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NATIONAL BUREAU OF STANDARDS REPORT

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AN OVEN TEST FOR SELF-HEATING TENDENCY OF MATERIALS

by

Marjorie W. Sandholzer

U. S. DEPARTMENT OF COMMERCE
NATIONAL BUREAU OF STANDARDS

THE NATIONAL BUREAU OF STANDARDS

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ABSTRACT

A simple oven test for the approximate evaluation of self-heating tendency in materials is described. The method appears suitable for preliminary screening purposes, and its possible usefulness for detecting a hazardous degree of self-heating in materials in general household use is discussed.

1. Introduction

In assessing the possible self-ignition hazard of a material, a simple and convenient means of obtaining a general idea of the self-heating tendency can be very helpful. An approximate determination may frequently be all that the situation requires, or it can serve to indicate an appropriate range for more precise measurement with refined equipment. A simple oven test, designed as a means of roughly surveying a group of foam rubber materials, appears promising for somewhat broader applicability and usefulness. The method is described with the thought that it may prove suitable for the detection of potential self-ignition hazards among materials in general household use.

2. Test Method

The method consists essentially in holding a well-insulated test specimen of the material in an oven at an appropriate temperature for a certain period of time, to determine whether ignition occurs under the selected conditions. With a judicious choice of temperatures, a considerable range of conditions may be studied.

A test specimen of about 8-10 in.^{3} is used. The specimen form is not critical but it should be fairly compact, with dimensions as uniform as may be readily feasible. In any case, the longest dimension should not exceed 3 inches and the shortest should not be less than 1 inch. Liquids may be distributed on cotton gauze, and pulverized or chipped materials may be confined in a gauze or glass cloth bag.

The specimen is wrapped in an initial covering of glass wool free of dressing or binder (preferably hard glass, available from most scientific supply houses) to provide an inert separation of the specimen from the outer insulation. About 1/3 ounce of glass wool is usually sufficient for the purpose. An outer wrapping of about 6 ounces of cotton batting is then applied uniformly to make a test ball 8-9 inches in diameter.

The oven in which the specimen is heated should have a chamber large enough to provide 2 or 3 inches of free space on all sides of the test ball. It should be equipped with forced air circulation, with the heating elements located in the air stream and well removed from the chamber walls, so that the temperature throughout the chamber is fairly uniform. The insulated specimen is placed in the preheated oven and held at a temperature of130±2°C for a period of 7 hours, or until ignition occurs. The production of smoke and char, usually accompanied by glow, is considered to constitute ignition, and is indicated by smoke issuing from the oven. With some materials, such as the rubbers, the odor developed provides a slightly earlier indication of ignition, but smoke also appears shortly and is considered a sufficiently accurate criterion for the purposes of an approximate test. Examination of the specimen upon removal from the oven supplies confirmation, or the final determination, of ignition. The photograph shows a specimen just after ignition.

3. Application of the Method

This oven test was recently used for preliminary work in a study of the effects of aging and cleaning on the self-heating tendency of foam rubbers, where it served to roughly assess differences in self-heating and to indicate materials on which more precise measurements were desirable. The refined measurements were made by an adiabatic furnace method (1) in which the temperature of the heating specimen is closely followed by the furnace temperature, and which provides valid information on the rate of self-heating of the material as a function of temperature. Although the methods differ decidedly, the furnace determinations permitted some appraisal of the usefulness of the oven test. In Table 1 the rates of self-heating of a number of rubbers at 130°C, as determined in the furnace, are listed together with the times required for the same rubbers to ignite in the oven

(1) Self-Ignition Temperatures of Materials from Kinetic-Reaction Data by Gross and Robertson, J. Research of NBS, No. 5, 413-17 (Nov. 1958).

at 130°C. Purified cotton linters and raw linseed oil were also tested to provide some comparison with materials known to differ widely in heating tendency.

A number of the determinations were based on only one test and hence took no account of possible non-uniformities in the material sample. However, in most instances where replicate tests were made, the individual results appeared reasonably consistent. Rubber No. 4 was a major exception, and the individual oven ignition times ranged from 3 hours to about 8 hours. It seems quite possible that in open storage the non-uniformity of a rubber sample may appreciably increase with age, because of the differences in exposure to light and air between the surfaces of the sample and its interior.

In order to determine the actual temperature behavior of the oven specimens, several of the oven tests were made with a thermocouple embedded in the center of the specimen and another placed on the specimen surface inside the glass wool wrapping. Automatic recording equipment provided a continuous record of the thermocouple readings. As would be expected, the temperature at the surface of the specimen was generally somewhat higher than that at the center, until the oven temperature was approached or appreciable self-heating developed. The center temperature appeared the more indicative of characteristic specimen behavior, and Figure 1 shows the time-temperature curves obtained for the specimen center with the four materials on which temperature records were made. The curves for linseed oil and foam rubber No. 4 represent one determination each, and those for cotton linters and foam rubber No. 6 represent the average of duplicate tests. For both of the latter materials, the two tests recorded gave almost coincident curves, with some difference in the early stages due, in part at least, to the variation of a few degrees in starting temperature. The average values for oven ignition time given in Table 1 include other determinations for which time-temperature records were not made.

4. Discussion

The conditions in the oven test and the adiabatic furnace differ considerably. The adiabatic furnace does not supply additional heat to the specimen after self-heating begins, but maintains an ambient temperature closely following the specimen temperature so that a minimum of self-generated heat is lost from the specimen. The rate of rise of the specimen temperature, therefore, depends entirely on the particular self-heating reaction involved. The oven, on the other hand, supplies heat to the specimen steadily up to 130°C, but no higher. The specimen is insulated, however, and the oven heat penetrates the insulation slowly to raise the specimen temperature very gradually. If self-heating begins below 130°C, the specimen will receive heat from both the oven and its own reaction until evolution from the latter source exceeds inflow from the oven, when the self-generated heat will be partially dissipated into the insulation around the specimen. At specimen temperatures above 130°C, there can be no inflow from the oven and there will certainly be some loss of self-generated heat. Hence, the particular self-heating reaction involved will generally be the dominant factor in the rate of temperature rise of a strongly self-heating specimen, but its effects will be more or less modified by such heat exchange with the oven as may occur. The time-temperature curves in Figure 1 illustrate the effects of this heat exchange. In view of the difference in the methods, only a general relationship between the oven ignition times and self-heating rates determined in the adiabatic furnace would be expected.

The cotton linters showed no self-heating below 130°C and, therefore, the time-temperature curve resulted from heating by the oven alone. Since the material specimen is small, the total specimen assembly consists primarily of cotton batting insulation, with a thin inner layer of glass wool, and these products are believed to be sufficiently uniform in density and thermal properties to warrant the assumption that the rate of heat penetration into the specimen assembly is reasonably similar in the different tests. The close similarity of the curves obtained in duplicate tests tends to support this assumption and to indicate that significant differences in insulation packing are not likely, at least among specimens prepared by the same operator. Hence, it would seem permissible to consider the curve for cotton linters fairly representative of the behavior of materials in general when heated by the oven alone, and to assume that pronounced deviations from that curve result from the effects of self-heating on the rate of rise of the specimen temperature.

The curves for the two foam rubbers, No. 4 and No. 6, indicated very similar self-heating characteristics until the specimen temperatures had nearly reached that of the oven, where heat loss from the specimen undoubtedly increased. The heat evolved by the reaction of rubber No. 6 at that temperature was evidently sufficient to continue its acceleration to immediate ignition in spite of the increased loss. The reaction of rubber No. 4, however, required an additional 1-1/2 hours to gradually build up to a temperature where it produced enough heat to compensate for loss to the oven and at the same time cause ignition. The behavior of these two rubbers indicate that, for a limited range of self-heating susceptibility, relatively small variations in the rate of heat evolution may produce widely divergent ignition times. It seems probable that the large variation in individual ignition times for rubber No. 4, noted earlier, reflected this situation and may have resulted from moderate differences in the specimens due to non-uniform aging or manufacture. The range of self-heating susceptibility in which this exaggerated differentiation occurs will depend on the oven temperature selected, and may be shifted to suit varying purposes.

Two tests, one on linseed oil and one on foam rubber No. 7, which were made with the oven temperature held at 115°C, illustrated the effects of a change in oven temperature. The average ignition times for these two materials at 130°C, as shown in Table 1, were quite similar at about 2-1/2 and 3 hours respectively, but at 115°C the linseed oil ignited at slightly over 3 hours while rubber No. 7 required 6-1/2 hours to ignite. Time-temperature records were not obtained for the tests made at 115°C. However, the curve for linseed oil tested at 130°C shown in Figure 1 suggests that at 115°C the linseed oil reaction would have already accelerated sufficiently to ignite the specimen, and that the ignition time was extended primarily by the slightly longer time required to reach that temperature. Presumably, the reaction of rubber No. 7 had not yet accelerated enough at 115°C to produce ignition, and the ignition time was decidedly extended by the increased heat loss above 115°C. The lower oven temperature had shifted the range for sharp differentiation among materials toward those more highly susceptible to self-heating.

5. Suggested Applications

Although there are basic differences between the two methods, the oven test results show a broad general agreement with those of the adiabatic furnace and appear sufficiently definitive to recommend the test for certain purposes. It can be a useful preliminary survey method, as in the present application to foam rubbers, where the effects of cleaning noted by the oven test led to more precise study with the adiabatic furnace. It may also be suitable for the detection of a hazardous degree of self-heating among household materials in ordinary consumer use.

For this purpose a general classification of self-heating tendency is sufficient, but it must be applicable to widely diverse materials and should be reasonably simple to determine. It should be noted that the oven test provides no information on the effect of specimen size on the self-heating hazard, and hence would be of little use in the investigation of warehouse problems involving large quantity storage.

Considering the possible application of the method to household materials, the conditions of the test as it has been described are certainly more severe than would normally occur in the home, and none of the rubbers tested would be likely to present any hazard of self-ignition under the usual conditions of consumer use. Most accidents occur, however, because of inadvertently abnormal conditions. It is believed that the test conditions employed may reasonably simulate such probable abnormalities as the development of a "hot spot" by a malfunctioning heating pad or other electrical appliance, or the careless insulation of a material from proper air circulation, and may provide a useful criterion for household safety. The potential hazard of improperly discarded oily rags is well known, and rubber No. 6 became available for test because of actual ignition some time after a small area of the auto seat had been overheated by a malfunctioning electrical appliance. In view of such instances, the data in Table 1 suggest that it might be appropriate to consider a material which ignites in 3 or 3-1/2 hours under the test conditions described, as potentially hazardous for general consumer use. Additional tests at a lower oven temperature would be informative and perhaps desirable for materials which ignited within that period. An oven temperature of about 130°C would appear suitable for initial testing, however, because it would permit considerable confidence in assuming the general safety of a material which failed to ignite during a 7-hour exposure period.

6. Summary

Several characteristics of the oven test which has been described recommend it as a useful tool in the study of self-heating tendency. It is simple and is applicable to a wide variety of materials. The range of self-heating tendency studied may be adjusted by appropriate selection of the oven temperature.

The test provides a convenient means of making a rough general survey of the self-heating character of a material, from which the desirability of further investigation may be judged. In addition, the approximate information obtained would appear adequate to indicate a potential self-ignition hazard in materials for general household use.

Table 1. Results of Self-Heating Determinations

Material		Oven Test (130°C)		Adiabatic Furnace	
No.	Description	No. of Tests	Approx. time to ignition hr.	No. of Tests	Rate of self-heating at 130°C °C/min
1	Foam rubber slab, natural and synthetic blend, shelf-stored 3 yrs.	1	No ignition	1	.20
2	No. 1 cleaned with tetrachlorethylene	3	4.3	2	.33
3	No. 1 cleaned with detergent in H_2O	1	4.5	1	.30
4	No. 1 stored 1-1/2 yrs. longer	5	5.2	1	.31
5	Foam rubber crumb pressed into sheet, attic-stored 5 yrs.	1	7.0	1	.16
6	Foam rubber slab from auto seat, 11 yrs old	6	2.8	1	.45
7	Foam rubber crumb pressed into sheet, chair pad 10 yrs. old	3	3.0	3	1.10
8	Raw linseed oil (1)	3	2.6	1	5.63
9	Cotton linters (2)	3	No ignition	1	0.00

(1) Distributed as uniformly as feasible on cotton gauze in the proportion of 1 part of oil to 6 parts of cotton by weight.

(2) Purified cotton linters containing less than 0.1% alcohol extractables.

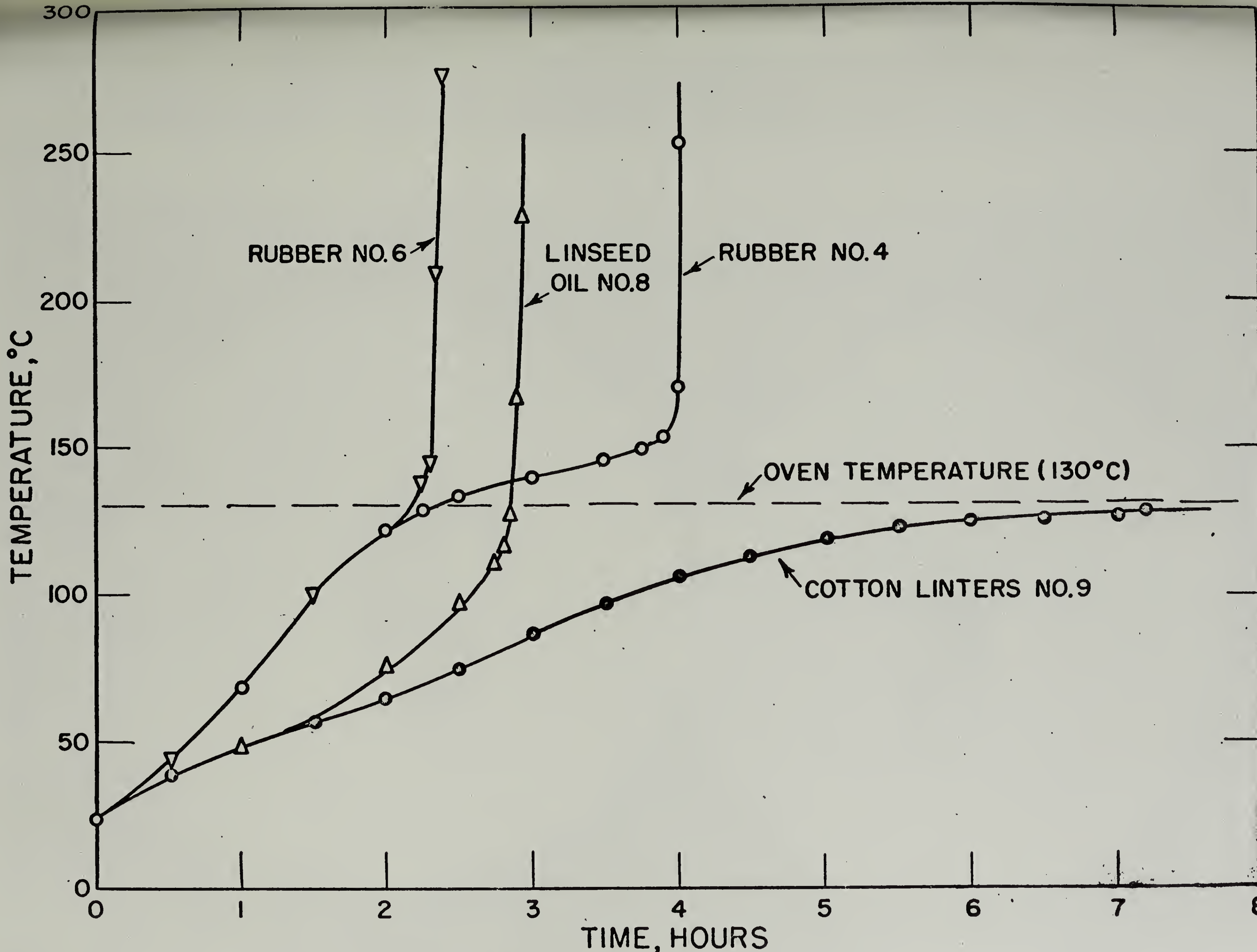

FIG.1— TIME-TEMPERATURE CURVES FOR OVEN TEST

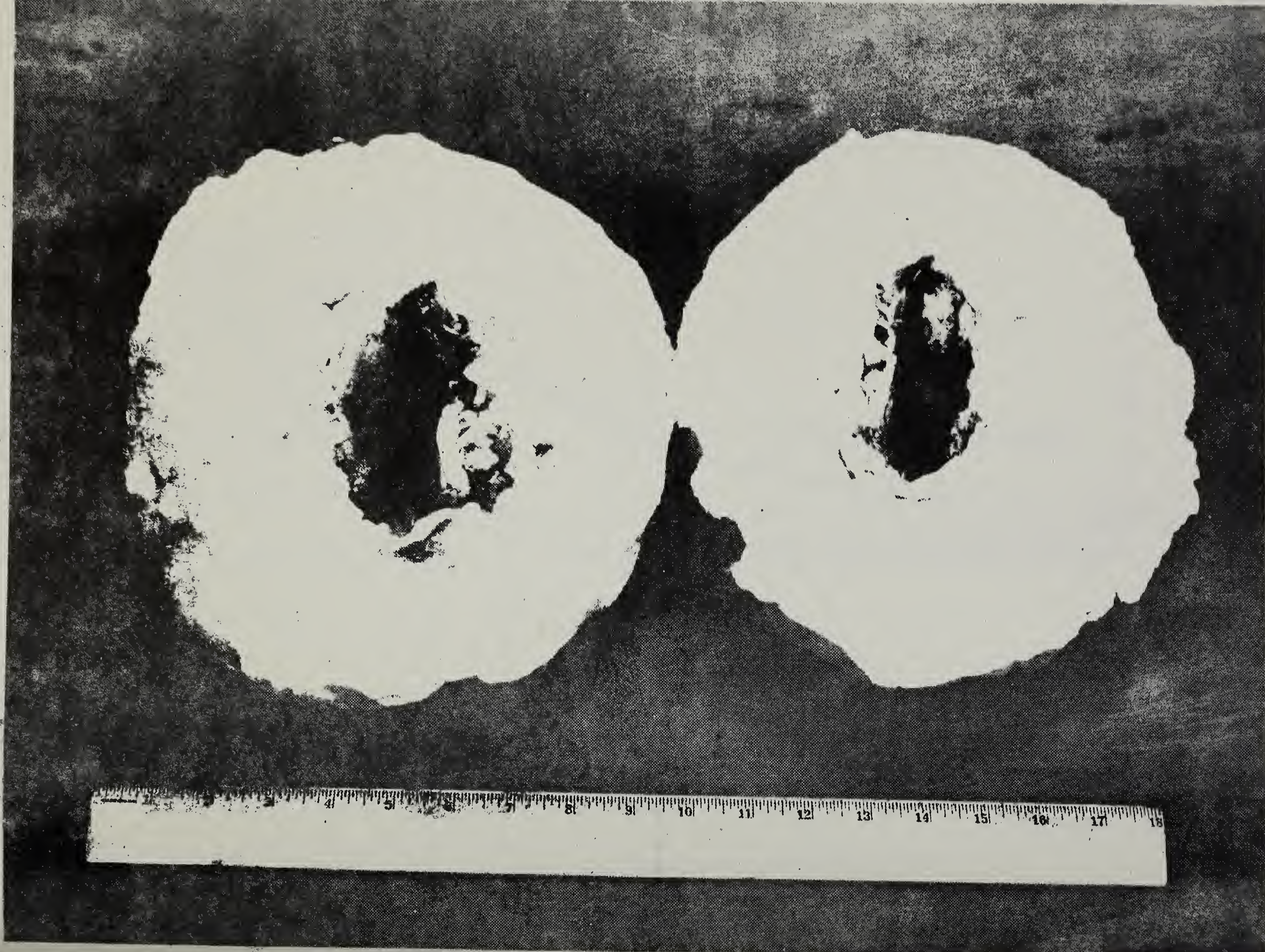

Fig. 2. Test assembly of Rubber No. 6, removed at the onset of ignition and split to show charred rubber specimen and initial charring of adjacent cotton insulation as ignition began to spread.

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Electricity. Resistance and Reactance. Electrochemistry. Electrical Instruments. Magnetic Measurements. Dielectrics. High Voltage.

Metrology. Photometry and Colorimetry. Refractometry. Photographic Research. Length. Engineering Metrology. Mass and Scale. Volumetry and Densimetry.

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Analytical and Inorganic Chemistry. Pure Substances. Spectrochemistry. Solution Chemistry. Standard Reference Materials. Applied Analytical Research. Crystal Chemistry.

Mechanics. Sound. Pressure and Vacuum. Fluid Mechanics. Engineering Mechanics. Rheology. Combustion Controls.

Polymers. Macromolecules: Synthesis and Structure. Polymer Chemistry. Polymer Physics. Polymer Characterization. Polymer Evaluation and Testing. Applied Polymer Standards and Research. Dental Research.

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Inorganic Solids. Engineering Ceramics. Glass. Solid State Chemistry. Crystal Growth. Physical Properties. Crystallography.

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Office of Weights and Measures.

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