEZE TESTS

Although WEP castings are solid, only the polyester hardens. The aqueous solution is actually still present as liquid droplets. A solid polyester matrix that locks the droplets in place is formed. If a casting is cooled sufficiently, the liquid droplets

* Registered tradename of Lucidal Div., Novadel-Agene Corp.

freeze and expand. To determine whether this expansion might cause stresses that could rupture the polyester matrix, WEP was tested for resistance to cracking at -40°C. The freezing point of the solution and volume changes with temperature were measured.

RESISTANCE TO CRACKING

The resistance of WEP to cracking was demonstrated by repeatedly exposing a cylindrical casting to temperatures <-40°C and then thawing the casting. The WEP casting weighed 11 kg and contained embedded thermocouples. After six cycles of cooling and thawing, the casting was sectioned and examined. No physical damage, such as cracking or crumbling, was detected.

In the freeze resistance tests, the cooling-thawing rates were high and the temperature of the cooler was much lower than -40°C. A freeze-thaw cycle was completed in ∿24 hours. Temperature gradients from the surface of the casting to the center were sometimes as high as 25°C. Such gradients probably caused stresses that would not have occurred at lower cooling-thawing rates. Also, temperatures of some parts of the casting were usually much lower than -40°C on each cooling cycle because the casting was cooled until every part of it was below -40°C. A plot of time versus temperature during a freeze-thaw cycle, measured by the embedded thermocouples, is given in Figure 1.

FREEZING POINT

An exact measurement of the freezing point of the aqueous solution used to make WEP is difficult. If the freezing point is taken as the temperature at which solids first form,[1] the freezing point of the solution is -35°C. Using the same interpretation, the freezing point of solution squeezed from a casting by mechanical pressure is -32°C. However, these first solid particles could be precipitated solute and not really frozen solution.

Although solids were visible in the solution at -35°C, a sample refrigerated to -50°C was still not completely solid.

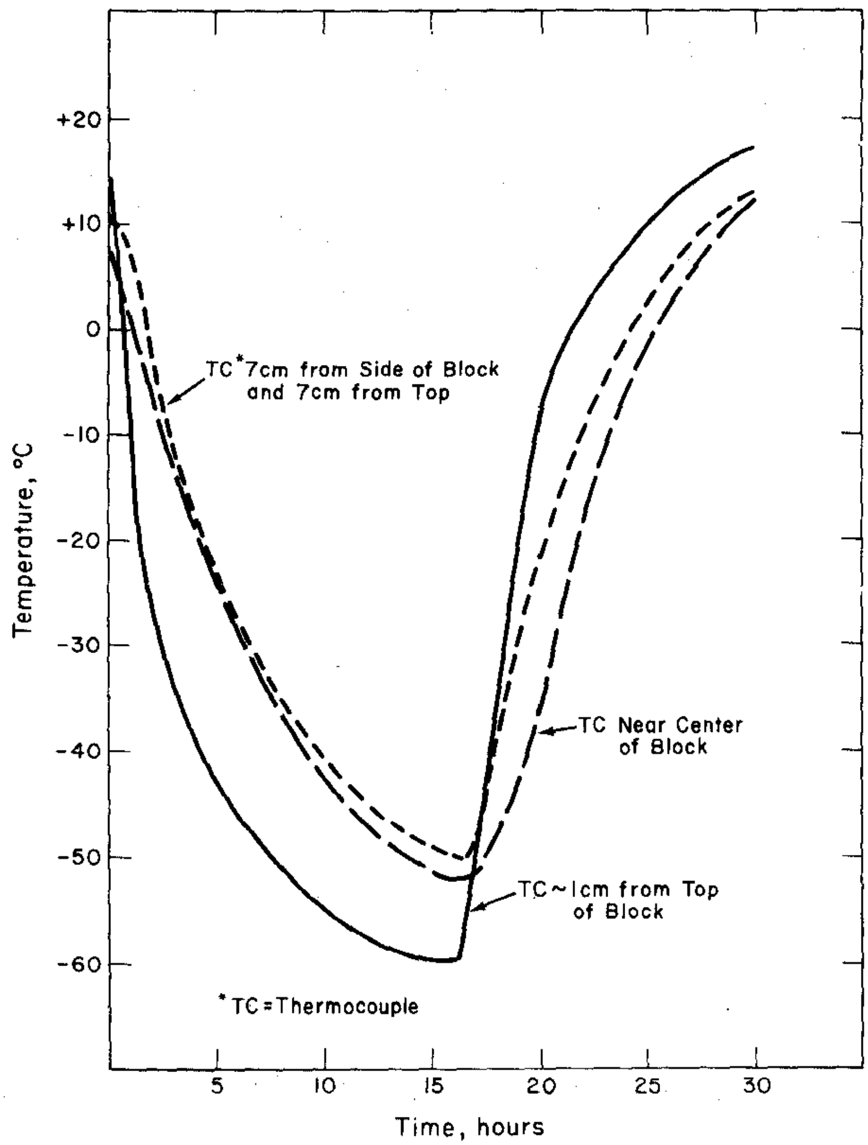

FIG. 1 TEMPERATURE CHANGES DURING A FREEZE-THAW CYCLE

VOLUME CHANGES WITH DECREASE IN TEMPERATURE

In tests with both the solution and the WEP casting, volume did not increase with decreasing temperature until the temperature was below -50°C.

In an aqueous solution test, a modified volumetric flask containing 100 ml of the solution was cooled in a Dewar flask containing powdered carbon dioxide. Toluene on the surface of the solution extended through the temperature gradient zone and filled a graduated tube at room temperature above the Dewar flask. Changes in the level of toluene in the graduated tube indicated volume changes in the solution. The toluene level decreased when cooling first began and then increased (Figure 2). The temperature measured by a thermocouple in the solution at the time of this increase was -54°C.

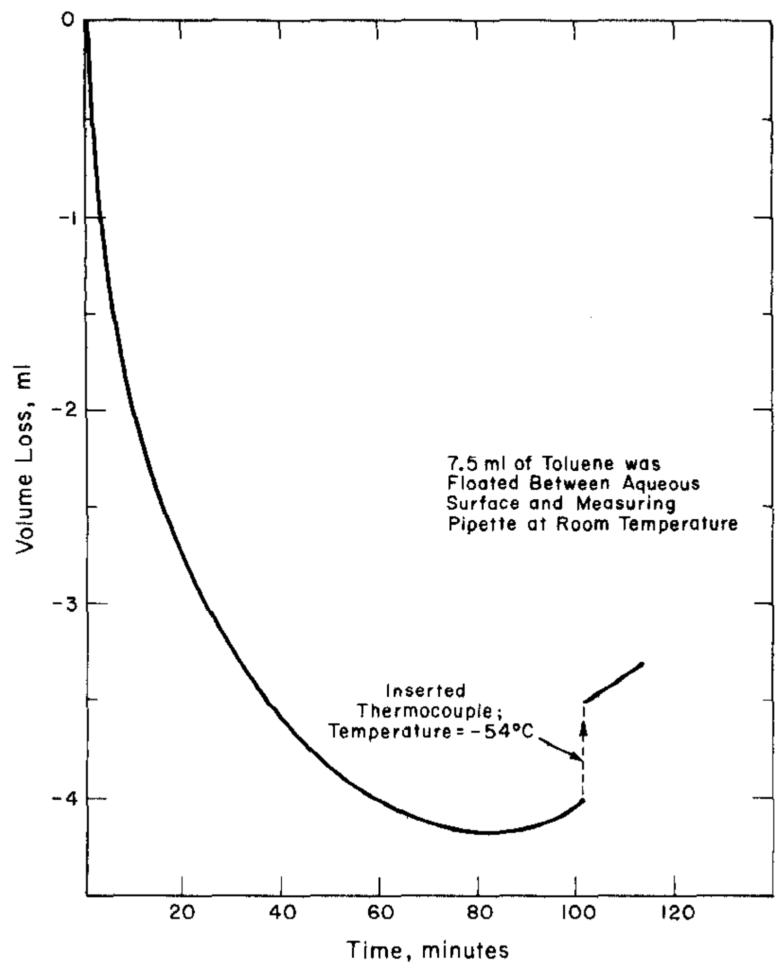

FIG. 2 VOLUME CHANGES DURING COOLING OF THE SOLUTION

In three progressive cooling tests on a 100 ml casting, phase changes and volume increases were observed only as temperatures decreased below -50°C (Figure 3). The apparatus for these tests was the same as that for the solution test. The thermocouple was embedded in the casting at the time the emulsion hardened.

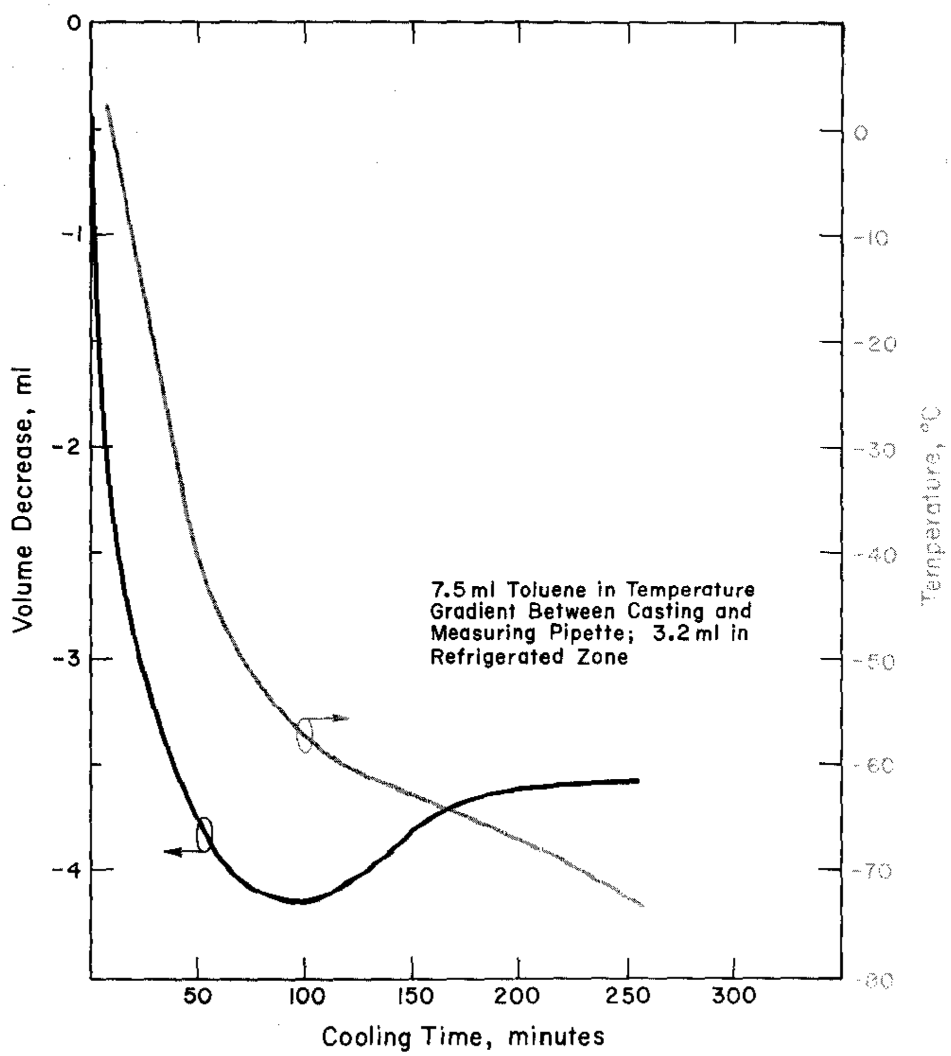

FIG. 3 VOLUME CHANGES DURING COOLING OF THE WEP CASTING

HIGH TEMPERATURE TESTS

A WEP casting encased in steel was heated by exposing one surface to a temperature of 800°C for 30 minutes. The casting was not seriously damaged.

PROCEDURE

In the high temperature test, one surface of an 11-kg WEP casting encased in steel (Figure 4) was exposed to a temperature of 800°C for 30 minutes. The casting and auxiliary equipment were positioned over the furnace, as shown in Figure 5. The test container was designed so that its dimensions simulated a segment of a spherical 5-ft-dia. steel shipping cask. The wall of the shipping cask was assumed to be 3/4 in. thick; and the space inside the sphere, except for a small cavity at the center, was assumed to be filled with WEP. The heated test container surface represented ∿0.69% of the cask surface.

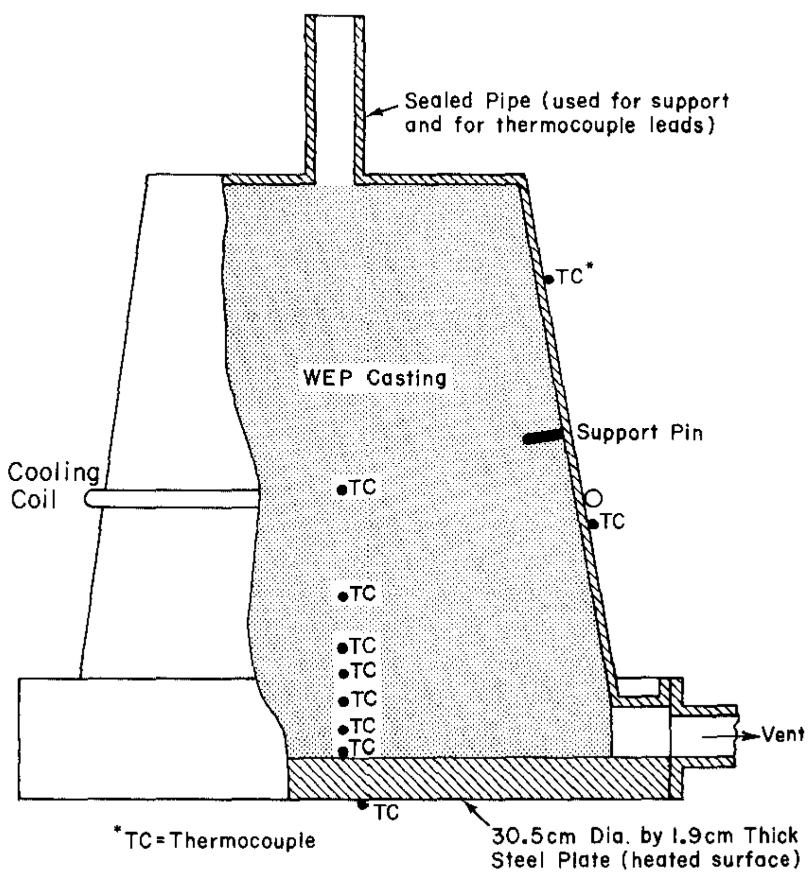

FIG. 4 HIGH TEMPERATURE TEST CONTAINER

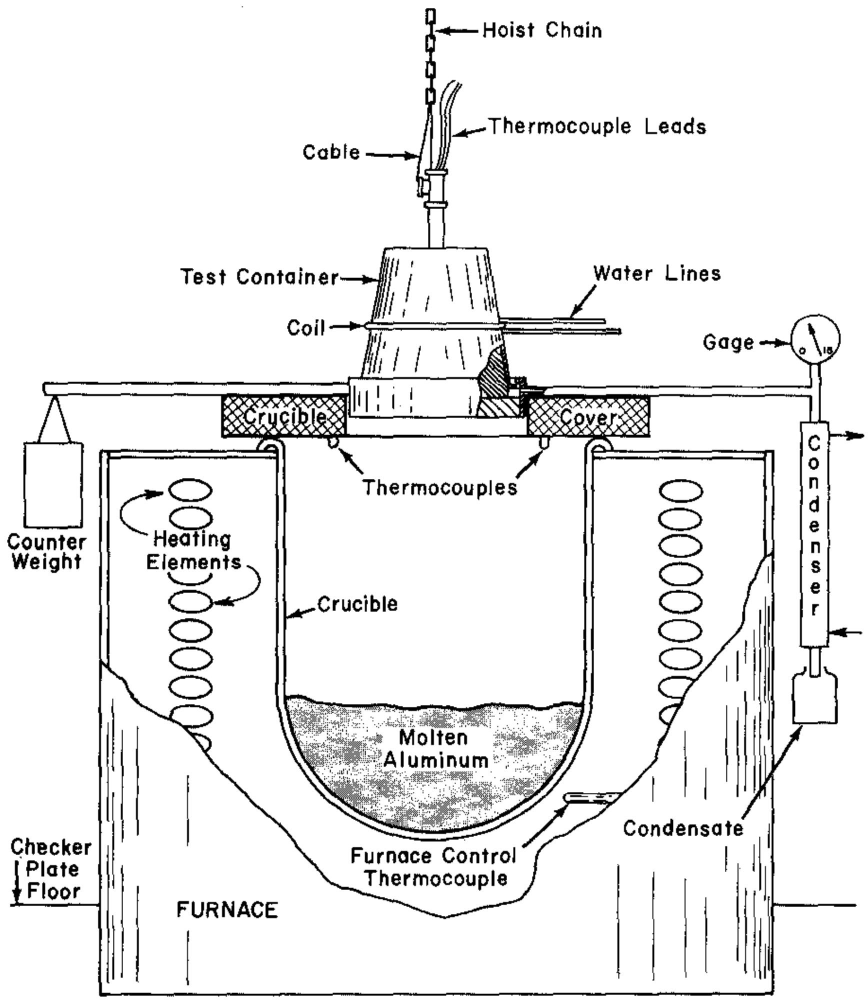

FIG. 5 ARRANGEMENT OF EQUIPMENT DURING HIGH TEMPERATURE TEST

During the 30-minute exposure, the temperatures were measured and recorded in the furnace, on the surfaces of the container, and at locations within the casting (Figures 6 and 7). Control of furnace temperature was lost during the last five minutes of the test, and the temperature went higher than 800°C. The set of low temperatures, inside of the casting and unheated surfaces of the container were extrapolated for the last 5-minute period (Figure 7).

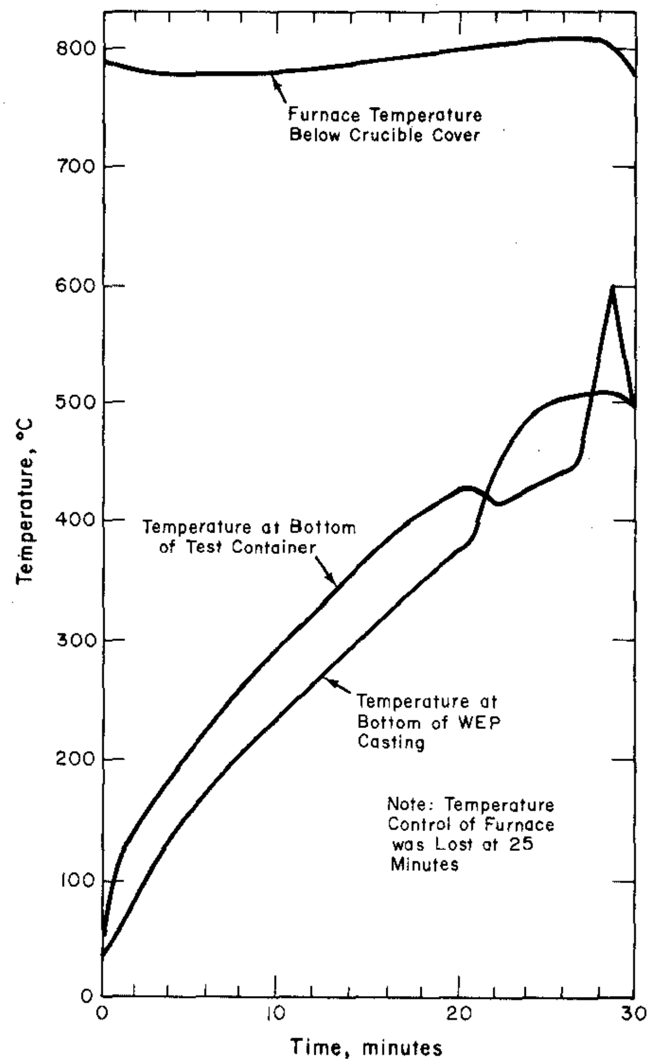

FIG. 6 TEMPERATURES IN THE FURNACE, ON THE HEATED SURFACE OF THE WEP CASTING, AND AT THE BOTTOM OF THE TEST CONTAINER DURING THE HIGH TEMPERATURE TEST

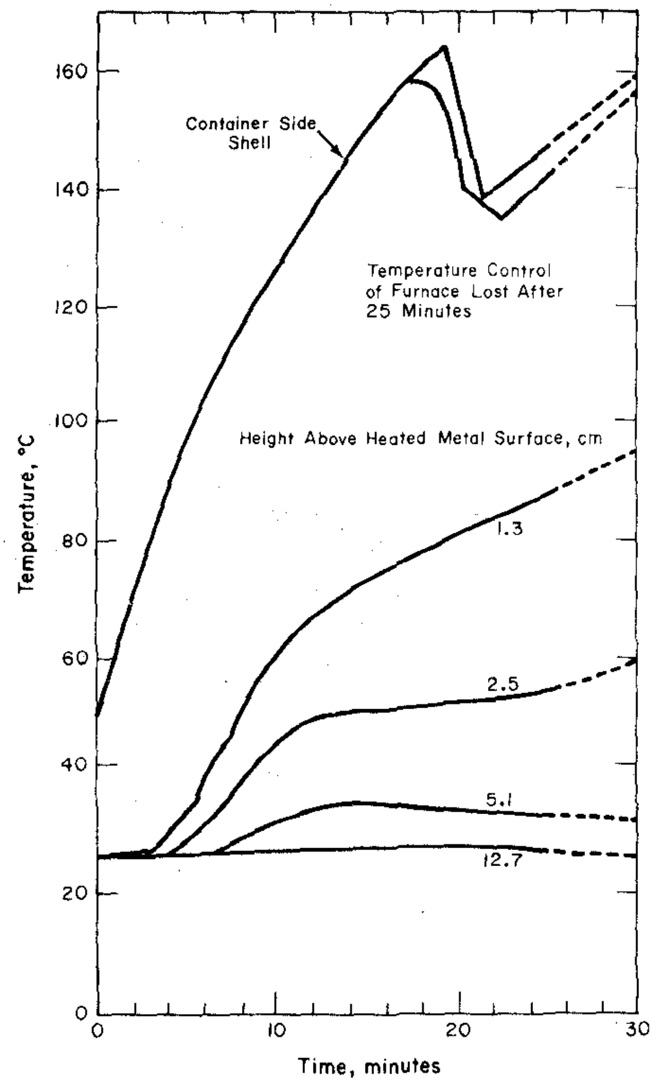

FIG. 7 TEMPERATURES WITHIN THE WEP CASTING AND UNHEATED CONTAINER SURFACES DURING THE HIGH TEMPERATURE TEST

EXAMINATION OF THE WEP CASTING

The test container and the casting were sectioned for examination. Plastic adjacent to the heated container wall had been destroyed to a depth of ~0.6 cm (Figure 8). The exposed plastic surface was charred and friable as shown in Figure 9. Flakes of solid material from the surface were flammable. Subsurface damage was detectable for ~0.8 cm, as shown in Figure 10. The test data indicate that if a cask loaded with a californium-252 source were subjected to the high temperatures of test conditions, the increase in radiation resulting from thermal damage would be less than 1%.

EVOLVED GASES

Gases driven out of the plastic during the thermal exposure period were condensed, and the liquid was collected. The first fraction to be collected, starting 8 minutes after the beginning of the exposure period, was colorless and transparent. About 2 minutes later, a dark-colored, turbid liquid, lower in density and immiscible with the first, began to be collected together with the first fraction. The less dense phase was later found to be flammable. The temperature of the condenser effluent was 28°C. A total of approximately 300 ml of liquid was collected, 100 ml of which was the less dense phase. The rate of evolution (14 ml/min) was almost constant. The total volume collected corresponded with the volume loss of the casting. Almost all gases driven off from the casting were condensible. Only a small amount of whitish fumes were discharged from the condenser.

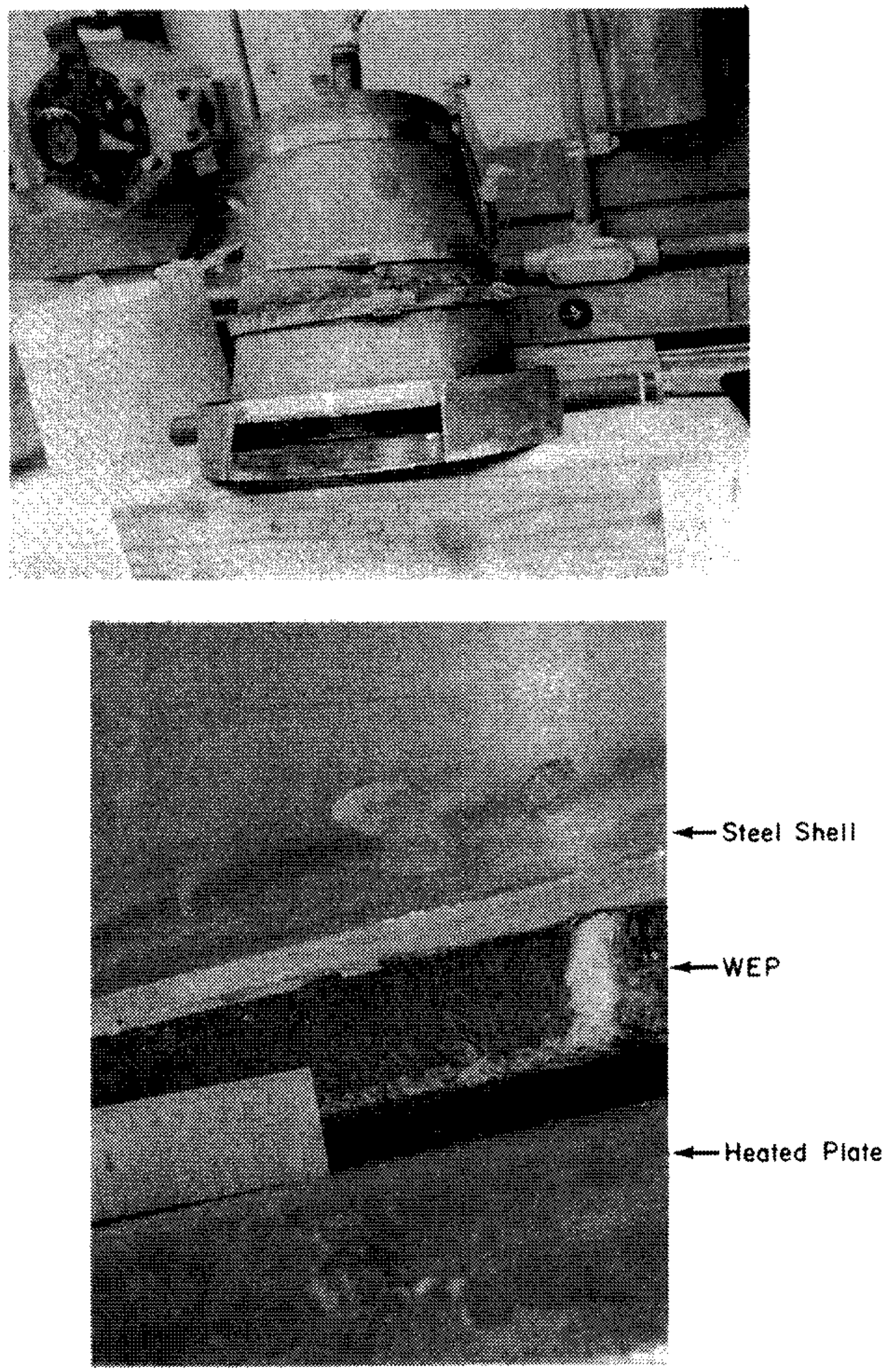

FIG. 8 DESTRUCTION OF WEP ADJACENT TO HEATED SURFACE

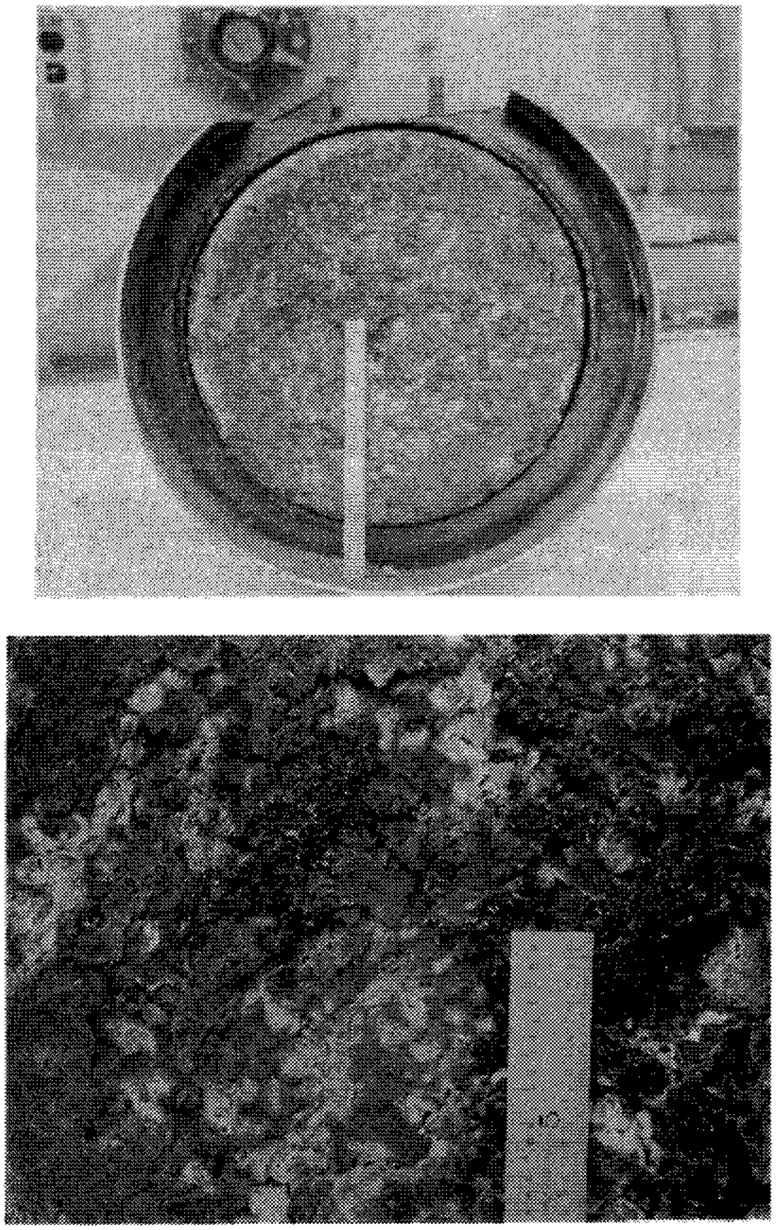

FIG. 9 SURFACE OF WEP CASTING AFTER THERMAL TEST

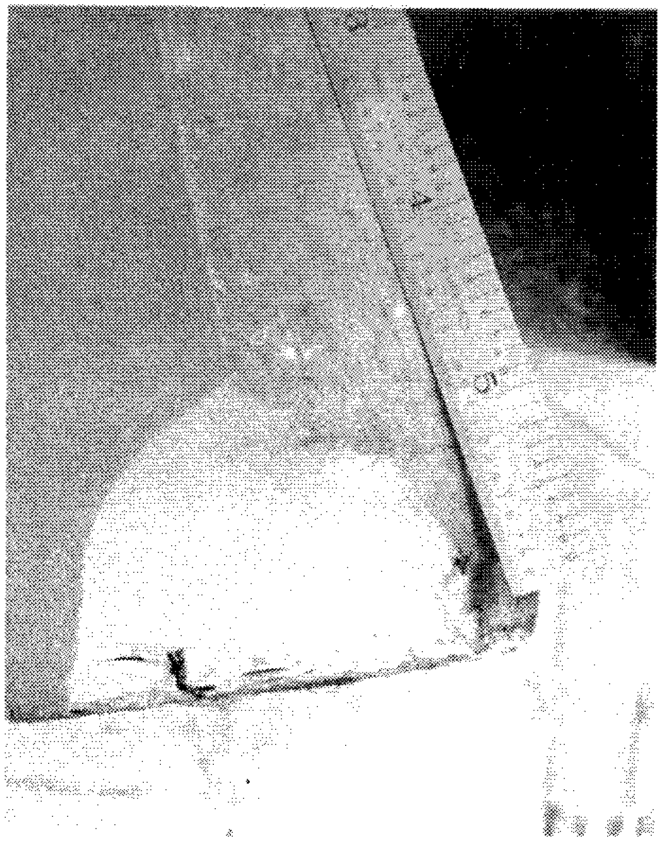

FIG. 10 SUBSURFACE THERMAL DAMAGE TO WEP CASTING

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2. *Annual ASTM Standards*. ASTM procedure D-2386-67, Vol. 17, p. 812, American Society for Testing and Materials, Easton, Md. (1971).