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(21) International Application Number: PCT/US95/12702 (22) International Filing Date: 12 October 1995 (12.10.95) (30) Priority Data: 08/322,879 13 October 1994 (13.10.94) US (71) Applicant: AKZO NOBEL NV [NL/NL]; Velperweg 76, P.O. Box 9300, NL-6800 SB Arnhem (NL). (71)(72) Applicants and Inventors: BRIGHT, Danielle, A. [US/US]; 21 Zabela Drive, New City, NY 10956 (US). AARONSON, Alan, M. [US/US]; 65-48 173 Street, Fresh Meadows, NY 11365-2033 (US). PIRRELLI, Ronald, L. [US/US]; 61 Oak Ridge Circle, Mahopac, NY 10541 (US). (74) Agent: FENNELLY, Richard, P.; Akzo Nobel Inc., 7 Livingstone Avenue, Dobbs Ferry, NY 10522 (US).		(81) Designated States: CA, JP, European patent (AT, BE, CH, DE, DK, ES, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE). Published <i>With international search report.</i>

(54) Title: VISCOSITY MODIFICATION OF HIGH VISCOSITY FLAME RETARDANTS

(57) Abstract

Alkylene-bridged diphosphate compounds can be used to modify, namely reduce, the viscosity of fluid flame retardants (polybrominated aryl oxides, oligomeric phosphate esters, etc.) which are useful in flame retarding polyurethane and thermoplastic compositions.

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VISCOSITY MODIFICATION OF HIGH
VISCOSITY FLAME RETARDANTS

BACKGROUND OF THE INVENTION

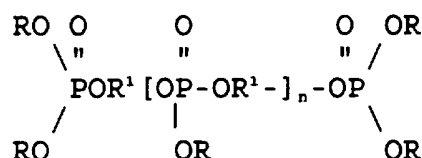
Polyurethane foams, coatings and elastomers are commonly
5 admixed with flame retardants to achieve the desired degree of
flame retardancy for the final material desired. Pumpable fluid
(or liquid) flame retardant compositions are a preferred class
since such pumpable formulations are needed in environments where
automation and machine mixing of the various components are used.
0 Examples of the chemical classes for such fluid or liquid flame
retardants include brominated aryl flame retardants, such as
polybromodiphenyl oxide, and various viscous organophosphorus flame
retardants such as the oligomeric phosphate esters, such as the
chlorinated oligomeric phosphate esters (e.g. FYROL 99 brand) and
5 the reaction product of 2-chloro-1-propanol phosphate (3:1) with
ethylene oxide and phosphorus pentoxide (FYROL PCF brand), and the
aromatic oligomeric phosphate esters such as those containing an
arylene bridging group derived from a diol such as bisphenol A,
hydroquinone or resorcinol.

0 High viscosity in such fluid or liquid flame retardant
compositions can be disadvantageous since it makes the pumping and
movement of such compositions more difficult thereby complicating
manufacturing operations. It can also hinder the flow of
thermoplastic polymers containing such a viscous flame retardant.
5 For example, it is known that polybrominated diphenyl oxide flame
retardants are very viscous. U.S. Patent No. 4,746,682 to J. Green
indicates that either alkyl diphenyl phosphates or alkylated
triaryl phosphates can be used to achieve blends having acceptably
low viscosity as compared to the polybrominated aryl flame
0 retardant itself. Copending U.S. Serial No. 215,406, filed March
14, 1994, describes the use of triphenyl phosphate as a viscosity
reduction additive for viscous flame retardants. However, the
relatively low volatility of the triaryl phosphates makes them
unsuitable for certain applications where high processing
5 temperatures may cause juicing.

SUMMARY OF THE INVENTION

It has now been found that an alkylene-bridged diphosphate compound is an effective viscosity modifying, namely, reducing agent in the aforementioned types of liquid, but viscous, flame retardant compositions. This type of material is less volatile than a triaryl phosphate compound. It also has a higher phosphorus content resulting in an increased flame retardancy efficacy as compared to a triaryl phosphate compound.

The terminology "alkylene-bridged diphosphate compound" is to be understood to encompass monomeric and low oligomeric species of the formula



Where R is aryl, such as unsubstituted phenyl, n is a number ranging from 0 to about 5, and R¹ is alkylene of from 1 to 8 carbon atoms.

The level of use of the diphosphate viscosity modification additive of the present invention with a particular flame retardant will depend upon its initial compatibility or miscibility with the flame retardant component whose viscosity is initially high and in need of reduction. The level of diphosphate to use is also dependent upon the ultimate viscosity that is desired for the composition containing it. Generally, the amount of diphosphate that needs to be added will range from about 5% to about 80%, by weight of the fluid or liquid flame retardant whose viscosity is to be reduced, preferably from about 5% to about 50%, more preferably from about 5% to about 30%.

The liquid flame retardants to which the instant invention can be added include polybrominated diphenyl oxide and aromatic oligomeric phosphates (e.g., resorcinol bis(diphenyl phosphate), bisphenol A bis(diphenyl phosphate), poly(resorcinol phenylphosphate), and the like.

5 The aromatic bisphosphates to which the above-described
alkylene-bridged diphosphate species can be added are of the same
formula given above with the exception that R¹ is derived from an
arylene diol, such as resorcinol, bisphenol A, or hydroquinone, and
n is generally from 0 to about 15. The diphosphate viscosity
reducing additive of the instant invention can also be used in
similar amount with flame retardant compositions which contain
oligomeric phosphate esters as the sole or predominant component.
Generally speaking, it has been found that up to about 50%, by
0 weight of the alkylene-bridged diphosphate compound, based on the
weight of the entire composition, can be used in such systems.

5 The instant invention allows for the obtaining of low
enough viscosities to achieve pumpable flame retardant compositions
which are required to satisfactorily process flexible and rigid
polyurethane foams, for example. Easier machine mixing and
miscibility are achieved by bringing the viscosities of the
components closer and preferably lower. Coatings and elastomers
also require low viscosities for better flowability and processing.

0 The present invention is illustrated by the Examples which
follow.

EXAMPLES 1-19

A series of compositions were tested in regard to their viscosity at 23°C ± 0.5°C. in a Brookfield viscometer. The Table given below shows the results which were obtained.

5 (The viscosity of the additives responsible for viscosity reduction were as follows: neopentyl glycol bis(diphenyl-phosphate) - 549; ethylene glycol bis(diphenylphosphate) - 319; and propylene glycol bis(diphenylphosphate) - 367.5.

10

TABLE

<u>Composition - Amount (wt %)</u>		<u>Viscosity (cps)</u>
	Bisphenol A bis(diphenyl phosphate) 100%	20,900
	Bisphenol A bis(diphenylphosphate) 90%	12,000
	Neopentyl glycol bis(diphenylphosphate) 10%	
15	Bisphenol A bis(diphenylphosphate) 80%	8,292
	Neopentyl glycol bis(diphenylphosphate) 20%	
	Bisphenol A bis(diphenylphosphate) 50%	2,616
	Neopentyl glycol bis(diphenylphosphate) 50%	
20	Bisphenol A bis(diphenylphosphate) 90%	11,700
	Ethylene glycol bis(diphenylphosphate) 10%	
	Bisphenol A bis(diphenylphosphate) 80%	6,548
	Ethylene glycol bis(diphenylphosphate) 20%	
	Bisphenol A bis(diphenylphosphate) 50%	1,752
	Ethylene glycol bis(diphenylphosphate) 50%	
25	Bisphenol A bis(diphenylphosphate) 80%	7,380
	Propylene glycol bis(diphenylphosphate) 20%	
	Bisphenol A bis(diphenylphosphate) 50%	2,052
	Propylene glycol bis(diphenylphosphate) 50%	
	Resorcinol bis(diphenyl phosphate) 100%	691

	Resorcinol A bis(diphenylphosphate)	90%	
	Neopentyl glycol bis(diphenylphosphate)	10%	670.5
	Resorcinol A bis(diphenylphosphate)	90%	
	Ethylene glycol bis(diphenylphosphate)	10%	636
5	Resorcinol A bis(diphenylphosphate)	90%	
	Propylene glycol bis(diphenylphosphate)	10%	666
	Pentabromo Diphenyloxide	70%	
	Neopentyl glycol bis(diphenylphosphate)	30%	8,670
	Pentabromo Diphenyloxide	50%	
0	Neopentyl glycol bis(diphenylphosphate)	50%	2,090
	Pentabromo Diphenyloxide	70%	
	Ethylene glycol bis(diphenylphosphate)	30%	5,090
	Pentabromo Diphenyloxide	50%	
	Ethylene glycol bis(diphenylphosphate)	50%	1,225
5	Pentabromo Diphenyloxide	70%	
	Propylene glycol bis(diphenylphosphate)	30%	5,590
	Pentabromo Diphenyloxide	50%	
	Propylene glycol bis(diphenylphosphate)	50%	1,395

0 The foregoing data is presented for purposes of illustrating certain embodiments of the present invention and, for that reason, should not be construed in a limiting sense. The scope of protection sought is set forth in the claims which follow.

We Claim:

1. A fluid flame retardant composition suitable for use in forming flame retarded polymer composition which comprises a normally viscous flame retardant and an effective amount of an alkylene-bridged diphosphate compound for viscosity modification thereof.

2. A composition as claimed in Claim 1 wherein the flame retardant composition comprises a major amount of a polybrominated diphenyl oxide.

3. A composition as claimed in Claim 2 wherein the flame retardant is pentabromodiphenyl oxide.

4. A composition as claimed in Claim 1 wherein the flame retardant composition comprises a major amount of an aromatic oligomeric phosphate ester flame retardant.

5. A composition as claimed in Claim 1 wherein flame retardant composition comprises a major amount of a high molecular weight chloroalkyl phosphate.

6. A composition as claimed in Claim 1 wherein the alkylene-bridged diphosphate is present at up to about 50%, by weight of the entire composition.

7. A composition as claimed in Claim 2 wherein the alkylene-bridged diphosphate is present at up to about 50%, by weight of the entire composition.

8. A composition as claimed in Claim 4 wherein the alkylene-bridged diphosphate compound is present at up to about 50%, by weight of the entire composition.

9. A composition as claimed in Claim 5 wherein the alkylene-bridged diphosphate compound is present at up to about 50%, by weight of the entire composition.

INTERNATIONAL SEARCH REPORT

International application No.

PCT/US95/12702

A. CLASSIFICATION OF SUBJECT MATTER

IPC(6) : C09K 21/08, 21/12

US CL : 252/609

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

U.S. : 252/609, 601, 602, 603, 604, 605, 606, 607, 608; 521/107; 558/164

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APS, STN/CAS**C. DOCUMENTS CONSIDERED TO BE RELEVANT**

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US, A, 4,433,071 (FESMAN) 21 February 1984, see abstract, and column 1, line 64 to column 3, line 17.	1-9
Y	US, A, 4,565,833 (BUSZARD ET AL.) 21 January 1986, see abstract, and column 2, lines 52-68.	1-9
Y	US, A, 4,681,902 (DUNCAN ET AL.) 21 July 1987, see abstract, column 1, line 48 to column 2, line 34, and Example 1.	1-9
Y	JP, A, 49-40342 (ASAHI DENKA KOGYO K.K.) 15 April 1974, see English translation, page 1 and page 8, lines 7-11.	1-9
X	US, A, 5,086,082 (STONE) 04 February 1992, see abstract, and column 3, lines 20-30.	1-3, 6-7



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