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(54) Flame retardant polyurethane foams resistant to cigarette smoldering ignition.

(57) The invention pertains to flame retardant flexible polyurethane foams resistant to cigarette smoldering after being subjected to flex fatigue prepared by reacting a polyether polyol, an organic isocyanate, and a blowing agent incorporating melamine in an amount ranging from about 5 weight percent to about 25 weight percent.

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FLAME RETARDANT POLYURETHANE FOAMS RESISTANT TO CIGARETTE SMOLDERING IGNITION

The present invention pertains to flexible foam compositions and in particular to flexible polyurethane flame-retarded foam compositions and methods for the preparation thereof. More particularly, the present invention relates to the preparation of flexible polyurethane flame-retarded foam compositions which will retain their resistance to cigarette smoldering even after service.

The preparation of flexible polyurethane flame-retardant foam compositions are generally well known as evidenced by the following prior art. U.S. 4,022,718 teaches the preparation of high resilience cold-cured polyurethane foams incorporating 2,3-dibromo-1,4-butenediol as a chain extender and flame-retardant component. U.S. 4,147,847 teaches a method of preparing flexible, flame-retarded, polyurethane foams by employing specific foam stabilizers which reduce the required amount of normal flame-retardant additives. U.S. 4,162,353 teaches the preparation of flexible polyurethane foams incorporating therein a halo-substituted alkyl phosphate such as, for example, tris(2-chlorethyl)-phosphate and an unsubstituted trialkyl-phosphate such as, for example, triethylphosphate. U.S. Patent 4,221,875 teaches the preparation of rigid polyurethane foam incorporating melamine as the sole flame retardant component. U.S. Patent 4,385,131 teaches the preparation of rebonded polyurethane foam incorporating urea and/or melamine for resistance to smoldering combustion. None of the prior art teaches, however, that incorporating an amount of melamine from about 5 weight percent to about 25 weight percent of the weight of the flexible foam, having a density from about 1.2 lbs/ft^3 to about 4 lbs/ft^3 will result in a foam resistant to cigarette smoldering upon being subjected to flex fatigue similar to actual servive.

The present invention applies to both high-resiliency, flexible polyurethane foam compositions and conventional flexible polyurethane foam compositions which retains its cigarette smoldering resistance after service consisting essentially of (a) a polyoxyalkylene polyether polyol, (b) an organic polyisocyanate, (c) a blowing agent, (d) a catalyst, (e) a surfactant, (f) melamine, and (g) optionally chain extenders and flame retardants other than melamine, wherein the concentration of melamine is from about 5 weight percent to about 25 weight percent of the weight of the foam and wherein the density of the foam is from at least 1.2 lbs/ft^3 to about 4 lbs/ft^3.

For a more complete understanding of the present invention, reference is made to the following detailed description and the examples thereof.

Figure I illustrates the effect of Roller shear fatigue on cigarette smoldering resistance of polyurethane foam in the absence of melamine at densities of 1.0, 1.4, and 2.1 pcf.

Figure II illustrates the effect of Roller shear fatigue on cigarette smoldering resistance of polyurethane foam in the absence and presence of 20 pbw melamine at a density of 1.6 pcf.

Figure III illustrates the effect of Roller shear fatigue on cigarette smoldering resistance of polyurethane foam in the absence and presence of 20 pbw melamine at a density of 2.3 pcf.

Figure IV illustrates the effect of Roller shear fatigue on cigarette smoldering resistance of polyurethane foam in the absence and presence of 20 pbw melamine at a density of 2.5 pcf.

It has unexpectedly been found that, in the preparation of flexible flame-retarded polyurethane foam, a mixture of (a) melamine incorporated into the polyoxyalkylene polyether polyol will result in foam which will retain its cigarette smoldering resistance even after being subjected to flex fatigue similar to actual service. It has been found that melamine in the amount ranging from about 5 weight percent to about 25 weight percent of the weight of the foam will enable polyurethane foam to be prepared which will retain its resistance to cigarette smoldering.

Flexible foams are generally defined as having a high tensile to compressive strength ratio (25% deflection) from 15 to 60 or 70 to 1, high elongation, a fast recovery rate and a high elastic limit. Rigid foams on the other hand have a high ratio of compressive to tensile strength, 0.5 to 1 or greater low elongation (less than 10%), a low recovery rate from distortion and a low elastic limit.

Representative polyols which may be employed in the preparation of the flexible flame retardant polyurethane foams are well known to those skilled in the art. They are often prepared by the catalytic condensation of an alkylene oxide or mixture of alkylene oxides either simultaneously or sequentially with an organic compound having at least two active hydrogen atoms, such as evidenced by U.S. Patent Nos. 1,922,459; 3,190,927; and 3,346,557. Representative polyols include polyhydroxyl-containing polyesters, polyoxyalkylene polyether polyols, polyhydroxy-terminated polyurethane polymers, polyhydroxyl-containing phosphorus compounds, and alkylene oxide adducts of polyhydric polythioesters, polyacetals, aliphatic polyols and thiols, ammonia, and amines including aromatic, aliphatic, and heterocyclic amines, as well as mixtures thereof. Alkylene oxide adducts of compounds which contain 2 or more different groups within the above-defined classes may also be used, for example, amino alcohols which contain an amino group and a

hydroxyl group. Also, alkylene oxide adducts of compounds which contain one SH group and one OH group as well as those which contain an amino group and an SH group may be used. Generally, the polyols will have an equivalent weight from 1000 to 10,000, preferably from 1500 to 3000 and a functionality of two to four.

Any suitable hydroxy-terminated polyester may be used such as are prepared, for example, from polycarboxylic acids and polyhydric alcohols. Any suitable polycarboxylic acid may be used such as oxalic acid, malonic acid, succinic acid, glutaric acid, adipic acid, pimelic acid, suberic acid, azelaic acid, sebacic acid, brassylic acid, thapsic acid, maleic acid, fumaric acid, glutaconic acid, α-hydromuconic acid, β-hydromuconic acid, α-butyl-α-ethyl-glutaric acid, α,β-diethylsuccinic acid, isophthalic acid, terephthalic acid, hemimellitic acid, and 1,4-cyclohexanedicarboxylic acid. Any suitable polyhydric alcohol, including both aliphatic and aromatic, may be used such as ethylene glycol, propylene glycol, trimethylene glycol, 1,2-butanediol, 1,3-butanediol, 1,4-butanediol, 1,2-pentanediol, 1,4-pentanediol, 1,5-pentanediol, 1,6-hexanediol, 1,7-heptanediol, glycerol, 1,1,1-trimethylolpropane, 1,1,1-trimethylolethane, 1,2,6-hexanetriol, α-methyl glucoside, pentaerythritol, and sorbitol. Also included within the term "polyhydric alcohol" are compounds derived from phenol such as 2,2-bis(4-hydroxyphenyl)propane, commonly known as Bisphenol A.

The hydroxyl-containing polyester may also be a polyester amide such as is obtained by including some amine or amino alcohol in the reactants for the preparation of the polyesters. Thus, polyester amides may be obtained by condensing an amino alcohol such as ethanolamine with the polycarboxylic acids set forth above or they may be made using the same components that make up the hydroxyl-containing polyester with only a portion of the components being a diamine such as ethylene diamine.

Any suitable polyoxyalkylene polyether polyol may be used such as the polymerization product of an alkylene oxide or a mixture of alkylene oxides with a polyhydric alcohol. Any suitable polyhydric alcohol may be used such as those disclosed above for use in the preparation of the hydroxy-terminated polyesters. Any suitable alkylene oxide may be used such as ethylene oxide, propylene oxide, butylene oxide, amylene oxide, and mixtures of these oxides. The polyoxyalkylene polyether polyols may be prepared from other starting materials such as tetrahydrofuran and alkylene oxide-tetrahydrofuran mixtures; epihalohydrins such as epichlorohydrin; as well as aralkylene oxides such as styrene oxide. The polyoxyalkylene polyether polyols may have either primary or secondary hydroxyl groups. Included among the polyether polyols are polyoxyethylene glycol, polyoxypropylene glycol, polyoxybutylene glycol, polytetramethylene glycol, block copolymers, for example, combinations of polyoxypropylene and polyoxyethylene glycols, poly-1,2-oxybutylene and polyoxyethylene glycols, poly-1,4-oxybutylene and polyoxyethylene glycols, and random copolymer glycols prepared from blends of two or more alkylene oxides or by the sequential addition of two or more alkylene oxides. The polyoxyalkylene polyether polyols may be prepared by any known process such as, for example, the process disclosed by Wurtz in 1859 and Encyclopedia of Chemical Technology, Vol. 7, pp. 257-262, published by Interscience Publishers, Inc. (1951) or in U.S. Patent No. 1,922,459. Polyethers which are preferred include the alkylene oxide addition products of trimethylolpropane, glycerine, pentaerythritol, sucrose, sorbitol, propylene glycol, and 2,2′-(4,4′-hydroxyphenyl)propane and blends thereof having equivalent weights of from 1000 to 10,000.

Suitable polyhydric polythioethers which may be condensed with alkylene oxides include the condensation product of thiodiglycol or the reaction product of a dicarboxylic acid such as is disclosed above for the preparation of the hydroxyl-containing polyesters with any other suitable thioether glycol.

Polyhydroxyl-containing phosphorus compounds which may be used include those compounds disclosed in U.S. Patent No. 3,639,542. Preferred polyhydroxyl-containing phosphorus compounds are prepared from alkylene oxides and acids of phosphorus having a P_2O_5 equivalency of from about 72 percent to about 95 percent.

Suitable polyacetals which may be condensed with alkylene oxides include the reaction product of formaldehyde or other suitable aldehyde with a dihydric alcohol or an alkylene oxide such as those disclosed above.

Suitable aliphatic thiols which may be condensed with alkylene oxides include alkanethiols containing one or two -SH groups such as 2-mercaptoethanol, 1,2-ethanedithiol, 1,2-propanedithiol, 1,3-propanedithiol, and 1,6-hexanedithiol; alkene thiols such as 2-butene-1,4-dithiol; and alkyne thiols such as 3-hexyne-1,6-dithiol.

Suitable amines which may be condensed with alkylene oxides include aromatic amines such as aniline, o-chloroaniline, p-aminoaniline, 1,5-diaminonaphthalene, methylene dianiline, the condensation products of aniline and formaldehyde, and 2,3- 2,6-, 3,4-, 2,5-, and 2,4-diaminotoluene; aliphatic amines such as methylamine, triisopropanolamine, ethylenediamine, 1,3-diaminopropane, 1,3-diaminobutane, 1,4-diaminobutane, and ammonia.

Also, polyols containing ester groups can be employed in the subject invention. These polyols are

prepared by the reaction of an alkylene oxide with an organic dicarboxylic acid anhydride and a compound containing reactive hydrogen atoms. A more comprehensive dis cussion of these polyols and their method of preparation can be found in U.S. Patents Nos. 3,585,185; 3,639,541 and 3,639,542.

Other polyols which may be employed have incorporated therein vinylic polymers. These polyols may be prepared (1) by the in situ free-radical polymerization of an ethylenically unsaturated monomer or mixture of monomers in a polyol, or (2) by dispersion in a polyol of a preformed graft polymer prepared by free-radical polymerization in a solvent such as described in U.S. Patents 3,931,092; 4,014,846; 4,093,573; and 4,122,056, the disclosures of which are herein incorporated by reference, or (3) by low temperature polymerization in the presence of chain transfer agents. These polymerizations may be carried out at a temperature between 65° C and 170° C, preferably between 75° C and 135° C.

The polyols which may be employed in the preparation of the graft polymer dispersions are well known in the art. Both conventional polyols essentially free from ethylenic unsaturation such as those described in U.S. Reissue Patent No. 28,715 and unsaturated polyols such as those described in U.S. Patent No. 3,652,659 and Reissue 29,014 may be employed in preparing the graft polymer dispersions used in the instant invention, the disclosures of which are incorporated by reference. Mixtures of graft polymer dispersions and conventional polyols may be employed.

The polyurethane foams employed in the present invention are generally prepared by the reaction of a polyoxyalkylene polyether polyol with an organic polyisocyanate in the presence of a blowing agent and optionally in the presence of additional polyhydroxyl-containing components, chain-extending agents, catalysts, surface-active agents, stabilizers, dyes, fillers and pigments. Suitable processes for the preparation of cellular polyurethane foams are disclosed in U.S. Reissue Patent 24,514 together with suitable machinery to be used in conjunction therewith. When water is added as the blowing agent, corresponding quantities of excess isocyanate to react with the water and produce carbon dioxide may be used. It is possible to proceed with the preparation of the polyurethane foams by a prepolymer technique wherein an excess of organic polyisocyanate is reacted in a first step with the polyol of the present invention to prepare a prepolymer having free isocyanate groups which is then reacted in a second step with water and/or additional polyol to prepare a foam. Alternatively, the components may be reacted in a single working step commonly known as the "one-shot" technique of preparing polyurethanes. Furthermore, instead of water, low boiling hydrocarbons such as pentane, hexane, heptane, pentene, and heptene; azo compounds such as azohexahydrobenzodinitrile; halogenated hydrocarbons such as dichlorodifluoromethane, trichlorofluoromethane, dichlorodifluoroethane, vinylidene chloride, and methylene chloride may be used as blowing agents.

Organic polyisocyanates which may be employed include aromatic, aliphatic, and cycloaliphatic polyisocyanates and combinations thereof. Representative of these types are the diisocyanates such as m-phenylene diisocyanate, 2,4-toluene diisocyanate, 2,6-toluene diisocyanate, mixtures of 2,4- and 2,6-toluene diisocyanate, hexamethylene diisocyanate, tetramethylene diisocyanate, cyclohexane-1,4-diisocyanate, hexahydrotoluene diisocyanate (and isomers), naphthalene-1,5-diisocyanate, 1-methoxyphenyl-2,4-diisocyanate, 4,4′-diphenylmethane diisocyanate, 4,4′-biphenylene diisocyanate, 3,3′-dimethoxy-4,4′-biphenyl diisocyanate, 3,3′-dimethyl-4,4′-biphenyl diisocyanate and 3,3′-dimethyldiphenylmethane-4,4′-diisocyanate; the triisocyanates such as 4,4′,4″-triphenylmethane triisocyanate, and toluene 2,4,6-triisocyanate; and the tetraisocyanates such as 4,4′-dimethyldiphenylmethane-2,2′-5,5′-tetraisocyanate and polymeric polyisocyanates such as polymethylene polyphenylene polyisocyanate. Especially useful due to their availability and properties are toluene diisocyanate, 4,4′-diphenylmethane diisocyanate and polymethylene polyphenylene polyisocyanate. Toluene diisocyanate is preferred.

Crude polyisocyanates may also be used in the compositions of the present invention, such as crude toluene diisocyanate obtained by the phosgenation of a mixture of toluene diamines or crude diphenylmethane isocyanate obtained by the phosgenation of crude diphenylmethane diamine. The preferred or crude isocyanates are disclosed in U.S. Patent No. 3,215,652.

Chain-extending agents which may be employed in the preparation of the polyurethane foams include those compounds having at least two functional groups bearing active hydrogen atoms such as water, hydrazine, primary and secondary diamines, amino alcohols, amino acids, hydroxy acids, glycols, or mixtures thereof. A preferred group of chain-extending agents includes water, ethylene glycol, 1,4-butanediol and primary and secondary diamines which react more readily with the prepolymer than does water such as phenylene diamine, 1,4-cyclohexane-bis-(methylamine), ethylenediamine, diethylenetriamine, N-(2-hydroxypropyl)ethylenediamine, N,N′-di(2-hydroxypropyl)ethylenediamine, piperazine, and 2-methylpiperazine.

Among the flame retardant compounds in conjunction with melamine which may be employed are tetrakis(2-chloroethyl) ethylene phosphonate, pentabromodiphenyl oxide, tris(1,3-dichloropropyl) phosphate,

tris(beta-chloroethyl) phosphate, molybdenum trioxide, ammonium molybdate, ammonium phosphate, pentabromodiphenyloxide, tricresyl phosphate, 2,3-dibromopropanol, hexabromocyclododecane, dibromoethyldibromocyclohexane, tris(2,3-dibromopropyl)phosphate, and tris(beta-chloropropyl)phosphate.

Any suitable catalyst may be used including tertiary amines such as, for example, triethylenediamine, N-methylmorpholine, N-ethylmorpholine, diethylethanolamine, N-cocomorpholine, 1-methyl-4-dimethylaminoethylpiperazine, 3-methoxypropyldimethylamine, N,N,N'-trimethylisopropyl propylenediamine, 3-diethylaminopropyldiethylamine, dimethylbenzylamine, and the like. Other suitable catalysts are, for example, stannous chloride,dibutyltin di-2-ethyl hexanoate, stannous oxide, as well as other organometallic compounds such as are disclosed in U.S. Patent No. 2,846,408.

A surface-active agent is generally necessary for production of high grade polyurethane foam according to the present invention, since in the absence of same, the foams collapse or contain very large uneven cells. Numerous surface-active agents have been found satisfactory. Nonionic surface active agents are preferred. Of these, the nonionic surface-active agents such as the well-known silicones have been found particularly desirable. Other surface-active agents which are operative, although not preferred, include polyethylene glycol ethers of long chain alcohols, tertiary amine or alkanolamine salts of long chain alkyl acid sulfate esters, alkyl sulfonic esters, and alkyl arylsulfonic acids.

The following examples illustrate the nature of the invention. All parts are by weight unless otherwise stated. In the examples, the physical properties of the polyurethane foam were determined by the following ASTM tests:

Density - D1622-63
Tensile Strength - D1623-72
Elongation - D412
Split Tear - D470
Compression Set - D395
Compression Load - D1564
Humid Aging - D1564
Dynamic Fatigue Test - D-3574I_2 and /D-3574I_3

Additional tests included the
California Bulletin No. 117 Test, Section D, Part II .

The following abbreviations are employed in the examples below:

Polyol A is a propylene oxide adduct of trimethylolpropane capped with 15 weight percent ethylene oxide having a hydroxyl number of 25.

Polyol B is a propylene oxide, adduct of glycerine capped with 16.5 weight percent ethylene oxide having a hydroxyl number of 35.

Polyol C is a propylene oxide adduct of trimethylolpropane capped with 13 weight percent ethylene oxide having a hydroxyl number of 35.

Polyol D is a propylene oxide adduct of trimethylolpropane capped with 4.8 weight percent ethylene oxide which is reacted with maleic anhydride and capped with 6 weight percent propylene oxide having a hydroxyl number of 22.

Polyol E is a mixture of Polyol C and Polyol D containing a graft polymer dispersion of 31 weight percent of 1:1 styrene:acrylonitrile having a hydroxyl number of 24.

Polyol F is a propylene oxide, ethylene oxide heteric adduct of glycerine containing 12 weight percent ethylene oxide having a hydroxyl number of 56.

Polyol G is a blend of 49.8 weight percent Polyol B, 38.4 weight percent Polyol E and 11.5 weight percent Polyol C.

Polyol H is a blend of 55 weight percent Polyol A and 45 weight percent Polyol E.

Polyol I is a propylene oxide ethylene oxide heteric adduct of glycerine containing 12.5 weight percent ethylene oxide having a hydroxyl number of 50.0.

Polyol J is a mixture of Polyol D and Polyol I containing a graft polymer dispersion of 40 weight percent of 2:1 styrene:acrylonitrile having a hydroxyl number of 29.0.

DEOA is diethanolamine.

TEOA is triethanolamine.

Silicone 5309 is a silicone surfactant manufactured by Dow Corning Corporation.

T-12 is dibutyltin dilaurate.

Freon 11 is monofluorotrichloromethane.

Dabco 33LV is a 33 percent solution of triethylene diamine in dipropylene glycol.

Niax A-1 is an amine catalyst manufactured by Union Carbide Corporation.

TDI is toluene diisocyanate.

Fyrol FR-2 is tri(β,β'-dichloro-isopropyl)phosphate manufactured by Stauffer Chemical Corporation.
Firemaster 836 is 3-bromo-2,2-dimethylpropyl-2-chloroethyl-2-bromoethyl phosphate.
Reactint Blue X-8515 is a polymeric colorant manufactured by Milliken Chemical.
Silicone DC-5043 is a silicone surfactant manufactured by Dow Corning Corporation.
Reactint Red X-26850 is a polymeric colorant manufactured by Milliken Chemical.
Reactint Yellow X-74 is a polymeric colorant manufactured by Milliken Chemical.
Silicone B-3640 is a silicone surfactant manufactured by Goldschmidt AG.
DOP is dioctylphthalate.
PCF is pounds per cubic foot.
CFM is cubic feet per minute.

Examples 1-41

Examples 1-41 were prepared by employing the following procedure:

The components as indicated in Tables I-VII which were placed into various tanks were metered into a Hennecke UBT foam machine running with a mixer speed of 5000 rpm in the amounts indicated. The mixture was discharged from the mixing head unto a conveyor. The foam expanded to its full height in about three minutes. After a sufficient cure time the foams were submitted for physical property determinations.

TABLE I

Example	1	2	3	4
FORMULATIONS				
Polyol H	100.00	---------->		
T-12	0.1	---------->		
Melamine	-	-	20.0	20.0
Fyrol FR-2	5.0	-	2.0	-
Reactint Blue X-8515	0.05	-	0.02	-
Silicone DC-5043	1.2	---------->		
Water	2.05	2.05	2.25	2.25
DEOA	0.80	0.80	1.08	1.08
Dabco 33LV	0.12	---------->		
Niax A-1	0.04	---------->		
TDI (110 Index)	28.25	28.25	31.14	31.14
FOAM PROPERTIES				
Density, pcf	2.56	2.46	2.61	2.59
Tensile strength, psi	23.2	21.7	15.8	16.1
Elongation, %	190	197	157	153
Tear, pi	2.4	2.3	1.9	1.7
Resilience, %	72	72	70	70
ILD, lb/50 sq.in. (4 inch)				
25%	34.7	33.1	34.4	33.1
65%	81.9	78.5	84.3	83.5
Sag Factor	2.36	2.37	2.45	2.52
Recovery, %	84.6	82.5	81.0	81.7
Compression sets, % set				
50%	6.0	6.2	6.8	8.0
90%	4.7	5.7	7.0	6.6
Humid aged 5 hrs. at 250°C				
CLD, % of original				
50%	58.9	52.3	58.8	54.1
Compression Sets, % Set				
50%	12.0	14.4	14.8	17.3
90%	10.8	48.6	12.4	13.5
H.A. Tensile, psi	25.9	17.3	13.5	10.9
Air flow, cfm	1.0	1.3	1.4	1.5
Pounding Fatigue, % Loss (4 inch)				
Height	1.3	1.2	1.7	1.5
40% ILD	14.0	16.5	20.8	20.6
COMPARISON OF FOAM FLAMMABILITY PROPERTIES				
CAL. 117 OPEN FLAME	Pass	Fail	Pass	Pass
CAL. 117 SMOLDERING		% Wt. Retained (min. 80.0%)		
Original	99.4	99.6	99.3	99.3
Air Flow, cfm	1.0	1.3	1.4	1.5
Pounding Fatigue	Burned	61.9	98.0	99.3
Air Flow, cfm	1.1	1.5	1.3	1.4
Roller Shear	Burned	31.1	93.6	98.6
Air Flow, cfm	2.3	2.7	2.5	2.7

TABLE II

Example	5	6	7	8
FORMULATIONS				
Polyol H	100.00	---------->		
T-12	0.1	---------->		
Melamine	-	-	20.0	20.0
Fyrol FR-2	5.0	-	2.0	-
Reactint Blue X-8515	0.05	-	0.02	-
Silicone DC-5043	1.2	---------->		
Water	2.8	2.8	3.0	3.0
DEOA	1.09	1.09	1.37	1.37
Dabco 33LV	0.12	---------->		
Niax A-1	0.04	---------->		
TDI (110 Index)	37.02	37.02	39.91	39.91
FOAM PROPERTIES				
Density, pcf	1.88	1.81	1.99	1.98
Tensile strength, psi	21.4	19.8	14.6	15.5
Elongation, %	190	187	153	157
Tear, pi	2.3	1.8	1.7	1.8
Resilience, %	62	65	62	62
ILD, lb/50 sq.in. (4 inch)				
25%	31.5	26.8	30.4	28.5
65%	69.6	61.9	72.2	68.0
Sag Factor	2.21	2.31	2.37	2.39
Recovery, %	75.9	77.9	75.2	74.9
Compression sets, % set				
50%	6.9	7.9	10.9	11.1
90%	7.5	6.7	69.7	73.5
Humid aged 5 hrs. at 250°C				
CLD, % of original				
50%	72.5	71.1	77.2	79.8
Compression Sets, % Set				
50%	17.6	21.7	43.6	57.1
90%	18.2	83.8	81.3	80.1
H.A. Tensile, psi	25.5	18.6	12.9	10.9
Air flow, cfm	1.1	1.3	1.3	1.4
Pounding Fatigue, % Loss (4 inch)				
Height	1.4	2.1	2.0	2.1
40% ILD	25.3	22.9	31.4	30.5
COMPARISON OF FOAM FLAMMABILITY PROPERTIES				
CAL. 117 OPEN FLAME	Pass	Fail	Pass	Pass
CAL. 117 SMOLDERING		% Wt. Retained (min. 80.0%)		
Original	99.7	99.5	99.1	99.1
Air Flow, cfm	1.1	1.3	1.3	1.4
Pounding Fatigue	64.9	79.6	98.7	99.4
Air Flow, cfm	1.3	1.2	1.4	1.6
Roller Shear	Burned	Burned	99.3	989.7
Air Flow, cfm	3.6	3.5	3.4	3.7

TABLE III

Example	9	10	11	12
FORMULATIONS				
Polyol H	100.00	---------->		
T-12	0.1	---------->		
Melamine	10.0	---------->		
Fyrol FR-2	2.0	-	2.0	-
Reactrint Blue X-8515	0.02	-	0.02	-
Silicone DC-5043	1.2	---------->		
Water	2.15	2.15	2.9	2.9
DEOA	0.94	0.94	1.23	1.23
Dabco 33LV	0.12	---------->		
Niax A-1	0.04	---------->		
TDI (110 Index)	29.42	29.42	38.19	38.19
FOAM PROPERTIES				
Density, pcf	2.39	2.36	1.76	1.74
Tensile strength, psi	17.2	18.4	17.2	15.9
Elongation, %	163	187	170	187
Tear, pi	2.2	2.1	2.2	2.1
Resilience, %	68	68	62	62
ILD, lb/50 sq.in. (4 inch)				
25%	31.5	31.1	24.9	23.3
65%	76.6	76.0	58.5	54.9
Sag Factor	2.43	2.45	2.35	2.36
Recovery, %	80.7	81.1	73.2	73.6
Compression sets, % set				
50%	7.9	8.2	13.3	14.2
90%	7.8	9.1	86.2	83.1
Humid aged 5 hrs. at 250°C				
CLD, % of original				
50%	57.4	53.1	84.3	92.1
Compression Sets, % Set				
50%	18.0	20.4	61.6	72.9
90%	19.0	38.4	89.3	90.9
H.A. Tensile, psi	14.1	13.3	14.7	12.3
Air flow, cfm	1.3	1.2	1.1	1.3
Pounding Fatigue, % Loss (4 inch)				
Height	1.7	1.8	3.8	1.6
40% ILD	22.5	25.1	32.9	21.5
COMPARISON OF FOAM FLAMMABILITY PROPERTIES				
CAL. 117 OPEN FLAME	Pass	Fail	Pass	Fail
CAL. 117 SMOLDERING		% Wt. Retained (min. 80.0%)		
Original	99.1	99.3	99.6	99.3
Air Flow, cfm	1.3	1.2	1.1	1.3
Pounding Fatigue	77.8	99.5	81.6	97.9
Air Flow, cfm	1.3	1.3	1.3	1.3
Roller Shear	99.3	83.9	58.6	93.7
Air Flow, cfm	2.5	3.2	3.4	3.4

TABLE IV

Example		13	14	15
FORMULATIONS	STREAM			
Polyol H	I	100.0	80.0	70.0
DEOA	I	1.2	0.96	0.84
T-12	I	0.15	0.12	0.105
Reactint Blue X-8515	I	0.1	0.08	0.07
Polyol G	II	-	20.0	30.0
DEOA	II	-	0.24	0.36
T-12	II	-	0.03	0.045
Reactint Red X-26850	II	-	0.01	0.015
Melamine	II	-	20.0	30.0
Water	III	2.8	2.8	2.8
DABCO 33LV	III	0.15	0.15	0.15
NIAX A-1	III	0.05	0.05	0.05
Silicone DC-5043	IV	1.2	1.2	1.2
FYROL FR-2	V	6.0	3.0	3.0
Reactint Yellow X-74	V	0.12	0.06	0.06
TDI (110 Index)	VII	38.31	38.31	38.31
FOAM PROPERTIES				
Density, pcf		2.00	2.50	2.65
Tensile strength, psi		15.7	17.7	18.4
Elongation, %		130	163	130
Tear, pi		1.4	1.8	1.8
Resilience, %		50	64	60
ILD, lb/50 sq.in. (4 inch)				
25%		32.4	34.4	41.2
65%		76.1	85.6	104.7
Sag Factor		2.35	2.49	2.54
Recovery, %		78.8	78.3	75.7
Compression sets, % set				
50%		12.5	9.7	9.3
90%		8.4	8.2	27.6
Air Flow Crushed		0.7	1.9	0.7
POUNDING FATIGUE, % LOSS				
Height		2.1	2.3	1.8
40% ILD		25.5	29.1	28.5
COMPARISON OF FOAM FLAMMABILITY PROPERTIES				
CAL. 117 OPEN FLAME				
Original		Pass	Pass	Pass
Heat Aged		Pass	Pass	Pass
Smoldering, % Wt.				
Original, Retained		84.5	99.3	99.5
Air Flow, Crushed Twice		0.7	1.9	0.7
Fatigue, Retained		64.9	99.2	99.3
Air Flow, Fatigued		0.8	2.6	1.3

TABLE V

Example		16	17	18	19
FORMULATIONS	STREAM				
Polyol H	I	100.0	80.0	70.0	60.0
DEOA	I	1.0	0.8	0.7	0.6
T-12	I	0.15	0.12	0.105	0.09
Reactint Blue X-8515	I	0.1	0.08	0.07	0.06
Polyol G	II	-	20.0	30.0	40.0
DEOA	II	-	0.2	0.3	0.4
T-12	II	-	0.03	0.045	0.06
Reactint Red X-26850	II	-	0.01	0.015	0.02
Melamine	II	-	20.0	30.0	40.0
Water	III	2.2	2.2	2.2	2.2
DABCO 33LV	III	0.18	0.18	0.18	0.18
NIAX A-1	III	0.06	0.06	0.06	0.06
Silicone DC-5043	IV	1.2	1.2	1.2	1.2
FYROL FR-2	V	6.0	3.0	3.0	3.0
Reactint Yellow X-74	V	0.12	0.06	0.06	0.06
DEOA	VI	-	0.23	0.23	0.23
TDI (110 Index)	VII	31.165	31.711	31.711	31.711
FOAM PROPERTIES					
Density, pcf		2.52	2.87	3.17	3.25
Tensile strength, psi		14.1	16.7	16.8	16.5
Elongation, %		117	133	127	117
Tear, pi		1.5	1.5	1.3	1.4
Resilience, %		64	66	62	64
ILD, lb/50 sq.in. (4 inch)					
25%		37.1	38.4	42.2	41.2
65%		88.9	100.2	115.9	118.2
Sag Factor		2.39	2.61	2.75	2.87
Recovery, %		80.3	81.3	80.6	79.6
Compression sets, % set					
50%		10.0	6.8	9.6	9.4
90%		7.2	6.3	8.3	7.5
Air Flow Crushed		1.6	0.9	1.0	1.2
POUNDING FATIGUE, % LOSS					
Height		1.9	2.1	0.6	2.1
40% ILD		17.0	17.7	23.7	19.9
COMPARISON OF FOAM FLAMMABILITY PROPERTIES					
CAL. 117 OPEN FLAME					
Original		Pass	Pass	Pass	Pass
Heat Aged		Pass	Pass	Pass	Pass
Smoldering, % Wt.					
Original, Retained		89.6	99.6	99.4	99.8
Air Flow, Crushed Twice		1.6	0.9	1.0	1.2
Fatigue, Retained		64.9	99.2	99.5	99.7
Air Flow, Fatigued		1.0	1.1	1.0	0.9

TABLE VI

Example	20	21	22	23	24	25	26	27	28	29
FORMULATION										
Polyol F	100.0	----------								
FYROL FR-2	12.0	----------					6.0	----------		
Melamine	-	-	-	-	-	-	15.0	----------		
Silicone B-3640	1.0	----------								
T-10/DOP (50/50)	1.3	1.1	0.9	1.3	1.1	0.9	1.1	0.9	1.1	0.9
Water	4.7	4.7	4.7	3.2	3.2	3.2	4.7	4.7	3.2	3.2
DABCO 33LV	0.26	0.26	0.26	0.18	0.18	0.28	0.26	0.26	0.18	0.18
Reactint Red X-26850	0.2	----------								
TDI (115 Index)	62.25	62.25	62.25	45.57	45.57	45.57	62.25	62.25	45.57	45.57
Density, pcf	1.39	1.42	1.47	1.80	1.86	1.91	1.52	1.54	2.02	2.01
FLAMMABILITY PROPERTIES										
California 117, Original										
Vertical	Pass	Fail	Fail	Pass	Pass	Pass	Fail	Fail	Pass	Pass
Smoldering	99.7	85.0	71.7	99.5	99.3	78.5	97.0	82.0	99.3	80.8
Air Flow, cfm	1.3	3.3	4.4	0.6	1.3	2.6	2.9	5.8	0.7	3.8
FATIGUE, 4000 CYCLES - 30 LBS.										
Smoldering	71.9	69.3	72.8	66.4	74.7	72.0	94.7	81.6	97.5	80.4
Air Flow, cfm	2.3	3.6	5.2	3.3	2.8	4.8	4.0	5.7	3.4	4.5

TABLE VI - continued

Example	30	31	32	33	34	35	36	37	38	39
FORMULATION										
Polyol F	100.0	----------								
FYROL FR-2	3.0	----------			-	-	-	-	-	-
Melamine	20.0	----------			25.0	----------				
Silicone B-3640	1.0	----------								
T-10/DOP (50/50)	1.1	0.9	1.1	0.9	1.3	1.1	0.9	1.3	1.1	0.9
Water	4.7	4.7	3.2	3.2	4.7	4.7	3.2	3.2	3.2	3.2
DABCO 33LV	0.26	0.26	0.18	0.18	0.26	0.26	0.26	0.18	0.18	0.18
Reactint Red X-26850	0.2	----------								
TDI (115 Index)	62.25	62.25	45.57	45.57	62.25	62.25	62.25	45.57	45.57	45.57
Density, pcf	1.53	1.56	2.02	2.03	1.49	1.498	1.48	1.91	1.98	20.1
FLAMMABILITY PROPERTIES										
California 117, Original										
Vertical	Fail	Fail	Pass	Pass	Fail	Fail	Fail	Fail	Pass	Pass
Smoldering	98.7	86.6	99.6	98.8	99.6	99.3	99.0	99.6	99.7	98.9
Air Flow, cfm	3.7	5.7	1.4	4.3	0.7	2.2	4.9	0.4	0.9	2.7
				FATIGUE, 4000 CYCLES - 30 LBS.						
Smoldering	97.6	97.5	98.6	99.1	98.6	98.3	99.5	98.5	95.6	98.5
Air Flow, cfm	3.5	5.5	3.6	4.4	1.3	4.1	2.9	1.6	4.9	3.7

TABLE VII

Example		40	41
FORMULATION	STREAM		
Polyol G	I	100.0	90.0
DEOA	I	1.0	
T-12	I	0.15	0.135
Reactint Blue X-8515	I	0.1	0.09
FIREMASTER 836	I	-	3.0
Reactint Yellow X-74	I	-	0.045
Polyol G	II	-	10.0
Melamine	II	-	5.0
DEOA	II	-	0.1
T-12	II	-	0.015
Reactint Red X-26850	II	-	0.01
Water	III	2.0	2.3
DABCO 33LV	III	0.18	0.21
NIAX A-1	III	0.06	0.07
Silicone L-5309	IV	1.2	1.2
FIREMASTER 836	V	6.0	
Reactint Yellow	V	0.1	
DEOA	V		1.035
TDI	VI	29.25	32.44
Index		110	110

TABLE VII - continued

Example	40	41
FOAM PROPERTIES		
Density, pcf	2.71	2.45
Tensile, psi	20.7	19.8
Elongation, %	147	157
Tear, pi	1.8	2.1
Resilience, %	52	60
ILD, lb/50 sq.in. (4 inch)		
25%	35.6	35.2
65%	88.6	84.4
Sag Factor	2.49	2.40
Recovery, %	84.3	79.6
Compression Sets, %		
50%	9.1	10.6
90%	7.8	75.2
Humid Aged 5 Hrs. at 250°F.		
CLD, % of Original 50%	54.2	47.3
Compression Set, % - 50%	25.3	26.8
- 90%	79.2	58.1
H.A. Tensile Strength, psi	20.4	17.7
Air Flow, cfm	1.0	0.9
Pounding Fatigue, % Loss (2 inch)		
Height	0.6	0.2
40% ILD	20.5	31.6
Flammability Properties		
California 117 Open Flame		
Original	Pass	Pass
Heat Aged	Pass	Pass
California 117 Smoldering		
Spec. Min.		
Original 80%	99.6	98.2
Crushed	61.6	95.8
Fatigued	64.1	95.5

TABLE VIII

Example	42	43	44
FORMULATION			
Polyol A	100.0	-	-
Polyol I	-	100.0	82.0
Polyol J	-	-	18.0
Stannous Octoate	0.37	0.24	0.11
NIAX A-1	0.16	0.12	0.09
Silicone 5309	1.85	1.05	1.10
Methylene Chloride	16.5	4.5	0.70
H_2O	3.90	3.85	2.85
FYROL FR-2	12.0	12.7	12.8
TDI	53.8	51.0	42.0
Index	1.2	1.1	1.2
Physical Properties			
Density, pcf	1.0	1.4	2.1
Air Flow, cfm	4.0	1.6	2.7

Examples 42-44 are illustrated in Figure I. The products illustrated in Figures II-IV were prepared in a manner similar to those in Examples 1-41.

Claims

1. A flame retardant flexible polyurethane foam which retains its cigarette smoldering resistance after service consisting essentially of (a) a polyoxyalkylene polyether polyol, (b) an organic polyisocyanate, (c) a blowing agent, (d) a catalyst, (e) a surfactant, (f) melamine, and (g) optionally chain extenders and flame retardants other than melamine, wherein the concentration of melamine is from about 5 weight percent to about 25 weight percent of the weight of the foam and wherein the density of the foam is from at least 1.2 lbs/ft^3 to about 4 lbs/ft^3.

2. The foam of claim 1 wherein the organic polyisocyanates is toluene diisocyanate.

3. The foam of claim 1 wherein the polyol has an equivalent weight from 1000 to 10,000 and a functionality of two to four.

4. The foam of claim 1 wherein the density of the foam is from 1.5 lbs/ft^3 to about 4 lbs/ft^3.

5. The foam of claim 1 wherein the density of the foam is from 2.0 lbs/ft^3 to about 3.5 lbs/ft^3.

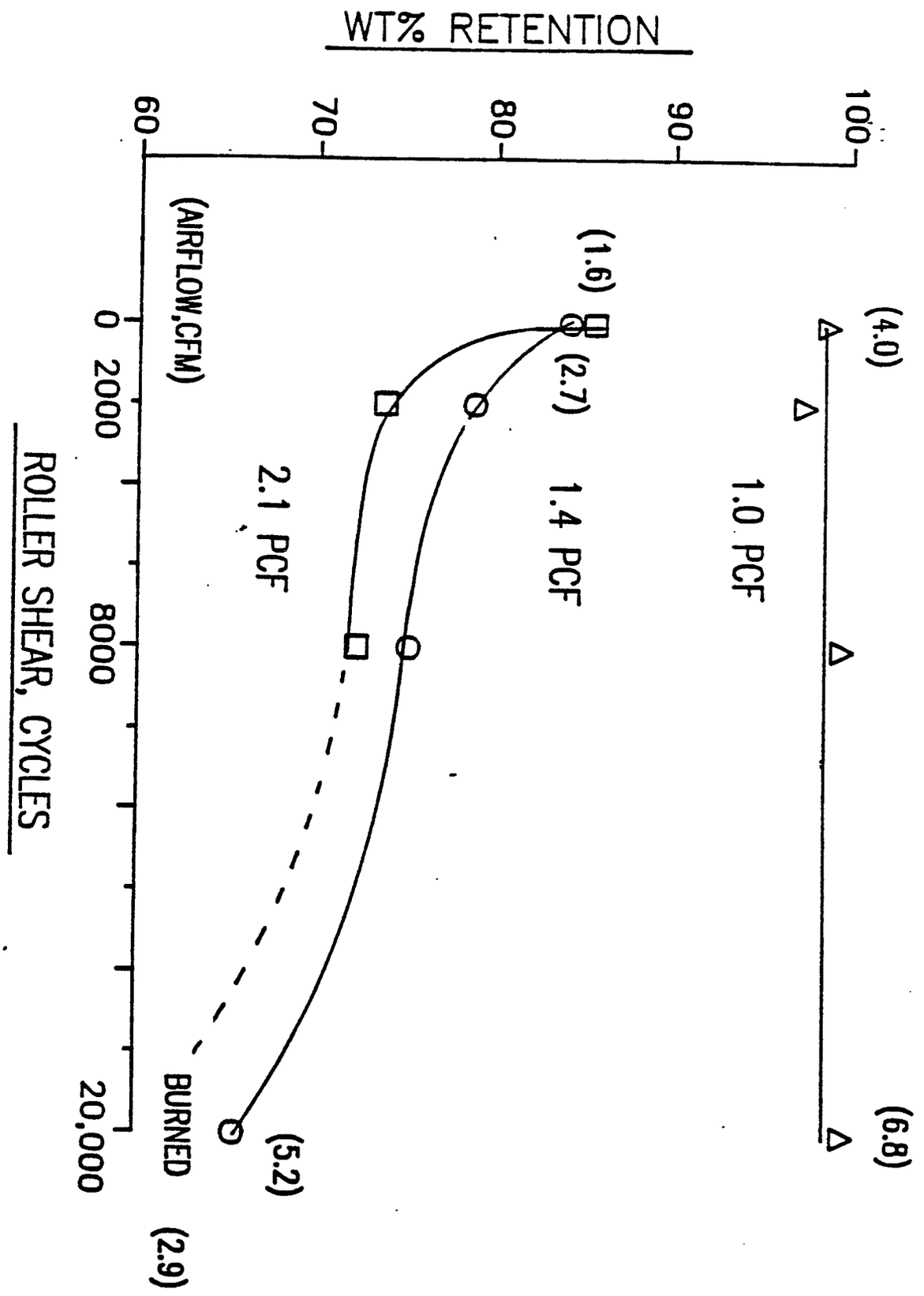
WT% RETENTION
60
70
80
90
100
(AIRFLOW,CFM)
0
2000
8000
20,000
ROLLER SHEAR, CYCLES
(1.6)
(2.7)
(4.0)
(6.8)
2.1 PCF
1.4 PCF
1.0 PCF
(5.2)
BURNED
(2.9)

FIGURE I

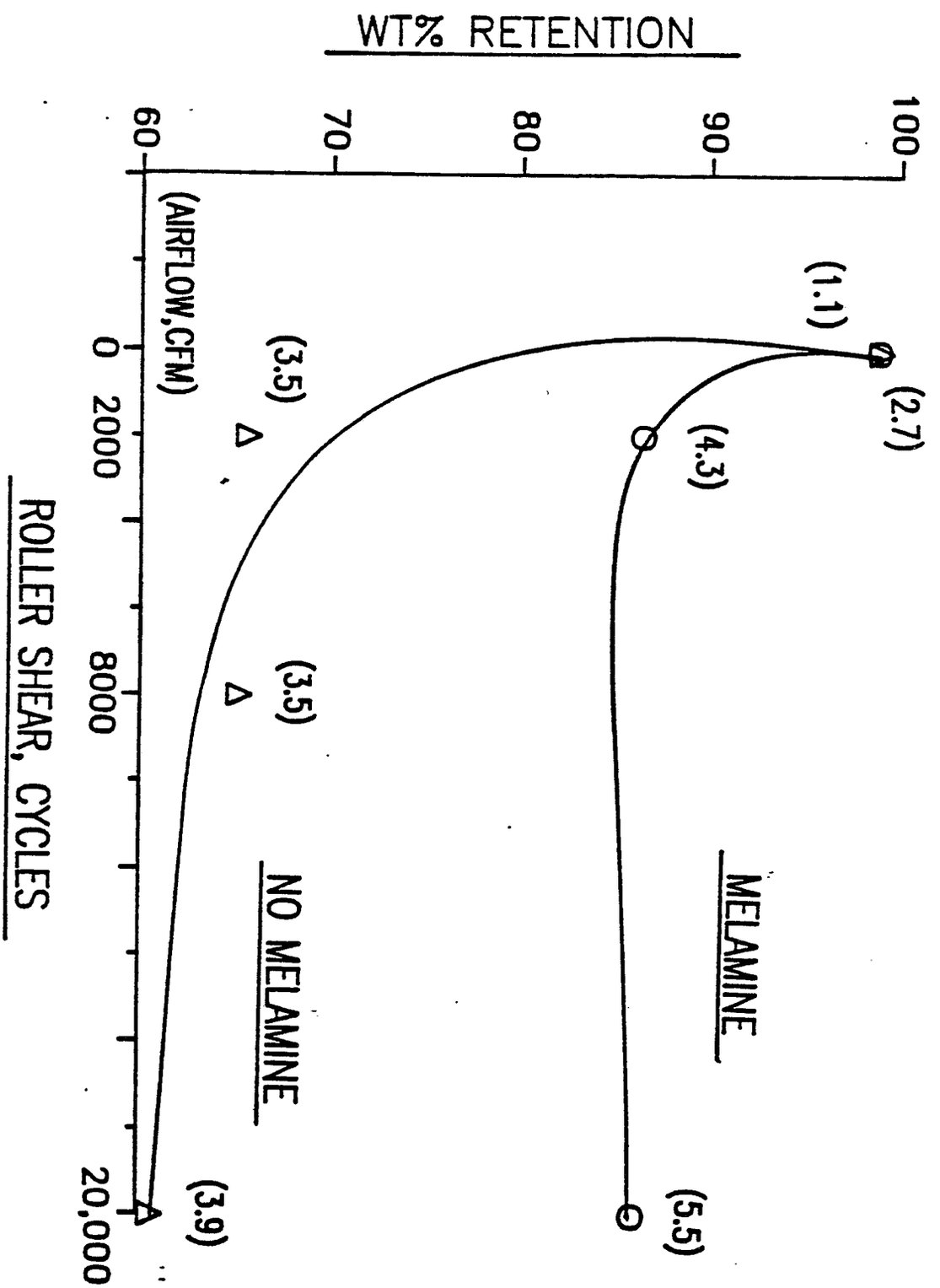
WT% RETENTION
60
70
80
90
100
(AIRFLOW,CFM)
0
2000
8000
20,000
ROLLER SHEAR, CYCLES
(1.1)
(2.7)
(4.3)
(3.5)
(3.5)
(3.9)
(5.5)
MELAMINE
NO MELAMINE

FIGURE II

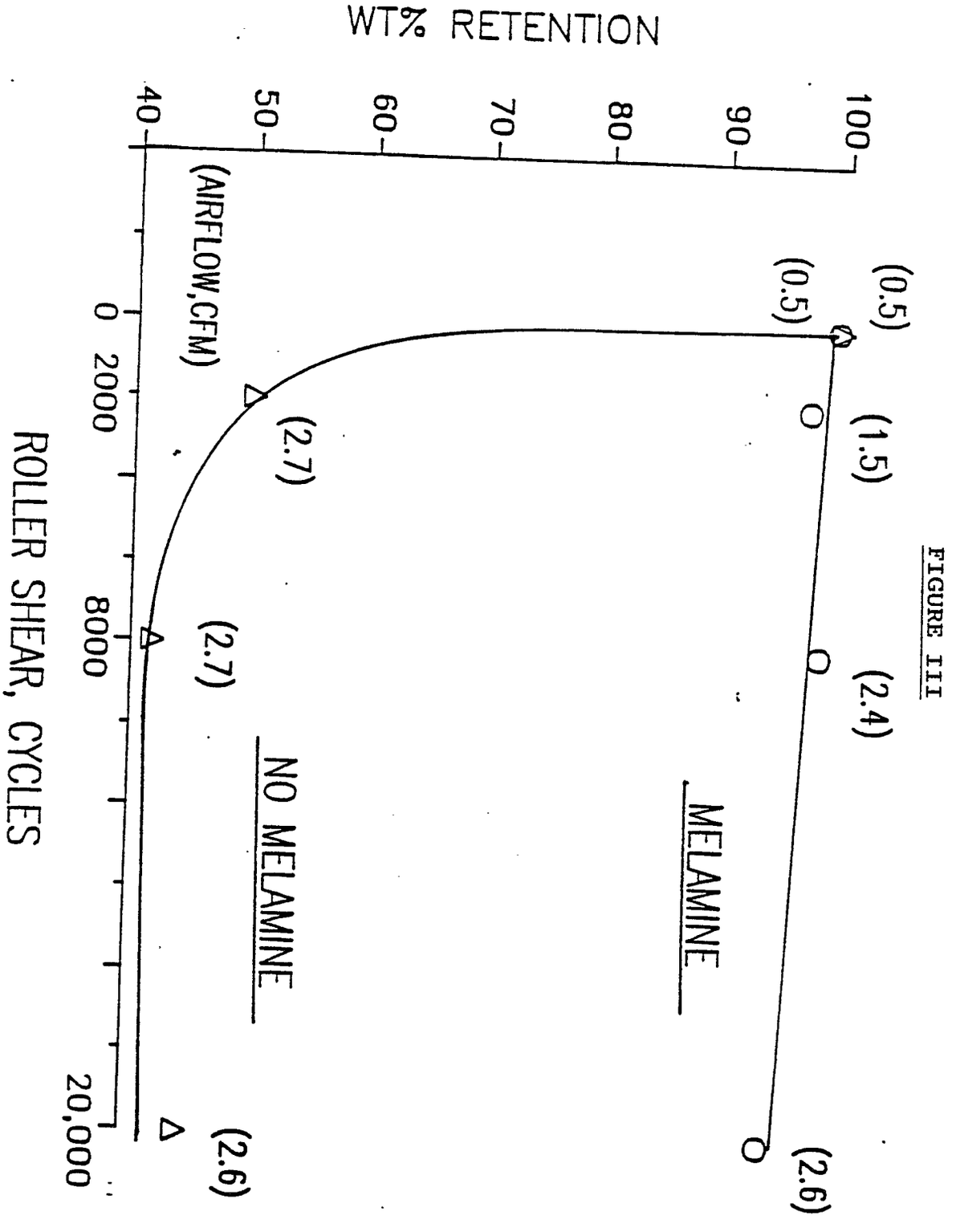
WT% RETENTION
40
50
60
70
80
90
100
(AIRFLOW,CFM)
0
2000
8000
20,000
ROLLER SHEAR, CYCLES
(0.5)
(0.5)
(1.5)
(2.4)
(2.6)
(2.6)
(2.7)
(2.7)
(2.6)
NO MELAMINE
MELAMINE

FIGURE III

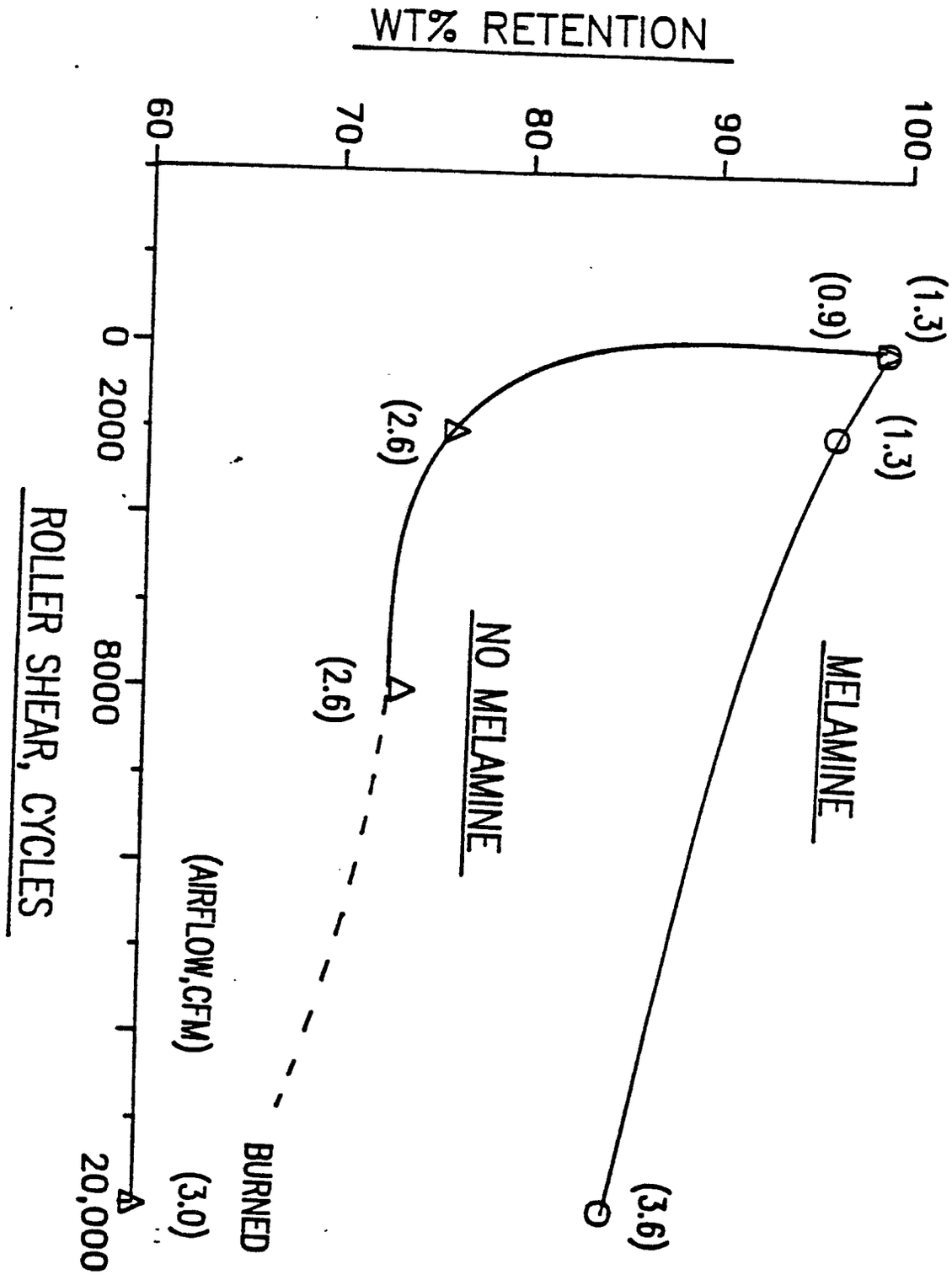
WT% RETENTION
60
70
80
90
100
0
2000
8000
20,000
ROLLER SHEAR, CYCLES
(AIRFLOW,CFM)
(3.0)
BURNED
NO MELAMINE
MELAMINE
(2.6)
(2.6)
(0.9)
(1.3)
(1.3)
(3.6)

FIGURE IV

European Patent Office

EUROPEAN SEARCH REPORT

Application Number

EP 88 11 0993

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl.4)
X	GB-A-2 177 405 (BASF CORP.) * Claims 1,2; page 1, lines 35-39; page 3, lines 13-55; tables 1-4 *	1-5	C 08 K 5/34 C 08 J 9/00 C 08 G 18/38 C 08 L 75/08
X	DE-A-2 809 084 (BRIDGESTONETIRE) * Claims 1,3,5,8; page 6, paragraph 2; page 7, paragraph 3; page 8, last paragraph - page 10, paragraph 1; examples 1-8; table 1 * & US-A-4 221 875 (Cat. A)	1-5	
A	GB-A-2 177 406 (BASF CORP.) * Claim 1 *	1	
A	EP-A-0 004 618 (BASF CORP.) * Claims 1,4-6; page 5, line 19 - page 6, line 14; page 7, lines 8-15; page 8, line 21 - page 9, line 11 *	1	
D,A	US-A-4 385 131 (R.N.FRACALOSSI et al.) * Claims 1-3 *	1	

TECHNICAL FIELDS SEARCHED (Int. Cl.4)

C 08 K
C 08 J
C 08 G

The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
THE HAGUE	09-11-1988	VAN PUYMBROECK M.A.

CATEGORY OF CITED DOCUMENTS

X : particularly relevant if taken alone
Y : particularly relevant if combined with another document of the same category
A : technological background
O : non-written disclosure
P : intermediate document

T : theory or principle underlying the invention
E : earlier patent document, but published on, or after the filing date
D : document cited in the application
L : document cited for other reasons

& : member of the same patent family, corresponding document

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