United States Patent [19]

Golborn et al.

[54] DIALKYL ALKYL AND AROMATIC SULFONAMIDOMETHYL PHOSPHONATES

- [75] Inventors: Peter Golborn, Lewiston; James J. Duffy, Buffalo, both of N.Y.
- [73] Assignee: Hooker Chemical & Plastics Corporation, Niagara Falls, N.Y.
- [22] Filed: Mar. 30, 1972
- [21] Appl. No.: 239,757
- [52] U.S. Cl..... 260/944, 117/136, 260/45.9 R,
- [58] Field of Search...... 260/944
- [56] **References Cited**
- UNITED STATES PATENTS

2,635,112 4/1953 Fields...... 260/944 FOREIGN PATENTS OR APPLICATIONS

1,226,577 10/1966 Germany 260/944

[11] **3,870,771**

[45] Mar. 11, 1975

Primary Examiner—Lorraine A. Weinberger Assistant Examiner—Richard L. Raymond Attorney, Agent, or Firm—Peter F. Casella; Donald C. Studley; William J. Crossetta, Jr.

[57] ABSTRACT

New compounds are disclosed of the formula:

RSO2NHCH2P(OR')2

wherein R is selected from the group consisting of lower alkyl of 1–6 carbon atoms, phenyl and alkyl substituted phenyl and R' is selected from the group consisting of phenyl, lower alkenyl and halogen substituted and unsubstituted lower alkyl of 1–6 carbon atoms. The compounds of this invention are useful as flame retarding agents for textile materials and in the production of polymers and copolymers which possess flame retardant properties.

6 Claims, No Drawings

DIALKYL ALKYL AND AROMATIC SULFONAMIDOMETHYL PHOSPHONATES

FIELD OF INVENTION

This invention relates to novel compounds of the for- 5 mula

RSO2NHCH2P(OR')2

wherein R is selected from the group consisting of lower alkyl of 1-6 carbon atoms, phenyl and alkyl substituted phenyl and R' is selected from the group consisting of phenyl, lower alkenyl and halogen substituted and unsubstituted lower alkyl of 1-6 carbon atoms. The 15 invention includes methods of applying the above novel compounds to normally flammable textiles and thermoplastic or thermosetting resin compositions so as to render them flame retardant.

BACKGROUND OF THE INVENTION

Many flame retarding agents and methods of application have been developed in attempts to obtain flame resistant textile materials and thermoplastic or thermosetting resin compositions.

Flame retardant textiles have been produced by depositing metal oxides, within or on the textile fibers, by the successive precipitation of ferric oxides and a mixture of tungstic acid and stannic oxide or by successive deposition of antimony trioxide and titanium dioxide. 30 Such processes require plural treatment baths in which strongly acidic solutions are employed thus posing the problem of possible textile degradation. Furthermore, metal oxide coatings on textile materials create difficulties in subsequent dyeing processes which deleteriously ³⁵ affect the hand of the finished product. Another process involves the use of a single processing bath wherein a dispersion of a chlorinated hydrocarbon and finely divided antimony oxide is padded on the textile material. Near the textile combustion temperature anti- 40mony oxide will react with hydrogen chloride, generated by degradation of the chlorinated hydrocarbon, to form antimony oxychloride which acts to suppress flame. This combination of a chlorinated hydrocarbon and finely divided antimony oxide are not acceptable finishes for closely woven textiles as they deleteriously affect the hand of the finished product. A further process for imparting flame resistance to cellulosic materials is by the esterification of the cellulose with diammo-50 nium hydrogen orthophosphate. Textile products so treated however are subjected to metathesis reaction with cations during washing, and must be regenerated by reacting the wash product with an ammonium chloride solution.

The production of thermoplastic resin compositions ⁵⁵ which are flame retardant is of considerable commercial importance. For example, such articles as castings, moldings, foamed or laminated structures and the like are required, or are at least desired, to be resistant to fire and flame and to possess the ability to endure heat without deterioration. The use of various materials incorporated into thermoplastic resins so as to improve the flame retardancy thereof has been known. Many compounds have been commercially available for such use, among them being chlorostyrene copolymers, chlorinated paraffin wax in admixture with triphenyl styrene, chlorinated paraffins and aliphatic antimonical

compounds, as well as antimony oxide - chlorinatd hydrocarbon mixtures. A problem associated with these compounds has been however, the fact that generally a large amount, i.e. upwards of 35 percent of additive, must be incorporated into the resin in order to make it sufficiently flame retardant. Such large amounts of additive may deliteriously affect the physical characteristics of the thermoplastic resin, as well as substantially complicating and increasing the cost of preparation 10 thereof. A further problem is that these prior art additives tend to crystallize or oil out of the resin after a relatively short time of incorporation. The present invention relates to a group of compounds which may be added to thermoplastic resins in relatively small amounts and still produce satisfactory flame retardant compositions which will not crystallize nor oil out of

OBJECTS OF THE INVENTION

20 It is, therefore, a principal object of this invention to provide novel compounds of the formula:

the resin after incorporation therein.

RSO₂NHCH₂P(OR')₂

wherein R is selected from the group consisting of lower alkyl of 1-6 carbon atoms, phenyl and alkyl substituted phenyl and R' is selected from the group consisting of phenyl, lower alkenyl and halogen substituted and unsubstituted lower alkyl of 1-6 carbon atoms.

It is also an object of this invention to provide flame retarding textile materials comprising normally flammable cellulosic, proteinaceous or blends thereof. Another object is to provide a method for treating normally flammable cellulosic, proteinaceous or blends thereof to render them flame retardant. Another object is to provide flame retarding thermoplastic or thermosetting resin compositions comprising normally flammable thermoplastic or thermosetting resin materials.

⁴⁰ A further object is to provide a process for treating normally flammable thermoplastic or thermosetting resin compositions to render them flame retardant. A particular object is to devise a composition comprising normally flammable cellulosic, proteinaceous or analogous man-made materials and an effective flame retardant amount of the compound represented by the formula

RSO2NHCH2P(OR')2

wherein R and R' are as above described.

A further particular object is to devise a composition comprising normally flammable thermoplastic or thermosetting polymer and an effective flame retarding amount of the before described novel compound.

These and other objects of the present invention will be obvious from the following description.

DESCRIPTION OF THE INVENTION

In accordance with this invention there are provided novel compounds, for imparting flame retardancy to textiles and thermoplastic or thermosetting resin materials, of the formula

25

5

wherein R is selected from the group consisting of lower alkyl of 1-6 carbon atoms, phenyl and alkyl substituted phenyl and R' is selected from the group consisting of phenyl, lower alkenyl and halogen substituted and unsubstituted lower alkyl of 1-6 carbon atoms. More specifically, the preferred compounds of the present invention include these compounds wherein R and R' are lower alkyl of 1-6 carbon atoms.

Illustrative examples of compounds of the present invention include, for instance, compounds of the gen- 10 CH₃-CH₂ eral formula such as

. 0

$$CH_{3} - SO_{2} - NH - CH_{2} - P(OCH_{3})_{2}$$

$$CH_{3} - CH_{2} - SO_{2} - NH - CH_{2} - P(OCH_{3})_{2}$$

$$CH_{3} - CH_{2} - CH_{2} - CH_{2} - SO_{2} - NH - CH_{2} - P(OCH_{3})_{2}$$

$$CH_{3} - SO_{2} - NH - CH_{2} - P(OCH_{2} - CH_{3})_{2}$$

$$CH_{3} - SO_{2} - NH - CH_{2} - P(OCH_{2} - CH_{2})_{2}$$

$$CH_{3} - SO_{2} - NH - CH_{2} - P(OCH_{2} - CH_{2}CH_{2})_{2}$$

$$CH_{3} - SO_{2} - NH - CH_{2} - P(OCH_{2} - CH_{2}CH_{2})_{2}$$

$$CH_{3} - SO_{2} - NH - CH_{2} - P(OCH_{2} - CH_{2}CH_{2})_{2}$$

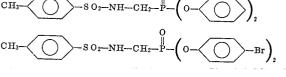
$$CH_{3} - SO_{2} - NH - CH_{2} - P(OCH_{3})_{2}$$

$$CH_{3} - OCH_{3} - SO_{2} - NH - CH_{2} - P(OCH_{3})_{2}$$

$$CH_{3} - OCH_{3} - SO_{2} - NH - CH_{2} - P(OCH_{3})_{2}$$

$$CH_{3} - OCH_{3} - SO_{2} - NH - CH_{2} - P(OCH_{2} - CH_{2}CH_{2}CH_{2})_{2}$$

$$CH_{3} - OCH_{3} - SO_{2} - NH - CH_{2} - P(OCH_{2} - CH_{2}CH_{2}CH_{2})_{2}$$



The synthesis of the compositions of the present invention is accomplished by reacting an N- 55 hydroxymethyl sulfonamide of the formula

R SO₂NHCH₂OH

with a trialkyl phosphite of the formula

(

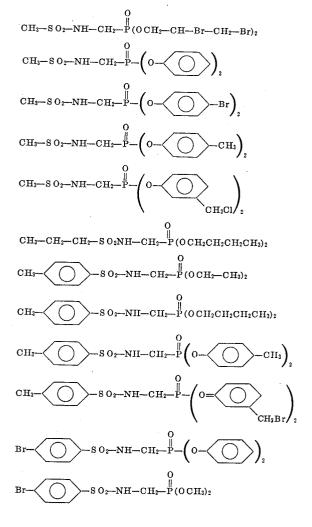
60

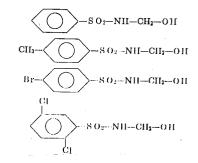
wherein R and R' are as previously described in a suitable solvent, with an excess of the phosphite, or without solvent. Typically, the reaction is continued for 65 about 1 to about 12 hours. Temperatures are generally about 50°C to about 160°C. Preferably reaction is continued from about 3 to about 6 hours at a temperature

of about 80°C to about 120°C. The solvent or other volatiles as thereafter stripped, or otherwise removed from the product. Suitable solvents include benzene, toluene, xylene, the glymes, dimethylformamide and aliphatic or aromatic hydrocarbons. Typical Nhydroxymethyl sulfonamides operable as reactants herein include

CH₃-SO₂-NH-CH₂-OH

CH₃-CH₂-CH₂-SO₂-NH-CH₂-OH $H-CH_2-OH$





One or more of the novel compounds of this invention may be applied to textile materials by conventional finishing techniques such as by thermal curing so as to incorporate into the textile a flame retardant amount thereof. The compounds of this invention have advantages over the flame retardant agents of the prior art in that they may be used on a variety of textile materials of different chemical composition, and they may be applied by a variety of methods. They may be applied to materials in either the fiber or fabric form to give flame retarding materials with minimum detectable physical changes in the quality or hand of the textile material.

Cellulosic textile materials may be made flame retardant by way of a variety of methods. Typically, the cellulose products of this invention may be applied to cellulosic materials in several ways to give a durable flame retardant treatment. For example, the products of this invention may be reacted with formaldehyde to give Nhydroxymethyl derivatives which can react with cellulosic materials in a known manner. Alternatively aqueous mixtures of the products with formaldehyde, urea, trimethylol melamine or other known cellulose crosslinking agents may be applied to a cellulose substrate with the acid of an acidic catalyst by pad dry process.

More preferably the N-hydroxymethyl derivative of the products of this invention prepared by the condensation of the products with formaldehyde, are mixed in an aqueous medium with trimethylol melamine and a Lewis acid catalyst such as NH₄Cl or Zn(NO₃)₂·6H₂O. The cellulosic material is immersed in a aqueous solution of the methylol derivative, trimethylol melamine, and Zn(NO₃)₂·6H₂O and squeezed on a two roll padder to 70-90 percent wet weight pick-up. The material is dried at 220°-270°F for 1-3 minutes and cured at 300° -370°F for 1-6 minutes in a circulating air oven. The samples are then washed in hot water and dried. The finished samples have a flame retardant add-on of about 5 to about 40 percent and preferably about 10 to about 25 percent by weight.

The flame retardant agents of this invention may be applied to various textiles such as cellulosic materials, proteinaceous materials and blends of cellulosic or pro- 40 teinaceous materials. By cellulosic materials, applicant intends to embrace cotton, rayon, paper, regenerated cellulose and cellulose derivatives which retain a cellulose backbone of at least one hydroxy substituent per repeating glucose unit. By proteinaceous material ap- 45 plicant intends to embrace those textile materials comprising the functional groups of proteins such as the various animal wools, hairs and furs. The flame retardant compounds or additives of the invention may be incorporated into thermoplastic or thermosetting resin 50 compositions by any known method. That is to say, the flame retardant additive may be added to the resin by milling the resin and the additive on, for example, a two-roll mill, or in a Banbury mixer etc., or it may be added by molding or extruding the additive and resin ⁵⁵ simultaneously, or by merely blending it with the resin in powder form and thereafter forming the desired article. Additionally, the flame-retardant may be added during the resin manufacture, i.e., during the polymerization procedure by which the resin is made, provided ⁶⁰ the catalysts etc. and other ingredients of the polymerization system are inert thereto. Generally, the compounds of this invention may be incorporated into the thermoplastic or thermosetting resin in flame-retarding 65 amounts, i.e. generally amounts ranging from about 5 percent by weight, to about 50 percent by weight, preferably from about 20 percent by weight, to about 40

6

percent by weight, based on the weight of the polymer, have been found sufficient.

The thermoplastic resin embraced within the scope of this invention include the homopolymers and copolymers of unsaturated aliphatic, alicyclic, and aromatic hydrocarbons. Suitable monomers are ethylene, propylene, butene, pentene, hexene, heptene, octene, 2-methylpropene-1, 3-methylbutene-1, 4-methylpentene-1, 4-methylhexene-1,5-methylhexene-1, bicyclo-(2.2.1)-2-heptene, butadiene, pentadiene, hexadiene, isoprene, 2,3-dimethylbutadiene-1,3, 2-methylpentadien-1,3 4-vinylcyclohexene, vinylcyclohexene, cyclopentadiene, styrene and methylstyrene, and the like.

Other polymers in addition to the above-described 15 olefin polymers that are useful in the invention include polyindene, indenecoumarone resins; polymers of acrylate esters and polymers of methacrylate esters, acrylate and methacrylate resins such as ethyl acrylate, nbutyl methacrylate, isobutyl methacrylate, ethyl metha-20 crylate and methyl methacrylate; alkyd resins and paint vehicles, such as bodied linseed oil; cellulose derivatives such as cellulose acetate, cellulose acetate butyrate, cellulose nitrate, ethyl cellulose, hydroxyethyl cellulose, methyl cellulose and sodium carboxymethyl cel-25 lulose; epoxy resins; furan resins (furfuryl alcohol or furfuralketone); hydrocarbon resins from petroleum; isobutylene resins (polyisobutylene); isocyanate resins (polyurethanes); melamine resins such as melamineformaldehyde and melamine-urea-formaldehyde; oleoresins; phenolic resins such as phenol-formaldehyde, phenolic-elastomer, phenolic-epoxy, phenolicpolyamide, and phenolic-vinyl acetals; polyamide polymers, such as polyamides, polyamide-epoxy and particularly long chain synthetic polymeric amides containing recurring carbonamide groups as an integral part of the main polymer chain; polyester resins such as unsaturated polyesters of dibasic acids and dihydroxy compounds, and polyester elastomer and resorcinol resins such as resorcinol-formaldehyde, resorcinol-furfural, resorcinol-phenol-formaldehyde, resorcinol-polyamide and resorcinol-urea; rubbers such as natural rubber, synthetic polyisoprene, reclaimed rubber, chlorinated rubber, polybutadiene, cyclized rubber, butadieneacrylonitrile rubber, butadiene-styrene rubber, and butyl rubber; neoprene rubber (polychloroprene); polysulfides (Thiokol); terpene resins; urea resins; vinyl resins such as polymers of vinyl acetal, vinyl acetate or vinyl alcohol-acetate copolymer, vinyl alcohol, vinyl chloride, vinyl butyral, vinyl chloride-acetate copolymer, vinyl pyrrolidone and vinylidene chloride copolymers; polyformaldehyde; polyphenylene oxide; polymers of diallyl phthalates and phthalates; polycarbonates of phosgene or thiophosgene and dihydroxy compounds such as bisphenols, phosgene, thermoplastic polymers of bisphenols and epichlorohydrin (trade named Phenoxy polymers); graft copolymers and polymers of unsaturated hydrocarbons and unsaturated monomer, such as graft copolymers of polybutadiene, styrene and acrylonitrile, commonly called ABS resins; ABS polyvinyl chloride polymers, recently introduced under the trade name of Cycovin; and acrylic polyvinyl chloride polymers, known by the trade name Kydex 100.

The polymers of the invention can be in various physical forms, such as shaped articles, for example, moldings, sheets, rods, and the like; fibers, coatings, films and fabrics, and the like.

The compounds of this invention have been found to have particular utility in ABS resins and in elastomeric materials such as acrylic rubber; acrylonitrilebutadiene styrene terpolymers; butadieneacrylonitrile copolymers; butyl rubber; chlorinated rubbers, e.g., 5 polyvinyl chloride resins, chloroprene rubber, chlorosulfonated polyethylene; ethylene polymers, e.g., ethylene-propylene copolymers, ethylene-propylene terpolymers; fluorinated rubbers, butadiene rubbers, e.g., butadiene polymers, polyisobutylene rubbers, polyisoprene rubbers; polysulfide rubbers; silicon rubbers; urethane rubbers; high styrene resins latices, high styrene resins, vinyl resins; sponge rubber; and the like.

the present invention to incorporate such ingredients as plasticizers, dyes, pigments, stabilizers, antioxidants, antistatic agents and the like to the novel composition.

ASTM Test D2863-70, used in accordance with the following examples, generally provides for the compar- 20 ison of relative flammability of self-supporting plastics by measuring the minimum concentration of oxygen in a slowly rising mixture of oxygen and nitrogen that will support combustion. The procedure encompasses supporting cylindrical test specimens 70-150 mm in length 25 \times 8.0 mm in diameter vertically in a glass tube fitted with controlled upward oxygen/nitrogen gas flow. The top of the specimen is ignited and oxygen flow is adjusted until it reaches that minimum rate at which the specimen is extinguished before burning 3 minutes or 3050 mm whichever happens first. The oxygen index(n)is then calculated as follows:

$n_{.}\% = (100 \times O_{2})/(O_{2} + N_{2})$

wherein O_2 is the volumetric flow of oxygen, at the min-³⁵ imal rate and N₂ is the corresponding volumetric flow rate of nitrogen.

A modification of ASTM Test D635-68 used in accordance with the following examples, generally provides for the comparison of burning rates, self- 40 extinguishment and non-burning characteristics of plastics in the form of sheets, bars, plates or panels. The procedure encompasses preparing 150-200 mm × 8 mm cylindrical plastic test samples with an without the 45 subject flame retardant additive. Each sample is marked at points 1 inch and 4 inches from its end and held, marked end in the flame, at a 45° angle in a controlled burner flame (1 inch flame length) for two 30 second attempts. The movement of the flame up the 50 length of the sample through the two points is measured for rate of burning, non-burning or selfextinguishing characteristics. A sample is rated SE(selfextinguishing) if the flame burns through the first point but extinguishes before reaching the second point. A 55 sample is rated NB(non-burning) if, upon ignition it does not burn to the first point.

AATCC test method 34-1969. The vertical Char Test, used in accordance with the following examples, generally provides for the comparison of relative flam-60 mability of 2% inch \times 10 inch fabric test specimens when exposed to a controlled burner flame, under controlled conditions, for periods of 12.0 and 3.0 seconds. Charred specimens are thereafter subjected to controlled tearing tests, using tabulated weights, to deter-65 mine the average tear length as representing the char length of the fabric. In addition, samples which are wholly consumed by the flame are rated (B) and samples which do not burn are rated (NB). For comparison purposes, it should be noted that untreated samples of the fabrics used in the examples of this case would be consumed for this test.

In all the examples of the application, the following general procedure was used except when otherwise specifically noted. Padding was done on a standard two roll laboratory padder at a gauge pressure of about 60 pounds per square inch in all cases. Drying and curing styrene-butadiene rubber, isobutylene polymers, poly- 10 during processing were done with a standard laboratory textile circulating air oven. Washing and drying was done in a standard, home, top loading, automatic washer and dryer.

The following examples are set forth for purposes of It should be noted that it is also within the scope of 15 illustration only and are not to be construed as limitations of the present invention except as set forth in the appended claims. All parts and percentages are by weight unless otherwise specified.

EXAMPLE I

A 1 liter flask, equipped with a mechanical stirrer, thermometer and reflux condenser, was charged with 171.2g (1.0 mole) of p-toluenesulfonamide, 30g. (1.0 mole) of paraformaldehyde and 200 ml. methanol. Sodium methoxide was added to the solution to obtain a pH of 10 and thereafter the solution was refluxed for 48 hours and cooled. 150g. (1.2 mole) of trimethyl phosphite was added to the cooled reaction mixture (N-hydroxymethyl-p-toluene sulfonamide) and, after an induction period of about 5 minutes, a vigorous exotherm ensued causing the reaction mixture to reflux. After the exotherm had subsided the reaction solution was heated at reflux for 15 hours and then cooled to room temperature. On standing, colorless crystals separated from the solution. The crystals were filtered off the solution and dried resulting in a yield of 179g. A further concentration of the mother liquor, after filtration, yielded a further 40g. of crystalline material. Total yield of product was 219g. of a colorless cyrstalline solid analyzed as essentially pure N-dimethyl phosphonomethyl-p-toluene sulfonamide.

Elemental	Calculated	C,41.0; H,5.5; N,4.8; P,10.6
Analyses:	Found	C,40.6; H,5.7; N,4.6; P,10.5

EXAMPLE II

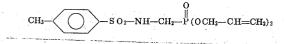
Following the procedure described in Example 1. Sodium methoxide was added to a solution of 95.1g (1.0 mole) of methane-sulfonamide, 30g. (1.0 mole) of paraformaldehyde and 300 ml. of methanol until a pH of 10 was obtained. The reaction mixture was refluxed for 15 hours then cooled and 124g. (1.0 mole) of trimethyl phosphite added. Refluxing was then continued for a further 15 hours. The reaction solution was stripped of methanol at 70° under a vacuum of 20 mm. mercury to yield 216g. of a colorless liquid product. Analyzed as essentially pure N-dimethyl phosphonomethyl methyl sulfonamide.

Elemental	Calculated	C,22.1; H,5.5; N,6.5; P,14.3	
Analyses:	Found	C,21.8; H,6.0; N,5.9; P,14.5	

5

25

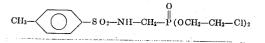
Preparation of



N-Hydroxymethyl-p-toluene sulfonamide prepared as in Example 1. Triallyl phosphite, 40.2g. (0.2 mole) was added to the hydroxymethyl compound, 20.1g. (0.1 mole), in a round bottomed flask, and warmed to and maintained at about 125°C for three hours. The mixture was then stripped at about 0.5 mm Hg and about 100°C to remove all volatile material. The product was 34.5g. of a colorless liquid whose structure was confirmed by infrared and nuclear magnetic resonance spectroscopy and elemental analysis to be that of the claimed product.

EXAMPLE IV

Preparation of



N-Hydroxymethyl-p-toluene sulfonamide was prepared as in Example 1. The hydroxymethyl derivative, $_{30}$ 20.1g. (0.1 mole) was then added to 27g. (0.1 mole) of tris-2-chloroethyl phosphite and warmed to 120°C. The reaction mixture was held at 120°C for two hours and then stripped at 100°C and 0.5 mm Hg to give the desired product. The product, a light brown solid, was ob- 35 tested by the padding solution and procedure of Examtained in quantitative yield and elemental and spectroscopic analysis showed it to be essentially pure.

analyses, infrared and nuclear magnetic spectroscopy confirming the structure as the desired product.

EXAMPLE VI

A padding solution was prepared by mixing Ndimethylphosphonomethyl-p-toluenesulfonamide,

(40.0g) with 60.0g of 40 percent formalin solution of a pH of 9-10 and stirring overnight. The pH was adjusted to 7.0 with hydrochloric acid and 23g. of a 50 10 percent solution of a methylolated melamine and 5g of ammonium chloride added.

6.0 oz. sq. yd. wool textile material was padded through the above mixture and the excess squeezed out by means of a two roll laboratory padder at 60 lb. gauge 15 pressure to a wet pickup of about 130 percent. The textile material was dried at about 250°F for about 2 minutes, and cured at about 350°F for about 4 minutes in a circulating air oven. Samples of the textile material was then washed in a standard automatic home washer, 20 for one or five wash cycles, using Tide as the detergent, and thereafter, subjecting to AATCC test method 34-1969 to determine flammability, the results therefore indicated in Table 1.

EXAMPLE VII

A padding solution, using dimethyl phosphonomethyl methane sulfonamide, was prepared as in Example VI. Cotton cloth 5.0 oz. sq. yd. was treated with the above padding solution in accordance with Example VI with results as indicated in Table 1.

EXAMPLE VIII

6.0 oz. sq. yd. wool textile material was treated and ple VII. AATCC test method 34-1969 results are as indicated in Table 1.

<u></u>			ТАВ	LEI ·						
<u> </u>		Compound				Percent wet pickup		Initial	Flammability	
Example	Substrate							OI	Initial	5 washes
VI	Wool	сн3-	>−S02-NH-CH	0 P(0(CH3)2			24	3.7	5.1
VII	Cotton	CII3-SO2-CH	$ \sum_{2}^{0} P(OCH_3)_2 $					22	5, 5	6.1
IX	Rayon	снз-	>−s0₂−NH−CH₂	-1'(0(H ₂ CH=CH ₂)	100	38	23	SE	SE
VIII	Wool	CH3-SO2-CH	$ \overset{O}{\parallel} \\ - P(OCH_3)_2 $			130	77	28	3.2	3 . 2
	FXAM	PIFV	5	5	· · · · · · · · · · · · · · · · · · ·	EVA				

EXAMPLE V

Preparation of

$$CII_{1}-SO_{2}-NII-CII_{2}-V = \left(O-O\right)_{2}$$

N-Hydroxymethyl methane sulfonamide was prepared as in Example 2 above. The hydroxymethyl derivative, 12.5g (0.1 mole) was added to 31.0g of triphenyl phosphite (0.1 mole), in a round bottomed flask, and heated at about 130°C for about five hours. 65 At the end of this time the reaction mixture was stripped, at about 2 mm Hg and about 120°C, to remove phenol and other volatiles. A viscous yellow liquid, was obtained in quantitative yield, with elemental

EXAMPLE IX

А padding solution, using Ndiallylphosphonomethyl-p-toluenesulfonamide, was prepared as in Example VI. Rayon sample fiber mate-60 rial was treated with the above padding solution in accordance with Example VI with the exception that drying and curing was at 250°F for about 3 minutes and 350°F for about 7 minutes respectively. Testing of the rayon sample fibers was accomplished by holding samples thereof in a controlled burner flame for 2 seconds then removing and observing for self-extinguishment (SE). The treated rayon fibers were self-extinguishing upon removal while untreated fibers were consumed.

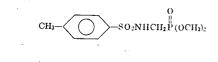
EXAMPLE X

30 parts of N-dimethyl phosphonomethyl-p-toluene sulfonamide was mixed with 70 parts of polypropylene and dry blended for about 5 minutes. This composition 5 was then heated to a melt and mixed in the molten state for about 15 minutes. The composition was allowed to cool and solidify after which it was cut into small pieces. These small composition pieces were then slowly placed into a 9mm glass tube, immersed in a hot 10 metal salt bath, maintained at a temperature above the melting point of the composition. A metal rod was then placed in the tube with a weight attached therto and the tube was cooled to solidify the composition. The composition was then removed from the tube and tested by 15 ASTM test methods D2863-70 and D635-68. The results of the testing are tabulated in Table II.

EXAMPLES XI-XIX

Using the same procedure as Example X, samples 20 were prepared using different sulfonamidomethyl phosphonate in varius plastics. The results of flammability testing thereon is indicated in Table II.

2. A compound of claim 1 of the formula



3. A compound of claim 1 of the formula



4. A compound of claim 1 of the formula

-SO2-NII-CH2-P(OCH2-CII=CII2) CH

Example	Plastic	Compound	Percent add-on	01	D-635
x	Polypropylene	$CH_{3} - \underbrace{\bigcirc}_{H_{3}} - SO_{2} - NH - CH_{2} - P(OCH_{3})_{2}$	30	19	NB
XI	Polystyrene	О СН3—S 02—NH—CH2—P (0 CH3)2	30	19. 7	NB
XII	ABS	CH ₃	30	20. 1	NB
XIII	Nylon	О Ш СH ₃ —SO ₂ —NH—CH ₂ —Р(ОСН ₃)2	30	23	NB
XIV XV XVI	E poxy	Same as abovedo	30 30 30		NB NB NB
XVII	Nylon	CH ₃	30	23	NB
xviii	Polypropylene	$CH_3 - SO_2 - NH - CH_2 - P(O)\phi_2$	30	22.7	NB
XIX	ABA	СH ₃ -СH ₃ -SO ₂ -NH-CH ₂ -Р(ОСH ₃) ₂	30	24, 2	NB
We claim:		50 - 5 A compound of	laim 1	of th	na form

TABLE II

1. A compound of the formula

RSO2NIICH2P (OR')2

wherein R is selected from the group consisting of 60 lower alkyl of 1-6 carbon atoms, phenyl and alkyl substituted phenyl and R' is selected from the group consisting of phenyl, lower alkenyl and halogen substituted and unsubstituted lower alkyl of 1-6 carbon atoms.

5. A compound of claim 1 of the formula

CH₂ -CH2-Cl)2

6. A compound of claim 1 of the formula

65

55