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FLAMEPROOFING OF TEXTILES

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PREFACE

To assist in answering requests for information on flameproofing of textiles, Bureau Letter Circular LC467 was issued in 1936, and a revision thereof, Letter Circular LC818, was issued in February 1946. These were prepared by S. B. Detweiler, Jr., S. H. Ingberg, and Marjorie W. Sandholzer, of the Bureau's Fire Resistance Section.

The text of the present printed Circular is based on the above revision and has been issued to meet the demand for a greater number of copies of the mimeographed Letter Circular than can be supplied conveniently.

The information in this Circular is intended to assist in decreasing the life loss and burns from readily combustible garments, tentage, and textile decorations and finish in buildings.

E. U. CONDON, *Director.*

FLAMEPROOFING OF TEXTILES

By Marjorie W. Sandholzer

ABSTRACT

During the past few years there has been an increased interest in treatments that will reduce the flammability of cotton and rayon textiles. This is due in part to large losses of life such as in the Boston night club fire and the Hartford circus disaster from rapid combustion of the untreated material. There has also been a large military demand for flameproofed tentage, camouflage, and clothing fabrics. Although the developments in textile flameproofing extend back over 100 years, the recent demands have been met by some improvements in methods that render the treatments more effective and more resistive to wear, weather, and other conditions of use.

The present Circular includes a review of the principles of flameproofing, a brief history of researches, formulas for various processes, and an outline of testing methods and requirements for the treated material. For the treated or intrinsically slow-burning materials a flame test with the fabric in vertical position is outlined. A method for determining the rate of burning in horizontal position is also defined, which is useful in indicating the relative flammability of untreated textiles.

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I. PRINCIPLES OF FLAMEPROOFING

Textile flameproofing is applied chiefly to cotton, rayon, and other fibers of vegetable origin. Such fibers are composed of cellulose, which when heated, decomposes into readily combustible gaseous and tarry products. Wool and other fibers of animal origin are less flammable, largely because the protein constituents decompose on heating to liberate nonflammable nitrogenous gases. The recently developed, truly synthetic fibers, nylon and Vinyon, are also less flammable than

cotton or rayon and do not present a serious fire hazard, although they melt at relatively low temperatures. Glass fibers are incombustible, and the amount of oil and coatings used in such finished cloth is generally not large enough to introduce a fire hazard.

Complete fireproofing of combustible textiles — making them fully resistant to burning, charring, and decomposition at high surrounding temperatures — has not yet been achieved, and is probably not possible. The most that can be expected of a flameproofed fabric is that it will resist ignition by a match or similar small source of heat, or that, if once ignited, it will not continue burning upon removal of the source of ignition. Such flameproofing may be accomplished by impregnating or coating the cloth with preparations of various chemicals, either singly or mixed.

1. FUNCTION OF FLAMEPROOFING CHEMICALS

There are several types of action by which the compounds are believed to flameproof the fabric. They may be listed as follows:

(a) Modification of the cellulose molecule chemically by the flameproofing agent either before or at the time of combustion, one effect of which is alteration in the decomposition products formed, i. e. less of the very flammable tarry products, and more of the nonvolatile charred residue.

(b) The production of a flux, or foam, from the fireproofing agent by the heat of the fire, thus coating the fibers of the fabric and retarding combustion.

(c) The evolution of noncombustible gases or vapors on decomposition of the flameproofing agent, which dilute the combustible gases from the cellulose decomposition or tend to blanket the fabric and exert a smothering influence on the flame.

(d) The physical weighting of the fabric with noncombustible material, thus rendering it less susceptible to ignition and less able to perpetuate its own kindling temperature.

Many of the flameproofing compounds may combine two or more types of action, and most investigators at present believe that chemical modification of the cellulose is probably responsible for the major result of the more effective treatments. In view of the small amount of a good flameproofing agent required for effective protection, it is scarcely plausible that either (c) or (d) can account entirely for the results obtained. The formation of a glaze (b) could more readily be accepted as a complete explanation of the action of such compounds as borax and boric acid, but there is indication that they also modify the cellulose by their action. Thus, Ramsbottom and Snoad [1]¹ in an extensive study using borax, boric acid, and mixtures of the two, conclude that these chemicals modify the action of heat on the fabric, lowering the temperature at which blackening of the fabric occurs and causing a reduction in the amount and the degree of flammability of the volatile combustibles produced. Their study indicated, in general, that efficient flameproofers increase the destructive action of heat on cellulose, lowering the blackening temperature of the fabric. Many of the ammonium salts of inorganic acids are efficient fire retardants and upon being heated evolve ammonia gas

¹Figures in brackets indicate the literature references at the end of this paper.

which may aid in reduction of flame propagation, but the acid and its reaction on the fabric may play the dominant part. Work recently carried out for the Quartermaster Corps of the United States Army also indicates that even small amounts of fire retardants modify the action of heat on cellulose, increasing the production of char, water, and nonflammable gas, and substantially reducing the amount of volatile tarry matter.

2. GLOWPROOFING

Satisfactory flameproofing of a fabric does not always prevent the occurrence of afterglow, that is, the continued slow burning of the fabric by glow even though it does not flame after ignition. If afterglow is confined to the areas that are already charred, it is not usually serious; but when afterglow spreads extensively into the undamaged fabric, it is a continued hazard and may completely consume the fabric. Good glowproofers appear to function either by coating the fibers, thus cutting off a sufficient air supply, or by chemically altering the course of the oxidation reaction. Among the effective glowproofers are ammonium phosphate and the urea phosphates that, on heating, yield phosphorus pentoxide and appear to control afterglow by chemical reaction. Boric acid and such compounds as zinc borate, which yield boric oxide when heated, also function as effective glowproofers. Recently, some organic compounds have been found effective.

II. HISTORICAL NOTES

The subject of fireproofing has been one of great interest since ancient times. It is said that the Romans attempted to fireproof their houses and war vessels by dipping the wood in a bath of vinegar and clay. An early mention of textile flameproofing occurs in a paper published in 1638 by Nikolas Sabbattini, who urged reforms in the construction and furnishing of Italian theaters from the standpoint of fire prevention, and recommended that the pigment used in painting theaters and scenery should be mixed with clay or gypsum.

In 1735 Jonathan Wild, of England, was granted a patent dealing with a flameproofing mixture composed of alum, ferrous sulfate, and borax. Wild's work was followed by other investigations and the subsequent history of textile flameproofing contains the names of many experimenters, of whom only a few can receive mention here.

Gay-Lussac [2] in 1821 published the results of an investigation suggested by Louis XVIII, in which he determined the fire-retardant properties imparted by numerous salts when deposited on linen and hemp cloth in the amounts of 10 and 20 percent. He found that ammonium phosphate, equal parts of ammonium chloride and ammonium phosphate, and equal parts of ammonium chloride and borax, were effective.

A worthy contribution was made by Versmann and Oppenheim, [3] who in 1859 reported to the British Association for the Advancement of Science the results of a study of the protective action on muslin of 40 chemicals. Their work was conducted on a commercial scale as well as in the laboratory. They found that only five of the

salts and mixtures tried had practical value: ammonium phosphate; sodium-ammonium phosphate; ammonium phosphate and ammonium chloride mixture; ammonium sulfate; and sodium tungstate.

At the instigation of Freeman [4], Whipple and Fay did considerable work on flameproofing theater scenery. Ammonium phosphate was found to be the most effective of many salts tried but was conducive to the growth of mold and mildew. Reporting the results, Freeman states emphatically that, although many substances were found that would make gauze and canvas proof against ignition by small flames, "nothing was found that would prevent the instant burning with a rush of flame when the test was made with a strong blaze on closely hung sheets of canvas." In later work on scenery, Kling and Florentin [5] of the Paris Municipal Laboratory found a solution of borax and boric acid in a 6:5 ratio to be the most suitable of many agents tried.

Ramsbottom and Snoch [1], working at the Royal Aircraft Establishment with cotton fabric, corroborated the effectiveness of a borax-boric acid mixture, but preferred a 7:3 ratio to the proportions of Kling and Florentin.

Perkin [6], seeking a permanent flameproofing process for cotton flannelettes and other highly flammable cotton goods, found the best treatment to be stannic oxide precipitated on the fibers by a method given in section IV of this paper.

The development of a commercial method of producing sulfamic acid [7] led to the use of ammonium sulfamate as a new and effective flameproofing agent [8], which is the basis of a proprietary product on the market.

Other investigators have tried to combine the desirable properties of borates and phosphates without use of ammonium salts, which sometimes produce a progressive and destructive acidity by gradual loss of ammonia. A satisfactory flameproofing agent in the form of a complex borophosphate resin [9] has been developed and is the basis of another proprietary product.

During the war emergency the armed services became deeply interested in the problem of flameproofing textiles, and have carried out considerable work on the fundamental principles of flameproofing as well as its practical applications. The Chemical Warfare Service Development Laboratory at Massachusetts Institute of Technology [10] has developed a formula for water-resistant flameproofing, using antimony oxide in a vinylite binder, which is described in section IV. The National Research Council Laboratories at Columbia University have carried out extensive investigations in the field. The formula for a water-soluble flameproofing agent which they have recently recommended to the Army Quartermaster Corps for general clothing use also appears in Section IV. This formula is considered as satisfactory as any of the water-soluble agents with respect to both flameproofing and glowproofing.

The work of Martin Leatherman [11] is representative of the basic development of the present fire- and weather-resistant finishes for cotton duck. Dr. Leatherman's early work was concerned with improvements in the Perkin process, particularly in preventing after-glow by applying an overcoat of chlorinated resin or rubber to the treated fabric. These materials were known to have slow-burning

properties. He found that the flameproofing oxides (the stannic oxide of the Perkin process and numerous others) can be mixed with the chlorinated resin or oil with retention of the flame and weather-resistant properties, thus eliminating the more complicated two-bath method of impregnation.

Interest in the flameproofing field is still very active and a number of laboratories are working on new or improved formulas. Commercial flameproofing of fabrics at the time of manufacture is feasible only when the treatment has a reasonable degree of permanence to laundering or water leaching. The present fire- and weather-resistant finishes for canvas and duck meet this requirement fairly well and factory treatment is common. However, flameproofing will not be of general interest to commercial finishers of fine fabrics until permanent treatments are developed that are effective in weightings compatible with the uses of such fabrics. Some progress along this line has been made in the field of oil-in-water and water-in-oil emulsions, and in treatments that will decidedly modify the cellulose molecule. Interest is also centered in the possibility of treating fibers before spinning to produce a permanently flameproofed material that will require no further flameproofing treatment after fabrication.

III. TYPES OF TREATMENTS

Three general types of fire-retardant treatments are in use at present. Type 1 combines weatherproofing with fireproofing and is commonly applied to canvas and duck intended for tentage, awnings, and similar outdoor purposes. Types 2 and 3 are either partially or completely removed by laundering or water leaching and are applicable to clothing, decorations, and for general indoor purposes.

1. FIRE- AND WEATHER-RESISTANT FINISHES (TYPE 1, SECTION IV)

The requirements of the armed forces have given considerable impetus to the recent development of improved finishes for heavy fabrics intended for outdoor exposure, combining flame- and weather-resistance. For the most part, the present finishes of this type are designed to meet requirements similar to those of Federal Specification CCC-D-746 for Fire-, Water-, and Weather-Resistant Cotton Duck [12].

As these finishes are designed to withstand outdoor exposure, flame tests after weathering, as well as on the freshly treated fabric, are essential in judging the effectiveness of a treatment. Accelerated weathering tests have been developed that attempt to produce in a short time the effects of longer natural exposures. In the development of specification requirements, the National Bureau of Standards has investigated the correlation between the effects of natural exposure and those of an accelerated weathering machine, in which the material is exposed to radiation from a carbon arc and to periodic applications of water spray. Tests were also made of the effects of water spray upon well-treated awning material. The results indicated that rainfall alone is not of primary importance in weathering as it affects flammability, but that the greatest deterioration is probably due to sunlight and the shock of interspersed sunlight and rainfall. Thus, artificial rainfall applied intermittently over a 40-hr. period, equiva-

lent in amount to about 12 years of natural rainfall in Washington, D. C., produced little loss in flameproof properties. On the other hand, natural weathering in Washington, D. C., for 6 months and 1 year, and artificial weathering (carbon-arc and water-spray exposure) for 15 and 30 days caused a much greater loss of fire-resistant properties.

The accelerated weathering can only approximate a speed-up of the natural weathering process. It is apparent that the great variations in climate mean a corresponding variation in the actual weathering a fabric may be required to withstand.

2. WATER-RESISTANT TREATMENTS SUITABLE FOR CLOTHING AND INDOOR PURPOSES (TYPE 2, SECTION IV)

Several processes have been developed that deposit in the fabric a water-insoluble compound and that will be retained through a number of launderings. Their application is often difficult and complicated and is best carried out in a commercial finishing plant. The antimony oxide-Vinylite process (No. 2 in section IV), one of the more recent of such developments, may be suitable for work clothing and perhaps such materials as upholstery and carpeting, but it so stiffens the fabric that a fairly severe softening process is necessary, and its value for very fine materials is doubtful.

Other recently developed processes are the bases of proprietary products which have been restricted to the use of the armed services. In some cases, flameproofing components have been included in resin treatments deposited on the fabric from a solvent or by use of water-in-oil and oil-in-water emulsions. With availability for general use, some of these new products may prove satisfactorily permanent and suitable for fine fabrics.

3. WATER-SOLUBLE TREATMENTS (TYPE 3, SECTION IV)

A number of the water-soluble flameproofers, such as ammonium salts, borates, phosphates, silicates, etc., have been used for curtains, interior decorations, and clothing for many years. A few, such as ammonium and urea sulfamates, borophosphate resins, and alkyl and aryl phosphates and borates, are of more recent application. In the choice of a flameproofing agent, several factors, the importance of which is dependent upon the intended use of the fabric, must be considered. Some chemicals discolor the cloth or injure it when ironed; others weaken the fabric or make it unduly harsh or heavy. Chemicals that absorb moisture from the air may promote growth of mildew and accumulation of dirt. Those that give off moisture to the air and cause dusting or discoloration, are also undesirable. Crystalline compounds deposited from concentrated solutions may mar the appearance of the fabric. Some chemicals cost too much or are toxic. Nevertheless, in general, it is not difficult to select a formula suitable for the desired purpose.

(a) PERMANENCE

All of the usual water-soluble chemicals are removed by laundering or water leaching, and the treatment must be renewed after each laundering. Most chemicals will be worn away or dusted out of the fabric with continued use or handling. In some recent wear tests

made with workmen's clothing that had been flameproofed with a borax-boric acid treatment, the flameproofing treatment remained entirely effective after two weeks of severe wear. After 1 month's use some parts of the garments showed a considerable loss in flameproofing, but reasonable efficiency was retained in most areas.

Some chemicals, boric acid in particular, are slightly volatile and are slowly dissipated over a long period of time. As a result of indoor sunlight aging tests made at the National Bureau of Standards, it was concluded that effective water-soluble treatments will protect indoor decorations for periods from 6 months to 2 years if the materials are not laundered. The period of retention depends upon both the flameproofing treatment used and the fabric treated. A light-weight white muslin deteriorated more rapidly than a heavier black velveteen.

The development of treatments resistive to dry-cleaning solvents has been fairly successful, as the chemicals in general use are less soluble in such solvents than in water. It should be remembered however, that present commercial dry cleaning procedures employ water-dissolved soaps which may remove a considerable part of flameproofing agents not affected by dry cleaning solvents alone. A number of proprietary products justifiably claimed to be resistant to pure dry-cleaning solvents would not entirely resist the regular dry-cleaning process on this account.

(b) METHOD OF APPLICATION

Soluble flameproofing agents are generally applied from water solution by immersion, brushing, or spraying, followed by drying. The method of application may have considerable effect on the success of the treatment. Hot solutions will generally penetrate more thoroughly, but in some cases their use is undesirable. Borax and boric acid, for instance, can be dissolved most readily in hot water, but it is advisable to cool the solution to approximately room temperature before application, as the hot solution appears to have a greater tendering effect upon the fabric. Dipping the material insures more complete penetration than brushing or spraying. With the latter methods a number of successive applications, with drying between them, may be required before effective protection is achieved. For best results all sizing or dressing should be washed out of the fabric before treatment. Where this is not feasible, the addition of a small amount (about 0.1 to 0.2 percent by weight) of an effective wetting agent to the treating solution will aid wetting and penetration into closely woven or heavily sized materials.

A more uniform distribution of the salts will be maintained if the articles can be dried in horizontal position. Drying in vertical position permits a certain amount of drainage of the solution, particularly from upper areas, depending on the wetness of the wrung articles. If horizontal drying is not feasible, it will be advisable to slightly increase the weighting applied in order to allow for some drainage. Materials treated with water-soluble compounds should not be sprinkled and rolled before ironing since an unsightly spotting from the chemicals may result. Dampening should be done with a steam cloth or similar arrangement at the time of ironing. Care should be exercised to avoid discoloration from a very hot iron.

(c) REQUIRED LOADINGS

The weighting with flameproofing treatments necessary for adequate protection will vary with the fabric treated, lighter weight fabrics requiring somewhat heavier loadings. In general, the water-soluble chemicals should be applied in weightings of from 8 to 15 percent; that is, the weight of fabric after treating and drying should be this much greater than that of the original dry fabric. In the immersion method the material is soaked in the solution until thoroughly saturated (about 15 to 20 min). If the wet fabric is wrung out by hand, it will usually weigh approximately 100 percent more than the original dry fabric, and the chemicals left in the material after drying will approximate their percentage concentration in the treating solution. However, if a mechanical wringer is used, more of the solution is likely to be extracted and a more concentrated solution may be necessary to obtain the desired weighting of salts in the fabric. The suggested concentrations of the water-soluble chemicals (type 3) of section IV, are, in general, suitable for hand wringing, and the quantity of water used should be varied to suit the method of application and type of fabric treated. If the concentration of solution desired exceeds the limit of solubility of the chemical, the wringer pressure may be reduced so that a less concentrated solution will provide the required weighting of salts in the fabric. The solubilities of some of the most commonly used salts and the concentrations which can be obtained are given in table 1.

TABLE 1—Solubilities of flameproofing chemicals
[Parts by weight of salt per 100 parts of water.]

Chemical	Temperature of solution				
	32° F	68° F	86° F	158° F	212° F
Ammonium chloride, NH_4Cl	29.7	37.3	41.6	59.9	76.0
Ammonium sulfate, $(\text{NH}_4)_2\text{SO}_4$	70.7	75.7	78.1	91.7	103.3
Diammonium phosphate, $(\text{NH}_4)_2\text{HPO}_4$..	43.6	59.4	93.8
Ammonium carbonate, $(\text{NH}_4)_2\text{CO}_3$	55.7
Boric acid, H_3BO_3	2.6	4.9	6.6	18.5	39.4
Borax, $\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$	2.1	4.9	7.6	35.3	75.7
Sodium tungstate, $\text{Na}_2\text{WO}_4 \cdot 2\text{H}_2\text{O}$	41.0	82.2	109.5
Di-sodium phosphate, $\text{Na}_2\text{HPO}_4 \cdot 12\text{H}_2\text{O}$	4.2	60.5
Alum, $\text{KAl}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$	5.6	10.8	15.2	65.9
Lead acetate, $\text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2 \cdot 3\text{H}_2\text{O}$	23.0	51.7	81.2
Zinc sulfate, $\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$	74.6	96.6	110.5

(d) APPLICATION IN COMMERCIAL LAUNDRY EQUIPMENTS

Treatment of materials with flameproofing solutions in commercial laundry operations requires certain precautions if the desired results are to be obtained. The temperature of the solution and operating temperatures of the equipment need, in general, to be lower than for the regular laundry process. Particular attention must be given to regulation of the extraction procedure and to the concentration of the flameproofing solution in order to insure the proper final weighting of chemicals in the fabric.

The articles to be flameproofed should first be laundered with the usual number of suds and rinses, and extracted. When highly soluble flameproofing chemicals are used, the articles may be passed directly from the extractor to the flameproofing solution, as the solution con-

centration can easily be increased to compensate both for the water remaining in the fabric before treatment and for the loss of solution from the fabric during the subsequent extraction. If less soluble chemicals, such as borax and boric acid, are used, it may be advisable to dry the articles before the flameproofing treatment, as the concentration of these compounds in solution is limited. Increased solubility of such chemicals may be obtained by heating the solution, but this is limited by the danger of tendering the fabric at high temperatures. Dilution of the flameproofing solution by introducing the materials damp instead of dry may be minimized by increasing the proportion of solution to goods to be treated.

Fabrics can be immersed and soaked for 15 min in a tank of the flameproofing solution or agitated for about 5 min in a wheel or washer containing the solution. If the articles have been dried before the flameproofing treatment, the solution should be used in a concentration about twice the desired final weighting of chemicals to allow for removal of the solution during extraction. A 15 min extraction in the usual equipment leaves moisture in the fabric to the extent of about 50 percent of its dry weight; that is, 150 lb of the damp, extracted materials will weigh approximately 100 lb when dried. Hence, if a final weighting of 10 percent of the flameproofing chemicals is required in the fabric, the treating solution should be used in a concentration of about 20 percent. If the materials are only extracted and not dried before the flameproofing treatment, a somewhat more concentrated solution will be required, depending on the amount of treating solution used in relation to the quantity of materials treated and the water remaining in the fabric before treatment. The articles may be taken from the flameproofing solution, extracted, and finished in the usual manner. The extractor and other equipment in which the flameproofing solution is used should be equipped to permit recovery of the solution, which may be used repeatedly, assuming clean and colorfast fabrics.

Possible damage to treated materials in the tumbler will depend upon the type of fabric, the treating solution used, and the temperature and time of the tumbler operation. More information on these factors is needed. If the goods can be passed directly from the extractor to the mangle for finishing, such possible effects are avoided, but consideration should be given to proper mangle temperatures.

In treating curtains, velour or velveteen draperies, and the like, some laundries prefer a procedure that requires more time but is more suitable for certain fabrics. The flameproofing solution is used in a concentration about equal to the final weighting of chemicals desired, and extraction is reduced to a time that leaves about 100-percent moisture in the fabric. The articles are then hung up to dry. For special work, this method may offer definite advantages and avoids possible damage from heating in the tumbler.

IV. PROCESSES AND FORMULAS

The flameproofing processes for textiles given below are grouped into three main divisions, as outlined in the preceding section. No formulas covered by patents or based on patented proprietary compounds are included except that some of the processes coming under

the general description of type 1 may be patented. Formulas are stated in terms of parts by weight and, also, where water is the solvent, in avoirdupois weight of chemicals and volume of water in United States gallons. Smaller or larger volumes can be prepared by decreasing or increasing all constituents, including the solvent, in the same proportion.

1. PROCESSES COMBINING FIREPROOFING AND WEATHERPROOFING, TYPE 1

No. 1 Finishes Meeting Requirements of Federal Specification CCC-D-746 and Similar Specifications:

The essential components of the treating compounds may be described in general as follows:

(a) An organic binder, such as chlorinated paraffin, chlorinated rubber, or vinyl resins as a film-forming material, which acts as a water repellent and also affords some degree of fireproofing. With the addition of proper plasticizers, such as tricresyl phosphate or alkyd resins, this binder counteracts much of the stiffening effect of the inorganic ingredients on the fabric.

(b) Inorganic flameproofing agents such as oxides, borates, and others.

(c) Pigments, fillers, stabilizers, and other materials, such as mildew-proofing agents, which add specific desirable properties. Pigments not only give color, but, being opaque, also protect the finish from the action of sunlight, which appears to decompose the chlorinated paraffin to a certain extent, forming hydrochloric acid. The finish may be further stabilized by the inclusion of basic compounds such as calcium carbonate, magnesium carbonate, and aluminum stearate, to absorb and counteract the hydrochloric acid which has a tendering effect on the fabric. The protection afforded by these materials is important not only to preserve the finish in its proper condition over a period of use, but also to increase the durability of the treated fabric.

(d) An organic solvent in which the other ingredients are dissolved or suspended during application. The solvent must be volatile and evaporate entirely in the drying. Water-in-oil emulsions have also been successfully used for impregnation.

The fabric is impregnated most effectively in a processing plant, but compounds that will give comparable protection when brushed or sprayed on untreated canvas, or when used to renew the treatment after a period of use and exposure, are available. Manufacturers usually advise renewal after a 1-year exposure and at varying intervals thereafter, depending on conditions of use and exposure. These treatments afford considerable protection to the fabric from the effects of weather, micro-organisms, and handling, and may extend materially the useful life of the fabric.

A considerable degree of permanence has been achieved in these finishes, but the weightings necessary for effective protection are high, from 40 to 60 percent of the weight of the cloth. They are not suitable for use on fine fabrics, as so heavy a loading alters the characteristics of the fabric more than is compatible with use for clothing and decorative purposes. The numerous compounds effective in lighter

weightings are not suitable for outdoor exposure. Many are water-soluble and removed by leaching; others, though insoluble, have not been deposited permanently enough to withstand any extended period of outdoor exposure. If treatments can be developed that effect a basic and permanent change in the cellulose molecule, these limitations may be overcome in whole or part. Attempts have been made to bind water-soluble flameproofers to the fabric with an overcoat of resin, but the results have not been satisfactory.

2. WATER-RESISTANT FINISHES, TYPE 2

The degree of resistance to water leaching will depend on the details of application and loading as well as on the chemicals used. Full resistance in this respect cannot be premised for any of them. Some information on the attainable degree of resistance, if known, is given under the description of the respective treatments.

No. 2 Chemical Warfare Service Process [10], Type 2:

Antimony oxide, Sb_2O_3	120 parts
Vinylite VYHH (vinyl chloride-acetate copolymer)	60 parts
Methyl ethyl ketone	420 parts

(This amount of methyl ethyl ketone is suggested for trial, but it should be varied as necessary to produce a 35-percent pickup of the Sb_2O_3 Vinylite fraction on the cloth.)

Where the white color of antimony oxide is objectionable, part of the oxide may be replaced by suitable coloring pigments matching the color of the untreated fabric. Improvement in glowproofing will be effected if 27 parts of zinc borate are added.

The Vinylite is dissolved in one-half the required methyl ethyl ketone, and the solution is agitated during the addition of the antimony oxide. If equipment is available, the mix is put through a paint mill to produce a more stable dispersion. Before use, sufficient methyl ethyl ketone is added to give a suitable viscosity and pickup.

All sizing and soil must be removed from the cloth by thorough washing and rinsing before treatment. The dry cloth is passed through the impregnating mix and put through squeeze rolls to remove the excess. The setting of the squeeze rolls may be varied to help adjust the pickup to the desired amount. The cloth is carefully dried, then softened by treating for 30 to 60 min on a scouring machine with a 0.2-percent solution of soap or other suitable detergent at 120° to 212° F, depending upon the type of material. The fabric is then rinsed, dried, and may be further softened by treatment on a sanding or sueding machine.

Duck treated by the above process has been commercially laundered 12 times with less than a 10-percent loss in weight of an original pickup of 40.4 percent and with no apparent loss in flameproofing. The treated fabric is also unaffected by Stoddard or similar dry-cleaning solvents, but will not withstand cleaning with chlorinated solvents. This treatment is recommended for cotton, wool, and wool-rayon fabrics.

There has been some indication that antimony oxide has a toxic effect upon certain individuals, and it is possible that fabrics carrying this treatment and worn next to the skin may be found irritant in some cases.

No. 3. *Perkin Process* [6], *Type 2*:

SOLUTION A		
Sodium stannate, $\text{Na}_2\text{SnO}_3 \cdot 3\text{H}_2\text{O}$	41.8 parts.	11 lb. 13 oz.
Water	100 parts.	12 gal.
SOLUTION B		
Ammonium sulfate, $(\text{NH}_4)_2\text{SO}_4$	15.0 parts.	15 lb.
Water	100 parts.	12 gal.

The cloth is thoroughly washed to remove oils, waxes, or other substances which might reduce its absorptive power; it is then acidified with acetic or other weak organic acid, washed, dried, and immersed and agitated in solution A, then squeezed and dried again. It is next run through solution B, squeezed, dried, and washed in cold water to remove the sodium sulfate formed by the interaction of the salts in the two solutions. If desired by the finisher, the sodium sulfate may be left in the cloth.

The goods must be heavily squeezed after being passed through the solutions, as the pressure seems to increase the affinity of the cloth for the stannic oxide formed, with the consequent deposition of more of the latter. The process may have to be repeated or more concentrated solutions used, preferably the former, in order to obtain the required loading of tin oxide which should approximate 20 percent.

The process has been used for flannelettes and other light cotton goods. Perkin states that the treatment does not injure delicate colors and is not harmful to the skin; that the stannic oxide gives the cloth a softer and fuller feel than that of the original flannelette; and that the material is considerably strengthened by the process.

The treatment, particularly in the lighter loadings, may not prevent propagation of glow. Although it was originally indicated that the effectiveness of the Perkin process lasted through 20 washings without loss in fire resistance, recent work has shown a large loss after only a few regular washings in present-day laundry equipment, each involving several suds and rinses of relatively long duration.

The difficulty experienced by many investigators in obtaining an effective application of this process may be due in part to the absorption of alkali by the stannic oxide. Leatherman [11] recommends the use of ferric sulfate or chloride instead of ammonium sulfate as the precipitating agent in order to maintain an acid solution.

3. WATER-SOLUBLE TREATMENTS, TYPE 3

No. 4. *Kling and Florentin* [5], *Type 3*:

Borax, $\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$	6.0 parts.	6 lb.
Boric acid, H_3BO_3	5.0 parts.	5 lb.
Water	100 parts.	12 gal.

The fabric is steeped in a cool solution until thoroughly impregnated, then dried. Heavy applications by spray or brush are usually reasonably effective. Such applications may have to be repeated two or three times with drying between applications to obtain the desired degree of flameproofing. The treatment has been used for many kinds of fabrics, including theater scenery. Grove-Palmer [13] recommends it for rayon. As in the case of most of the other formulas listed, care must be taken in ironing the fabric to avoid discoloration by heat.

The treatment is effective in weightings from 8 to 12 percent, depending upon the type of fabric. Handwringing the above solution from a fabric leaves a weighting of 10 to 12 percent after drying.

No. 5. *Ramsbottom and Snoad [1], Type 3:*

Borax, $\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$	7.0 parts.	7 lb.
Boric acid, H_3BO_3	3.0 parts.	3 lb.
Water	100 parts.	12 gal.

The amount of water may be varied, and should depend upon the absorptive capacity of the fabric to be treated. Loadings from 8 to 10 percent of the weight of the dry cloth usually will be found effective. Handwringing the above solution from a fabric will give approximately these weightings. Fabrics so treated retain their flexibility and softness. They do not become dusty, feel damp, or lose their strength under ordinary conditions of use. The chemicals are nonpoisonous and do not promote the growth of destructive micro-organisms. This treatment, followed by an acetate dope, has been recommended for flameproofing airplane cloth [14]. Brosnan [15] recommends the solution in 7-percent strength for rayon and sheer fabrics.

No. 6. *Army Quartermaster Corps, Type 3:*

Borax, $\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$	7 parts.	7 lb.
Boric acid, H_3BO_3	3 parts.	3 lb.
Diammonium phosphate, $(\text{NH}_4)_2\text{HPO}_4$	5 parts	5 lb.
Water	110 parts	13-1/5 gal.

This formula gives very satisfactory results both in flameproofing and glowproofing. It will be found effective in weightings of 7 to 15 percent, depending upon the fabric treated. Handwringing the above solution from a fabric leaves weightings of about 10 to 12 percent.

No. 7. *Matthews [16], Type 3:*

Ammonium phosphate, dibasic $(\text{NH}_4)_2\text{HPO}_4$..	7.5 parts.	7½ lb.
Ammonium chloride, NH_4Cl	5.0 parts.	5 lb.
Ammonium sulfate, $(\text{NH}_4)_2\text{SO}_4$	5.0 parts.	5 lb.
Water	100 parts.	12 gal.

Either the solution can be applied directly to the cloth, or it can be used in making a starch sizing. The formula has been used for flameproofing curtains; Matthews recommends it for cotton fabrics in general. The ammonium chloride and, to less extent, the ammonium phosphate are hygroscopic; therefore this formula may not be advisable for flameproofing materials exposed to dampness. The treatment is effective in weightings of 10 to 18 percent, depending upon the type of fabric treated. Handwringing the above solution from a fabric leaves a weighting of about 16 to 18 percent.

No. 8. *Martin [16], Type 3:*

Ammonium sulfate, $(\text{NH}_4)_2\text{SO}_4$	8.0 parts.	8 lb.
Ammonium carbonate, $(\text{NH}_4)_2\text{CO}_3 \cdot \text{H}_2\text{O}$	2.5 parts.	2½ lb.
Borax, $\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$	8.0 parts.	8 lb.
Boric acid, H_3BO_3	3.0 parts.	3 lb.
Starch	2.0 parts.	2 lb.
Dextrin	0.4 parts.	6½ oz.
Water	100 parts.	12 gal.

The amount of water may be varied. The mixture should be applied at 86° to 100° F. This solution is useful for many fabrics, particularly for laces, curtains, and aprons and is effective in loadings of 14 to 28 percent, depending upon the fabric. Handwringing the above solution from a fabric leaves a weighting of about 28 percent.

No. 9. *Chesneau* [17], *Type 3*:

Sodium tungstate, $\text{Na}_2\text{WO}_4 \cdot 2\text{H}_2\text{O}$	20.6 parts.	20 lb. 9½ oz.
Sodium phosphate, dibasic, $\text{Na}_2\text{HPO}_4 \cdot 12\text{H}_2\text{O}$	0.6 parts	9½ oz.
Water	100 parts	12 gal.

Sodium tungstate has been used for flameproofing theater scenery in Paris, London, and elsewhere. The addition of the sodium phosphate is recommended by Chesneau to prevent crystallization, which results from the formation of an acid sodium tungstate. Handwriting the above solution from a fabric leaves a weighting of about 23 percent. The treatment is effective in preventing flaming, but afterglow continues for a considerable time and may seriously extend the char.

No. 10. *National Bureau of Standards*, *Type 3*:

Sodium tungstate, $\text{Na}_2\text{WO}_4 \cdot 2\text{H}_2\text{O}$	24.0 parts.	24 lb.
Ammonium phosphate, dibasic $(\text{NH}_4)_2\text{HPO}_4$..	6.0 parts.	6 lb.
Water	100 parts.	12 gal.

Handwriting the above solution from a fabric leaves a weighting of about 30 or 31 percent. The solution is very effective in preventing flaming, but there is some afterglow.

No. 11. *Matthews* [16], *Type 3*:

Starch (or flour, sago, dextrine, etc.).....	22.0 parts.	22 lb.
Sodium tungstate, $\text{Na}_2\text{WO}_4 \cdot 2\text{H}_2\text{O}$	11.0 parts.	11 lb.
Borax, $\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$	7.0 parts.	7 lb.
Water	as desired.	

This formula is used for sizing. Although the tungstate and borax reduce the flammability of the fabric to some extent, their proportion to the amount of starch is relatively so low that adequate flame protection is not provided.

V. TESTS AND TESTING METHODS

Through the years various methods of testing and comparing the effectiveness of flameproofing treatments have been devised by different experimenters. In the earlier work on test methods, particular recognition should be accorded the work of Whipple and Fay [4], J. R. Freeman [4], and Ramsbottom and Snoad [1], who thoroughly investigated the subject of tests and testing procedure. In 1938 the National Bureau of Standards [18] made a comparative study of a number of testing procedures, including the National Bureau of Standards vertical flame test, the Underwriters' Laboratories flammability test, the Freeman stove pipe test, the Whipple-Fay burning test, the British Standards Institution test, and a fold test. As space does not permit a full discussion of the various testing methods developed, descriptions in this circular will be limited to those in present use at the National Bureau of Standards.

1. VERTICAL FLAME TEST

This test is similar to one devised earlier by Ramsbottom and Snoad [1]. It is fairly severe and is the performance test required by Federal Specification CCC-D-746 for Duck, Cotton; Fire, Water and Weather Resistant [12] and is suitable for flameproofed textiles, in general.

A specimen 2 inches wide by $12\frac{1}{2}$ inches long is clamped in a vertical position with the lower end free, and $\frac{1}{2}$ inch of the upper end in the clamp, so that a 12-inch length is exposed. The lower end of the specimen is $\frac{3}{4}$ inch above the top of a Bunsen or Tirrill gas burner which has a tube of $\frac{3}{8}$ inch inside diameter. A $1\frac{1}{2}$ -inch luminous flame is used for ignition and is applied to the center of the lower end of the specimen for 12 sec. To protect the sample from drafts, the apparatus is enclosed in a sheet-metal shield 14 inches wide, 12 inches deep, and 30 inches high, open at the top, and provided with a door containing a glass inset for observing the burning specimen. A metal bar secured to the base of the burner and extending outside the cabinet through a slot in one side permits manipulation of the burner without opening the door. In this way the burner is moved under the specimen for the ignition period only, and the flame is left burning continuously throughout a series of tests.

A record is made of the time the specimen continues flaming after the igniting flame is removed, the time of glow after flaming has ceased, and the char length. "Char length" is defined as the distance from the end of the specimen that was exposed to the fire to the end of a tear made lengthwise through the center of the charred area in the following way. A hook is inserted in the specimen at one side of the charred area $\frac{1}{4}$ inch in from the adjacent outside edge. A weight that, together with the hook, is equal to approximately 10 percent of that required to tear the unburned cloth is attached to the hook, and a tearing force is applied gently to the specimen by grasping the corner of the fabric at the other side of the char and raising the specimen and the weight clear of the support.

The following criteria indicate satisfactory performance of flame-proofed material:

No specimen shall continue flaming for more than 2 sec after the igniting flame is withdrawn. The average char length of 10 specimens shall be not more than $3\frac{1}{2}$ inches, and the maximum length of char for any one of the 10 specimens shall be not more than $4\frac{1}{2}$ inches. For very thin fabrics, however, these requirements are somewhat severe. Although the flame spread is greatest for the specimen in vertical position, as in this test, it is not as severe as a multiple vertical strip- or fold-test but gives a greater difference in results between well-treated and poorly treated fabrics.

Although fairly consistent results have been obtained with the vertical test just described, the following modifications have been applied recently to enable closer control of test conditions:

Making the specimen wider and securing the vertical edges in a light sheet-metal clamp to give an exposed width of 2 inches centered over the flame.

Connecting the gas-supply line for the burner to a gas chamber to reduce fluctuations in the flame; and for the same purpose,

Applying top closure to the shield or box around the burner with a baffled vent placed therein above the burner location.

2. CLAMP FIELD TEST

This test [19] was designed for field use and gives results comparable to those of the vertical laboratory flame test described above. The clamp field test may be applied to any horizontal edge or slit in the

fabric (or any edge that can be placed in a horizontal position for testing) without the necessity of cutting out samples or of removing the material from its position.

The clamp is made of duplicate rectangular pieces of sheet metal about $1/16$ inch thick and having an area 2 inches wide by 5 inches deep cut from the center of one end, leaving U-shaped plates. The two plates are clamped together by spring-type paper clips, $1\frac{1}{4}$ inches wide, at the ends of the U-prongs. The clamp is used with the U in an inverted position, the cloth being slipped between the plates until the horizontal edge of the cloth is even with the ends of the U-prongs. Thus, an area of cloth 2 inches by 5 inches is exposed, held in a vertical position, and firmly clamped in a metal shield, which prevents flame from spreading beyond the exposed area. The source of ignition is a paraffin candle $\frac{3}{4}$ inch in diameter carried in a holder hinged to one side of the clamp in such manner that the candle can be swung away from the fabric for adjustment. When used for ignition, the candle holder swings against a stop when the center of the candle is directly under the 2-inch wide exposed lower edge of the specimen. Before use the tapered portion of the candle is allowed to burn until a normal constant flame is obtained. It is then adjusted in the holder so that the top of the wick will be $1/10$ inch below the edge of the fabric when in position. To ignite the fabric the flame is applied to the edge of the fabric for 12 seconds.

A satisfactorily flameproofed fabric will meet the following requirements: It will not continue flaming more than 2 sec. after the igniting flame is removed. The average length of char in three tests will be not more than $2\frac{1}{2}$ inches. By "length of char" is meant the distance from the edge of the fabric to the end of a tear through the charred area, made by hand with enough force to tear through the charred or scorched portion but not sufficient to break undamaged threads.

Vertical flame tests, such as those described above, are too severe for satisfactory application to fabrics that have not been flameproofed, as nearly all untreated fabrics burn so rapidly that little differentiation is possible. For the comparison of rates-of-burning of such untreated fabrics, the National Bureau of Standards has developed the following horizontal test:

3. HORIZONTAL RATE-OF-BURNING TEST

A specimen $4\frac{1}{2}$ inches wide and $12\frac{1}{2}$ inches long is cut from the cloth in the warp direction or in the direction indicated as most hazardous by the pattern of pile or tufting. It is slipped into a steel frame that clamps each long edge of the specimen, leaving a center strip 2 inches wide and $12\frac{1}{2}$ inches long taut and exposed. The frame is $15\frac{1}{2}$ inches long by 4 inches wide and 1 inch thick in over-all size, and consists of two matching sections made of $\frac{1}{2}$ -inch-thick nickel-plated steel. The sections are alined by means of two pins at one end of the lower section, which fit into holes in the upper section. The sides of the frame are 1 inch wide, leaving an open center space 2 inches wide by $13\frac{1}{2}$ inches long, so that there is a $\frac{1}{2}$ -inch space between the frame and each end of the inserted sample. Heat-resistant marking wires attached to the upper section, but clear of the specimen, cross it at a point $1\frac{1}{2}$ inches from the end that is to be ignited and 1 inch from the other end, which leaves a specimen length of 10 inches

between the wires. A third wire, crossing at the end that is to be ignited, serves as a guide for adjusting the position of the sample in the frame.

During a test the frame is supported horizontally in a metal cabinet, 8 inches wide, 15 inches long, and 14 inches high inside, to protect the burning sample from drafts. It is inserted through a slot in one end of the cabinet and slides on a track until it strikes a stop. When the center of the end of the specimen is $\frac{3}{4}$ inch above the top of a $\frac{3}{8}$ -inch Tirrill gas-burner tube. A $1\frac{1}{2}$ -inch luminous flame is used for ignition and may be left burning continuously throughout a series of tests. A large wheel extending outside the cabinet is attached to the gas-supply valve of the burner to permit easy regulation of the flame height. Five $\frac{3}{4}$ -inch holes in each side of the raised floor of the cabinet and a $\frac{1}{2}$ -inch free space all around under the cover provide ventilation. Two electric strip heaters, installed one on each side a little above the ventilating holes, are operated to maintain a temperature of approximately 140°F inside the cabinet, as determined by a partial immersion thermometer inserted through the cover. The front of the cabinet and spaces in the cover are glazed to enable observation of the sample. The time required for the flame front to travel the 10 inches between the marking wires (a $1\frac{1}{2}$ -inch length burns before the timing is started) is measured with a stop watch, and the rate of burning or flash (flame flashing through the nap) is calculated.

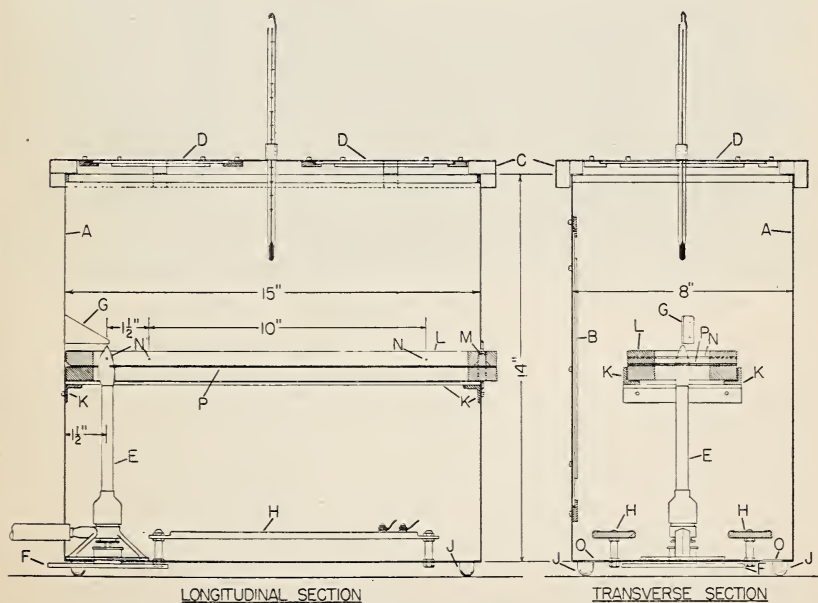


FIGURE 1.—Details of horizontal rate-of-burning equipment.

A, Cabinet of No. 18 gage steel; B, glass observation window; C, removable cover with $\frac{1}{2}$ in. ventilating clearance all-round; D, Pyrex observation windows; E, Tirrill burner, $\frac{3}{8}$ -in. tube; F, adjusting wheel attached to burner needle valve; G, gage for flame height; H, electric strip heaters; J, supports raising cabinet $\frac{3}{8}$ in. from table; K, support and slide for sample frame; L, sample frame, nickel-plated steel; M, aligning pins for sample frame; N, marking wires; O, five $\frac{3}{4}$ -in. ventilating holes on each side; P, sample in frame.

The details of the equipment are shown in figure 1, and a photograph thereof in figure 2. The dimensions of the cabinet and method of inserting and igniting the specimen can be varied somewhat without appreciably changing the results. If matches or candles are used for ignition, the frame is turned around before being inserted into

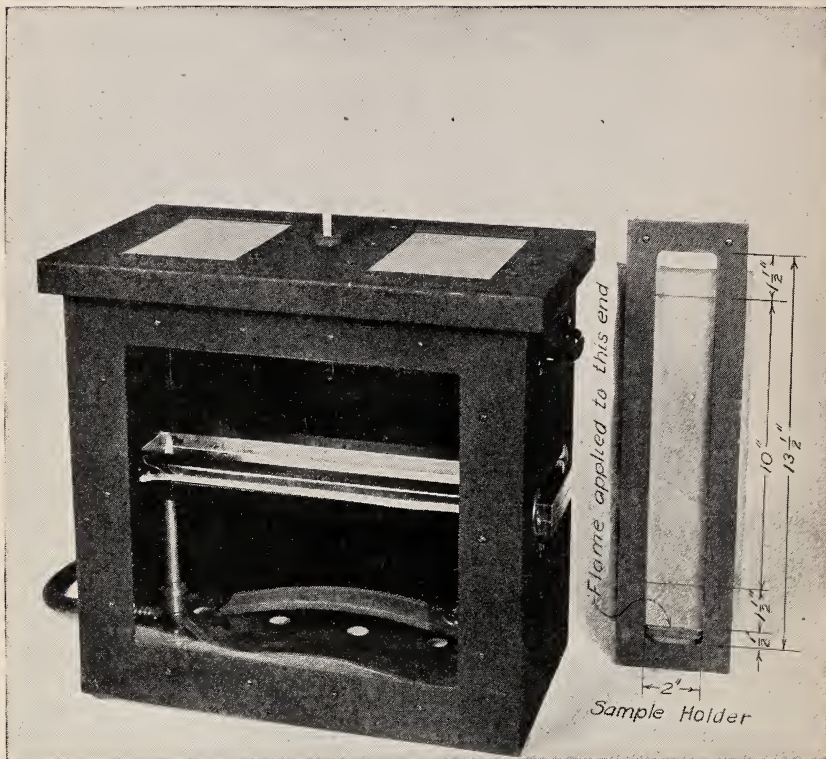


FIGURE 2.—Horizontal rate-of-burning apparatus.

the cabinet, and the end to be ignited is left protruding until ignition is accomplished, when it is pushed inside. There should be sufficient ventilation for free combustion without direct drafts on the burning sample. A $1\frac{1}{2}$ -inch length of the sample should burn before the significant length is reached over which the burning rate is determined, in order to obtain a uniform and easily observable flame front.

In the test procedure used at the National Bureau of Standards the samples are conditioned at 140°F for 1 hr before a test. Upon removal from the conditioning oven, if the fabric has a nap, or pile, the sample is combed to restore the nap. This is accomplished by drawing it slowly under a coarse comb having seven to eight teeth per inch, with smooth rounded ends and weighted to approximately 7 grams per tooth in contact with the fabric. The specimen is combed twice against the nap before it is placed in the sample frame for testing. With the burner flame turned as low as possible, the frame is pushed

into the cabinet until the end of the frame is over the flame. In this position the entire length of the sample is inside the cabinet, but there is no possibility of premature ignition while the sample is held in the cabinet for a 2-min reconditioning period before the test is made. After 2 min the frame is moved from its position over the burner, the flame adjusted to proper height, and the sample ignited.

For use with double-faced napped fabrics, particularly chenilles with tufting on both sides, a stop is used to prevent a flash from traveling across the underside of the fabric and igniting the other end of the sample before the flash has traveled across the upper surface where it can be accurately timed. The stop consists of a cross strip of No. 26 gage sheet steel, $\frac{1}{4}$ inch wide, removable and fitted on the lower section of the frame midway of its thickness, or $\frac{1}{4}$ inch below the fabric mounting plane. The stop strip crosses the under surface of the sample about 3 inches from the end to be ignited and is far enough below the fabric to have no effect on the burning over the upper surface, while it effectively prevents a quick flash over the lower surface.

The rate of burning of a fabric is given as the average of not less than five tests unless the spread between individual results is large. If the spread is more than 40 percent of the average, 10 tests should be made, and the average flash or burning rate based on the five highest rates from the individual tests.

Most fabrics when subjected to this test burn at a rate well under 15 inches a minute. Some fine-napped materials have shown a flash rate of 400 inches a minute. Light netting and thin, coated fabrics give intermediate results.

4. CONDITIONING OF TEST SAMPLES

A procedure for conditioning samples before testing, regardless of the test employed, has not been satisfactorily standardized, although it is generally agreed that conditioning is an important factor in performance. For most tests of textiles—strength, weight, etc.—the samples are conditioned for at least 24 hr at 70°F and 65-percent relative humidity. It is questionable whether relative humidity as high as 65 percent is representative for fire-hazard tests as room humidities, particularly during the heating season, are generally much lower. It has been the practice at the National Bureau of Standards to dry the samples in a large ventilated oven at temperatures of 100° to 140°F until equilibrium is reached, after which they are kept in a container conditioned with chemicals that hold the relative humidity at about 20 percent. The preliminary drying, particularly for samples that have been subjected to accelerated weathering, is important as a very large amount of moisture cannot be extracted by the humidity jar.

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