

PATENT SPECIFICATION

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(54) PREPARING FIRE RETARDANT ORGANO SILOXANE FOAM AND FOAM PREPARED THEREFROM

(71) We, DOW CORNING CORPORATION, of Midland, Michigan, United States of America, a Corporation organized under the laws of the State of Michigan, United States of America, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to fire retardant organosiloxane foams.

Siloxane foams are known to the art, however, these foams have not had an acceptance in commerce as has other types of foams, such as polyurethane foams. Such a siloxane foam is described by Leonard B. Bruner in U.S. Patent No. 3,070,555 in which he describes mixing a hydroxylated organopolysiloxane containing silicon-bonded hydrogen, a hydroxylated compound and a stannous salt of a hydrocarbon-soluble carboxylic acid and thereafter allowing the mixture to foam to an elastic product. Another siloxane foam is described by Siegfried Nitzsche et al. in U.S. Patent No. 3,338,847 in which a foam is prepared from a mixture of a linear hydroxyl endblocked diorganosiloxane polymer, an organohydrogensilicon compound, an aliphatically unsaturated organohydroxy silicon compound and a metal salt of a monocarboxylic acid. Another foam is described by Harold R. Hersh in U.S. Patent No. 3,429,838 in which a siloxane foam is prepared by mixing a silicone rubber containing a linear, fluid methylpolysiloxane having terminal silicon-bonded hydroxyl groups, diatomaceous earth, zinc oxide, calcined clay, polyethylsilicate and tin octoate with a silicone blowing agent which was a trimethylsilyl chain-stopped dimethylpolysiloxane fluid with some of the silicon-bonded methyl groups replaced by silicon-bonded hydrogen atoms and stannous octoate catalyst.

Also known to the art are fire resistant and

self-extinguishing siloxane foams. Ronald L. Joyce in U.S. Patent No. 2,956,032 describes a fire resistant organosiloxane elastomer foam of the type described by the Bruner patent containing in addition a compound of nickelous bromide, aromatic pentabromoethylbenzene or aromatic pentabromotoluene. Siegfried Nitzsche et al. teach in U.S. Patent No. 3,428,580 a self-extinguishing organopolysiloxane resin foam containing a resin organosiloxane polymer having alkoxy and hydroxy functionality, a fluid organohydrogen siloxane polymer, a compound having non-acidic reactive hydroxyl groups and a catalyst of quaternary ammonium compounds and carboxylic acid salts of heavy metals.

Although foams can be prepared as described above, there is a problem in use over broad temperature ranges such as from room temperature up to 300° C. and when fire retardant foams are made they often give off toxic vapor ingredients when heated. Thus, a more useful elastomeric siloxane foam is desirable and one which is also non-toxic particularly when it is fire retardant. It is known that platinum provides fire retardant properties to gum based silicone rubbers as described in U.S. Patent No. 3,514,424 and from U.S. Patent No. 3,652,488 that certain silicone rubbers can be made fire retardant by using a combination of platinum and carbon black. From U.S. Patent No. 3,734,881, it is known that certain room temperature silicone elastomers, namely those containing acetoxy and ketoxime functionality, can be made self-extinguishing and non-toxic by using platinum and carbon black.

From the above prior art, siloxane foams have been prepared and fire retardant siloxane foams have been prepared, however, it was not known heretofore that siloxane foams could be prepared using a platinum catalyst nor was it known that such foams would be both fire retardant and non-toxic and that increased fire retardancy would be obtained by using carbon

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black. It was unexpected that a fire retardant foam could be obtained by using a platinum catalyst in that foams act as very thin section materials compared to the usual solid material and it is known that the same material in thinner section will burn more rapidly than a thicker section, thus foams are generally more combustible than solid materials and it is therefore unexpected that a siloxane foam would be made fire retardant without the conventional fire retardant additives.

It is an object of this invention to provide a method for making a siloxane foam using platinum catalysts and a fire retardant siloxane foam which is non-toxic.

This invention relates to a method of making an organosiloxane foam by mixing an organohydrogensiloxane, a hydroxylated organosiloxane and a platinum catalyst and thereafter allowing a foam to form. These foams are particularly useful for applications of cushioning and sealing where fire retardancy is of importance.

This invention consists of a method of preparing an organosiloxane foam comprising mixing an organohydrogensiloxane having an average of at least three silicon-bonded hydrogen atoms per molecule, an average of no more than one silicon-bonded hydrogen atom per silicon atom and an organic radical selected from the group consisting of alkyl radicals having from one to six carbon atoms per radical, phenyl and 3,3,3-trifluoropropyl, a hydroxylated organosiloxane having an average of from greater than 1.0 to 2.5 silicon-bonded hydroxyl radicals per molecule and having an average of at least one organic radical per silicon atom selected from the group consisting of alkyl radicals having from one to six carbon atoms per radical, phenyl and 3,3,3-trifluoropropyl and a platinum catalyst in an amount of from 5 to 200 parts by weight platinum per one million parts by weight total composition, said organohydrogensiloxane and said hydroxylated organosiloxane being present in sufficient amounts to provide a molar ratio of silicon-bonded hydrogen atoms to silicon-bonded hydroxyl radicals of 2.5 to 40, where a mixture is obtained having a viscosity of less than 100,000 cs. at 25° C., thereafter allowing a foam to form.

The method of this invention is to mix the ingredients and obtain a foam at ambient temperature. These ingredients can be mixed in any manner. Thus, the organohydrogensiloxane can be mixed with the platinum catalyst and then mixed with the hydroxylated organosiloxane or the platinum catalyst can be mixed with the hydroxylated organosiloxane and then mixed with the organohydrogensiloxane. Other methods of mixing are also suitable such as the hydroxylated organosiloxane can be divided into two proportions where one proportion is mixed with the platinum catalyst and the other proportion is

mixed with the organohydrogensiloxane and then the two mixtures are combined to form a foam. Additionally, the various optional ingredients can be mixed with one or more of the three required ingredients in any manner as long as a premature reaction does not take place before all three ingredients are present in the mixture. For purposes of storage, the organohydrogensiloxane and the platinum catalyst should not be stored as a mixture because gasing can occur.

To control the foaming and curing reactions which are taking place simultaneously, a platinum catalyst inhibitor, such as polymethylvinylsiloxane cyclic compounds and acetylenic alcohols can be added. The platinum catalyst inhibitors are known in the art and many varieties are available. These inhibitors should however not interfere with the foaming and curing in such a manner that destroys the foam product of this invention. The mixture of ingredients should be placed in the desired place where they are to be used as soon as they are mixed because foaming begins immediately, unless a platinum catalyst inhibitor is used to extend the pot life such that they can be mixed and then put in the desired place of use. The amounts of inhibitors are present in relatively small amounts, such as up to 2 parts by weight polymethylvinylsiloxane cyclics can be used to control the initiation of the foaming and curing. The polymethylvinylsiloxane cyclics are known in the art and can be prepared by hydrolyzing methylvinylchlorosilane, for example.

This method can also be extended to include adding the mixture or organohydrogensiloxane, hydroxylated organosiloxane and platinum catalyst to other room temperature vulcanizing silicone elastomer compositions, such as those which contain a vinyl organopolysiloxane, an organohydrogensiloxane and a platinum catalyst wherein a useful foam is also obtained. The room temperature vulcanizing silicone elastomer can be mixed with the organohydrogensiloxane, or the platinum catalyst, or it can be mixed with both the organohydrogensiloxane and the platinum catalyst and then the hydroxylated organosiloxane can be added to the mixture. A convenient and preferred method is to add the organohydrogensiloxane last.

The organohydrogensiloxane can be any siloxane having an average of at least three silicon-bonded hydrogen atoms per molecule and an average of no more than one silicon-bonded hydrogen atom per silicon atom. The remaining valences of the silicon atoms are satisfied by divalent oxygen atoms and by monovalent radicals of alkyl having one to six carbon atoms per radical, such as methyl, ethyl, propyl, isopropyl, butyl, tertiary butyl, pentyl, hexyl, cyclohexyl, or phenyl or 3,3,3-trifluoropropyl. The organohydrogensiloxanes can be homopolymers, copolymers and mix-

tures thereof which contain siloxane units of the following types; $\text{RSiO}_{1.5}$, R_2SiO , $\text{R}_3\text{SiO}_{0.5}$, RHSiO , $\text{HSiO}_{1.5}$, $\text{R}_2\text{HSiO}_{0.5}$, H_2SiO , $\text{RH}_2\text{SiO}_{0.5}$ and RSiO_2 , where R is the monovalent radical defined above. Some specific examples include polymethylhydrogensiloxane cyclics, copolymers of trimethylsiloxy and methylhydrogensiloxane, copolymers of dimethylhydrogensiloxy and methylhydrogensiloxane, copolymers of trimethylsiloxy, dimethylsiloxane and methylhydrogensiloxane and copolymers of dimethylhydrogensiloxane, dimethylsiloxane and methylhydrogensiloxane. Preferably, the organohydrogensiloxanes have an average of at least five silicon-bonded hydrogen atoms per molecule.

The hydroxylated organosiloxanes can be any organosiloxane mixture of organosiloxanes having an average of from greater than 1.0 to 2.5 silicon-bonded hydroxyl radicals per molecule. The organic radicals can be any of those monovalent radicals described above for the organohydrogensiloxane. The hydroxylated organosiloxanes can be homopolymers, copolymers or mixtures thereof. They can be mixtures of different molecular weight species and varying hydroxyl contents as long as the average falls within the defined range. The hydroxylated organosiloxane contains at least one organic radical per silicon atom. Examples of the hydroxylated organosiloxane include hydroxyl endblocked polydimethylsiloxane, hydroxyl endblocked polydiorganosiloxane having siloxane units of dimethylsiloxane and phenylmethylsiloxane, hydroxyl endblocked polymethyl - 3,3,3 - trifluoropropylsiloxane and hydroxyl endblocked polyorganosiloxane having siloxane units of monomethylsiloxane, dimethylsiloxane, monophenylsiloxane and diphenylsiloxane. The hydroxylated organosiloxanes of this invention also includes mixtures of hydroxylated organosiloxane polymers and hydroxylated organosilanes, such as a mixture of hydroxyl endblocked polydimethylsiloxane and diphenylmethylsilanol. As long as some hydroxylated organosiloxane polymer having two or more hydroxyl radicals per molecule is present, hydroxylated silanes can be used to provide from greater than 1.0 to 2.5 silicon-bonded hydroxyl radicals per molecule.

Platinum catalyst can be any of the known forms, ranging from platinum as deposited on carriers such as silica gel or powdered charcoal, to platonic chloride, salts of platinum and chloroplatinic acid. Any of these forms will function in the instant curing system. A preferred form of platinum is the chloroplatinic acid either as the commonly obtainable hexahydrate or the anhydrous form, on account of its easy dispersability in organosilicon systems and its non-effect on color of the mixture. Another preferred platinum catalyst is a chloroplatinic acid catalyst complex as prepared by the method described in

U.S. Patent No. 3,419,593, where chloroplatinic acid hexahydrate is mixed with symmetrical divinyltetramethyldisiloxane to provide the complex. Another similar complex is one prepared from the chloroplatinic acid hexahydrate, symmetrical divinyltetramethyldisiloxane, symmetrical tetramethyldisiloxane and alcohol solvent. Additional platinum compounds which include,



platinum bromides, a complex of platinous halide and an olefin such as ethylene, propylene, butylene, cyclohexene and styrene, $\text{Pt}(\text{CH}_2\text{CN})_2\text{Cl}_2$, $[\text{Pt}(\text{CH}_2\text{CN})_2(\text{CH}_3)_4]\text{Cl}_2$, $\text{Pt}(\text{NH}_3)_2\text{Cl}_2$, $\text{K}[\text{PtCl}_2\text{CH}_2\text{CH}_2\text{CH}_2\text{OH}]$, $\text{PtBr}_2(\text{C}_2\text{H}_4)_2$, $\text{K}[\text{PtBr}_2(\text{C}_2\text{H}_4)]$, $\text{PtCl}_2(\text{C}_2\text{H}_4)_2$, $(\text{CH}_3)_2\text{C}=\text{CH}_2\cdot\text{PtCl}_2$, $\text{H}_2\text{Pt}(\text{CN})_4\cdot 5\text{H}_2\text{O}$, $\text{H}[\text{PtCl}_2(\text{CH}_2\text{CN})]$, $\text{Pt}(\text{NH}_3)_2(\text{CNS})_2$, $\text{PtCl}_2\text{PCl}_3$, $[\text{Pt}(\text{NH}_3)_4]\cdot[\text{PtCl}_6]$, $\text{PtCl}_2[\text{P}(\text{CH}_2\text{CH}_3)_3]_2$, $\text{PtCl}_2\cdot\text{P}(\text{OH})_3$, $\text{PtCl}_2\cdot\text{P}(\text{OCH}_2\text{CH}_3)_3$, $\text{PtCl}_2\cdot[\text{P}(\text{OCH}_2\text{CH}_3)_3]_2$, $\text{Pt}(\text{OOCH}_2\text{SCH}_2\text{CH}_3)_2$, $\text{Pt}(\text{CN})_2$, $(\text{CH}_3)_4\text{Pt}$, $(\text{CH}_3)_3\text{Pt}-\text{Pt}(\text{CH}_3)_3$, $\text{H}-\text{O}$

$(\text{CH}_3)_3\text{Pt}(\text{CH}_2\text{COCH}=\text{CCH}_3)$, PtCl_2CO and PtBr_2CO .

The platinum catalyst is present in amounts sufficient to provide from 5 to 200 parts by weight platinum per one million parts by weight total composition (p.p.m.). Preferably, the platinum catalyst is present in amounts to provide from 10 to 50 p.p.m. platinum. Amounts of platinum catalyst which provide less than 5 p.p.m. platinum are insufficient to cause a foam to form and amounts in excess of 200 p.p.m. platinum are uneconomical. For this invention the higher amounts, i.e. 50 p.p.m. to 200 p.p.m. of platinum catalyst should preferably be used with a platinum catalyst inhibitor because the rate of foaming and curing increases with increasing platinum concentration and if the rate becomes too fast, mixing the ingredients is a problem. The amount of platinum should also be sufficient to provide fire retardant properties, either alone or in combination with carbon black.

The organohydrogensiloxane and hydroxylated organosiloxane are combined in amounts sufficient enough to provide a ratio of silicon-bonded hydrogen atoms to silicon-bonded hydroxyl radicals of from 2.5 to 40, preferably from 4 to 10. Ratios of less than 2.5 provide foams, but these foams are too friable and weak to be useful in the known applications. The same problem exists at the upper limit. Within the range of the molar ratio of silicon-bonded hydrogen to silicon-bonded hydroxyl, the foam density will decrease as the ratio increases where other factors are constant.

The molecular weight of any particular ingredient is not significant except that it is compatible or dispersible in the total combination and that the total combination has a viscosity of less than 100,000 centistokes (cs.) at 25° C., preferably less than 25,000 cs. at 25° C. Above 100,000 cs., the mixing of the ingredients becomes a problem. The kind of mixing also influences the foam characteristics, such as void size and density. Some mechanical mixing is desirable to produce a foam but extreme mixing should be avoided unless it is predetermined by actual testing that it is suitable. Because there are so many variables of mixing speeds, equipment types, shearing conditions and the like, it is not practical to define meaningful conditions. Additionally any set of conditions would vary as to suitability if the nature and amounts of ingredients were changed.

Other ingredients can be present and include, platinum catalyst inhibitors, such as polymethylvinylsiloxane cyclics and acetylenic inhibitors, for example 3-methyl-1-butyne-3-ol and others as described in U.S. Patent No. 3,445,420, which shows acetylenic compounds and are used to extend the pot life before foaming occurs.

Benzene-soluble vinyl containing triorganosiloxy endblocked polydiorganosiloxanes having an average of about two vinyl radicals per molecule can also be added in amounts up to 85 weight percent based on the combined weight of the vinyl containing triorganosiloxy endblocked polydiorganosiloxane and the hydroxylated organosiloxane. These vinyl containing polydiorganosiloxanes can be mixtures or single specie types and can have organic radicals as defined for the organohydrogen-siloxane above plus vinyl. Examples of these types of polymer are widely shown in the prior art such as in U.S. Patent No. 3,445,420 and U.S. Patent No. 3,453,234, which show vinyl containing triorganosiloxy endblocked polydiorganosiloxanes. The vinyl containing triorganosiloxy endblocked polydiorganosiloxanes are preferably present in amounts of 50 weight percent or less, based on the combined weight of the vinyl containing triorganosiloxy endblocked polydiorganosiloxane and the hydroxylated organosiloxane. Although useful foams can be prepared using between 50 and 85 weight percent vinyl containing siloxane, better foams are obtained with mixtures having less than 50 weight percent vinyl containing siloxanes.

The addition of the benzene-soluble vinyl containing triorganosiloxy endblocked polydiorganosiloxanes, whether added as such or added as part of a room temperature vulcanizing silicone elastomer composition can be used to increase the strength of the foam product. When these vinyl siloxanes are present in the mixture, the molar ratio of silicon-bonded hydrogen to silicon-bonded

hydroxyl can be varied over a range of from 0.5 to 40. It is not fully understood why this ratio can be varied more broadly with these ingredients, however, the reactions are apparently complex in such mixtures and have not been fully studied.

Fillers can also be added to the present mixtures which are used in the method of making foams, such as fume silica, diatomaceous earth, zinc oxide, calcium carbonate, crushed quartz, and the like. The maximum amounts of fillers used will be dependent upon the final viscosity of the composition.

The foams of the present invention have fire retardant properties, as prepared, but for many applications it is desirable to increase this property to give added protection and broaden its areas of use. The fire retardant properties of the foams can be increased without adding ingredients which can give off toxic vapors when burned or in contact with fire. By adding, to the ingredients to be mixed to make a foam, from 0.01 to 2 parts by weight carbon black based on the total weight of the composition, preferably from 0.05 to 1 parts by weight, the fire retardant property of the foam is increased. When the carbon black is used, the foams are self-extinguishing in short times and do not give off toxic fumes nor do they contain toxic ingredients.

The method of this invention provides a fire retardant siloxane foam which is obtained at room temperature. The ingredients are mixed and then the resulting mixture foams and cures at the same time. The use of the inhibitors permits a level of control of the pot life or working time before foaming begins. The amount of platinum also permits some control of the pot life. The control of pot life is important to allow one to mix the ingredients in a container other than the place where the foam is to be used. Under some circumstances, the mixture does not immediately foam because the combination has a long pot life, as in cases where there is inhibitors present and where the molar ratio of silicon-bonded hydrogen atoms to silicon-bonded hydroxyl radicals is low and a benzene-soluble vinyl containing triorganosiloxy endblocked polydiorganosiloxane is present. Where one wishes to hasten the foam or foam on demand, mild heating will accelerate the foaming process.

The foams prepared as described herein are fire retardant and self-extinguishing and are useful for pads, cushions, pillows, ablative pads, fire retardant cushioning of electronic components, fire retardant walls, fire retardant seals, non-burning uses in areas where persons are confined such as buses, trains, airplanes, and the like.

These foams can be varied in density from 0.10 to 0.80 grams per cubic centimeter. These densities can be changed by varying the ratio of silicon-bonded hydrogen to silicon-

bonded hydroxyl as well as the filler loadings. The above densities are suggested for conventional types of fillers; however, it may be possible to obtain densities outside this range by using certain non-conventional or exotic fillers.

The following examples are presented for illustrative purposes only. In the Examples, the number of Si—H atoms/molecule, Si—H atoms/Si atom, Si—OH radicals/molecule, organic radicals/Si atom and the viscosity of the mixtures are all within the scope of claim 1.

Example 1.

A siloxane foam was prepared by hand mixing at room temperature 50 parts by weight of a phenylmethylvinylsiloxyl endblocked polydimethylsiloxane having a viscosity of 12,500 cs. at 25° C., 10 parts by weight of a trimethylsiloxyl endblocked polymethylhydrogensiloxane having about 1.5 weight percent silicon-bonded hydrogen atoms, 10 parts by weight of a hydroxyl endblocked polydimethylsiloxane having about 4 weight percent silicon-bonded hydroxyl radicals and 46 parts by weight platinum catalyst in the form of a chloroplatinic acid complex with symmetrical divinyltetramethyldisiloxane containing about 0.65 weight percent platinum. The polymethylhydrogensiloxane was the last ingredient added to the mixture. The resulting mixture had a molar ratio of silicon-bonded hydrogen atoms (SiH) to silicon-bonded hydroxyl radicals (SiOH) of about 6.4 and began foaming almost before the mixing was completed. The resulting product was a cured, dry elastomeric foam.

The above procedure was followed except one part by weight of polymethylvinylsiloxane cyclics was added to the polymethylhydrogensiloxane which was then added to the other ingredients. The foaming and curing reactions were retarded for approximately 10 minutes after which time the mixture foamed and cured rapidly to a dry, elastomeric foam similar to the foam obtained without the polymethylvinylsiloxane cyclics.

Example 2.

A siloxane foam was prepared by hand mixing 50 parts by weight of a phenylmethylvinylsiloxyl endblocked polydimethylsiloxane having a viscosity of 12,500 cs. at 25° C., 6 parts by weight of a phenylmethylvinylsiloxyl endblocked polydimethylsiloxane having a viscosity of about 2,000 cs. at 25° C., 10 parts by weight hydroxyl endblocked polydimethylsiloxane having about 4 weight percent SiOH, 10 parts by weight of trimethylsiloxyl endblocked polymethylhydrogensiloxane having about 1.5 weight percent SiH, 1 part by weight of zinc oxide, 0.5 part by weight carbon black, 0.5 part by weight polymethylvinylsiloxane cyclics and 41 p.p.m. platinum

in the form defined in Example 1. A mixture of the polymethylhydrogensiloxane and the polymethylvinylsiloxane cyclics was the last ingredient added to the mixture. The resulting product was a cured, elastomeric foam which did not burn when an attempt was made to ignite it. The foams of Example 1 in comparison could be ignited.

Example 3.

Siloxane foams were prepared by mixing at room temperature the ingredients described in Tables 1 and 2. The combination of the ingredients of this example began to foam and cure from 25 to 60 seconds after the polymethylhydrogensiloxane was added. The ingredients were all combined in each case except a mixture of the polymethylhydrogensiloxane and the polymethylvinylsiloxane cyclics was added last. In Tables 1 and 2, "Polymer A" refers to a hydroxyl endblocked polydimethylsiloxane having a viscosity of 12,500 cs. at 25° C., "Polymer B" refers to a hydroxyl endblocked polydimethylsiloxane having about 4 weight percent SiOH, "Polymer C" refers to a phenylmethylvinylsiloxyl endblocked polydimethylsiloxane having a viscosity of about 400 cs. at 25° C., "Polymer D" refers to a trimethylsiloxyl endblocked polymethylhydrogensiloxane having about 1.5 weight percent SiH and "Cyclics" refers to polymethylvinylsiloxane cyclics. The p.p.m. of platinum was added in the form as described in Example 1. The wt. % in Tables 1 and 2 are weight percentages based on the total weight of the composition. Elastomeric siloxane foams were obtained in all combinations defined in Tables 1 and 2. The foam prepared with the ingredients defined in Table 2, Ref. No. 1 did not rise as much as did the foam obtained with the ingredients defined in Table 2, Ref. No. 2. The foams obtained as defined by Table 2, Ref. No. 3 and Ref. No. 4 had densities of 0.32 grams per cubic centimeter and 0.67 grams per cubic centimeter, respectively. Foams were also made using calcium carbonate. The foams obtained in accordance with both Table 1 and Table 2 were self-extinguishing. The fire retardancy was determined in accordance with the procedure set forth by Underwriters' Laboratories, Inc. "The Standard For Tests For Flammability Of Plastic Materials For Parts In Devices And Appliances, UL 94, First Edition, Second Impression, Dated June 1, 1973" SE-1 Foam classification. The results for the foam obtained with ingredients defined by Table 1, Ref. No. 1, after a 60 second flame exposure, the test specimen extinguished in less than 2 seconds and had a burn distance of less than 1.27 cm. (0.5 in.).

The ingredients as defined by Table 1, Ref. No. 1 were combined as described above except the polymethylvinylsiloxane cyclics were replaced by 50 p.p.m. 3-methyl-1-

butyne-3-ol. The foaming and curing began approximately 120 seconds after the polymethylhydrogensiloxane was added.

- 5 Several samples of the foam product prepared from the ingredients defined by Table 1, Ref. No. 1 were tested and found to have a density of 0.16 to 0.27 grams per cubic centimeter, an average of 30 pores per linear inch, 85 to 95 percent closed cells, a tensile strength of about 6 p.s.i. and a compression deflection of about 1.5 p.s.i. at 25 percent compression where both tensile strength and compression deflection were determined by ASTM—D1564—59T and the acoustics were determined by ASTM—C423 where the noise reduction coefficient was 47.0.

Example 4.

- 20 (A) A mixture was prepared by blending 11.4 parts by weight of Polymer C as defined in Example 3, 11.4 parts by weight of five micron quartz, 1.42 parts by weight of a phenylmethylvinylsiloxy endblocked polydimethylsiloxane having a viscosity of about 2000 cs. at 25° C., 0.24 parts by weight zinc oxide, 0.12 parts by weight carbon black and a platinum catalyst as defined in Example 1 in an amount sufficient to provide 130 p.p.m. platinum.

- 30 (B) A mixture was prepared by blending 11.6 parts by weight of Polymer C, 11.6 parts by weight of five micron quartz, 1.3 parts by weight of a trimethylsiloxy endblocked polyorganosiloxane having an average of 5 methylhydrogensiloxane units and 3 dimethylsiloxane units and 0.5 part by weight of polymethylvinylsiloxane cyclics.

- 40 (C) A blend of 25 parts by weight of mixture (A) and 25 parts by weight of mixture (B), 10 parts by weight of Polymer B as defined in Example 3, 70 parts by weight of diphenylmethylsilanol and 10 parts by weight

of Polymer D as defined in Example 3 was prepared by mixing at room temperature which cured to an elastomeric foam with an increase in volume compared to the unfoamed mixture of about 50 percent. This foam had fire retardant properties and had greater than 90 percent closed cells. The blend of (C) had a molar ratio of SiH to SiOH of about 0.5, had 23 p.p.m. platinum and about 0.08 weight percent carbon black.

(D) A blend of 24 parts by weight of mixture (A) and 25 parts by weight of mixture (B), 5 parts by weight of diphenylmethylsilanol, 5 parts by weight of Polymer B and 30 parts by weight of Polymer D was prepared by mixing at room temperature which cured to an elastomeric foam with an expansion of about 100 percent. This foam had fire retardant properties, had a density of about 0.13 grams per cubic centimeter and had greater than 90 percent closed cells. The blend of (D) had a molar ratio of SiH to SiOH of about 13, had 36 p.p.m. platinum and about 0.13 weight percent carbon black.

Example 5.

A siloxane foam was prepared by mixing at room temperature 25 parts by weight of a hydroxyl endblocked polydimethylsiloxane having a viscosity of about 4000 cs. at 25° C., 25 parts by weight calcium carbonate, 10 parts by weight of Polymer B, 10 parts by weight of diphenylmethylsilanol, 32 p.p.m. platinum in the form of the platinum catalyst as defined in Example 1 and thereafter mixing therewith a mixture of 30 parts by weight of Polymer D and 0.5 parts by weight of polymethylvinylsiloxane cyclics. The resulting mixture had a molar ratio of SiH to SiOH of about 6. An elastomeric foam which had fire retardant properties, was obtained.

TABLE I

Ref. No.	1	2	3	4	5
Polymer A, wt. %	78.5	80.7	82.2	83.8	86.8
Polymer B, wt. %	8.7	9.0	9.1	9.3	—
Polymer C, wt. %	2.8	2.9	2.9	3.0	3.1
Polymer D, wt. %	9.0	6.5	4.7	2.9	9.0
Cyclics, wt. %	0.045	0.033	0.024	0.015	0.045
Platinum, p.p.m.	14	15	15	15	16
Zinc Oxide, wt. %	0.46	0.48	0.49	0.50	0.51
Carbon Black, wt. %	0.24	0.24	0.25	0.25	0.26
Molar Ratio SiH to SiOH	5.5	3.9	2.8	1.7	30.0

TABLE 2

Ref. No.	1	2	3	4
Polymer A, wt. %	39.25	52.3	71.6	56.7
Polymer B, wt. %	8.7	8.7	8.0	6.3
Polymer C, wt. %	42.0	20.0	2.5	2.0
Polymer D, wt. %	9.0	9.0	9.0	9.0
Cyclics, wt. %	0.045	0.045	0.045	0.045
Platinum, p.p.m.	14	14	13	10
Zinc Oxide, wt. %	0.46	0.46	0.42	0.34
Carbon Black, wt. %	0.24	0.24	0.21	0.17
Quartz (5 μ) wt. %	—	—	8.0	25.2
Molar Ratio SiH to SiOH	6.0	5.9	6.0	7.6

WHAT WE CLAIM IS:—

- 5 1. A method of preparing a fire retardant organosiloxane foam which comprises mixing at ambient temperature

- 10 (1) an organohydrogensiloxane having an average of at least three silicon-bonded hydrogen atoms per molecule, an average of no more than one silicon-bonded hydrogen atom per silicon atom and an organic radical selected from alkyl radicals having from one to six carbon atoms per radical, phenyl and 3,3,3-trifluoropropyl radicals;
- 15 (2) a hydroxylated organosiloxane having an average of from greater than 1.0 to 2.5

- silicon-bonded hydroxyl radicals per molecule and having an average of at least one organic radical per silicon atom selected from an alkyl radical having from one to six carbon atoms per radical, a phenyl and 3,3,3-trifluoropropyl radical;
- 20 (3) a platinum or platinum compound catalyst to foam and cure the organosiloxane in an amount of from 5 to 200 parts by weight platinum per one million parts by weight total composition; and optionally
- 25 (4) a benzene-soluble vinyl containing triorganosiloxy terminated polydiorganosiloxane having an average of two vinyl
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TABLE I

Ref. No.	1	2	3	4	5
Polymer A, wt. %	78.5	80.7	82.2	83.8	86.8
Polymer B, wt. %	8.7	9.0	9.1	9.3	—
Polymer C, wt. %	2.8	2.9	2.9	3.0	3.1
Polymer D, wt. %	9.0	6.5	4.7	2.9	9.0
Cyclics, wt. %	0.045	0.033	0.024	0.015	0.045
Platinum, p.p.m.	14	15	15	15	16
Zinc Oxide, wt. %	0.46	0.48	0.49	0.50	0.51
Carbon Black, wt. %	0.24	0.24	0.25	0.25	0.26
Molar Ratio SiH to SiOH	5.5	3.9	2.8	1.7	30.0

TABLE 2

Ref. No.	1	2	3	4
Polymer A, wt. %	39.25	52.3	71.6	56.7
Polymer B, wt. %	8.7	8.7	8.0	6.3
Polymer C, wt. %	42.0	20.0	2.5	2.0
Polymer D, wt. %	9.0	9.0	9.0	9.0
Cyclics, wt. %	0.045	0.045	0.045	0.045
Platinum, p.p.m.	14	14	13	10
Zinc Oxide, wt. %	0.46	0.46	0.42	0.34
Carbon Black, wt. %	0.24	0.24	0.21	0.17
Quartz (5 μ) wt. %	—	—	8.0	25.2
Molar Ratio SiH to SiOH	6.0	5.9	6.0	7.6

WHAT WE CLAIM IS:—

1. A method of preparing a fire retardant organosiloxane foam which comprises mixing at ambient temperature

(1) an organohydrogensiloxane having an average of at least three silicon-bonded hydrogen atoms per molecule, an average of no more than one silicon-bonded hydrogen atom per silicon atom and an organic radical selected from alkyl radicals having from one to six carbon atoms per radical, phenyl and 3,3,3-trifluoropropyl radicals;

(2) a hydroxylated organosiloxane having an average of from greater than 1.0 to 2.5

silicon-bonded hydroxyl radicals per molecule and having an average of at least one organic radical per silicon atom selected from an alkyl radical having from one to six carbon atoms per radical, a phenyl and 3,3,3-trifluoropropyl radical;

(3) a platinum or platinum compound catalyst to foam and cure the organosiloxane in an amount of from 5 to 200 parts by weight platinum per one million parts by weight total composition; and optionally (4) a benzene-soluble vinyl containing triorganosiloxy terminated polydiorganosiloxane having an average of two vinyl

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5 radicals per molecule and present in an
amount equal to no more than 85 percent
by weight of the combined weight of the
hydroxylated organosiloxane and the
benzene soluble vinyl containing triorgano-
siloxyl terminated polydiorganosiloxane, the
remaining organic radicals of the benzene-
soluble vinyl containing triorganosiloxyl
terminated polydiorganosiloxane being alkyl
radicals having from one to six carbon atoms
per radical phenyl, vinyl or 3,3,3-trifluoro-
propyl radicals; the organohydrogensiloxane
and the hydroxylated organosiloxane being
present in amounts sufficient to provide a
molar ratio of silicon-bonded hydrogen
atoms to silicon-bonded hydroxyl radicals
of 2.5 to 40 when components (1) to (3)
are mixed and a ratio of 0.5 to 40 when
components (1) to (4) are mixed, the mix-
ture obtained having a viscosity of less than
100,000 cs. at 25° C., and thereafter
allowing a foam to form.

2. A method as claimed in Claim 1 wherein
the hydroxylated organosiloxane has an average
of from 1.5 to 2.5 silicon-bonded hydroxyl
radicals per molecule, and the organohydrogen-
siloxane and hydroxylated organosiloxane are
present in amounts sufficient to provide a
molar ratio of silicon-bonded hydrogen atoms
to silicon-bonded hydroxyl radicals of 0.5 to
40.

3. A method as claimed in Claim 1 sub-
stantially as described with reference to any
of the Examples.

4. An organosiloxane foam whenever pro-
duced by a method as claimed in any of the
preceding Claims.

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