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PATENT SPECIFICATION

NO DRAWINGS

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COMPLETE SPECIFICATION

A process for the production of Polyurethane Foam

We, TAKEDA CHEMICAL INDUSTRIES LIMITED, a company organised under the laws of Japan, of 27 Doshomachi, 2-chome, Higashi-ku, Osaka, Japan, 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 a process for the production of flexible polyurethane foam by using dimethylbenzene ω, ω' -diisocyanate as an isocyanato component in the presence of octaalkyl-stannoxanes and monoaminoalcohols.

It has been proposed to produce polyurethane foam from an aromatic isocyanate (e.g. tolylene diisocyanate, diphenylmethane diisocyanate,) and a polyether polyol or a polyester polyol. However, foam produced in this way is yellowed soon after its exposure to atmosphere, so that its practical application to practical use is remarkably limited.

On the other hand, polyurethane resins produced by using an aliphatic diisocyanate (e.g. hexamethylene diisocyanate) as an isocyanato component, are not so readily yellowed as those derived from an aromatic isocyanate, but have much inferior physical properties.

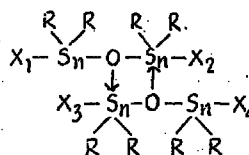
Furthermore, it is substantially impossible to prepare polyurethane foam economically by using an aliphatic diisocyanate, because of the remarkably low reactivity of the aliphatic isocyanate with polyols.

It was found that by using dimethylbenzene ω, ω' -diisocyanate as isocyanato component and octaalkyl-stannoxane as catalyst, a polyurethane foam can be produced which has good physical properties and is not yellowed even after exposure to atmosphere for a considerable period of time. However, foams produced by this method are often subject to cracking.

We have now discovered that the presence

of a certain monoaminoalcohol during production of polyurethane foam by the above mentioned method can effectively inhibit occurrence of cracks in the foam.

According to the invention there is provided a process for the production of polyurethane foam comprising reacting dimethylbenzene ω, ω' -diisocyanate with an isocyanate-modified polyether polyol having a hydroxyl number of 25 to 75 or a polyester polyol having a hydroxyl number of 25 to 75 in the presence of a blowing agent, a compound of the formula:



wherein X_1 , X_2 , X_3 and X_4 may be the same or different and represent halogen, —OH, alkoxy having not more than 4 carbon atoms, formyloxy, alkylcarbonyloxy having not more than 17 carbon atoms, —NCO and —NCS, and R represents alkyl having not more than 4 carbon atoms; and a monoaminoalcohol having not more than 4 carbon atoms.

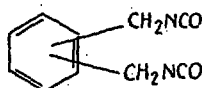
The present invention makes it possible to use lactone-polyester polyol as a polyol component in a so-called one-shot process for producing polyurethane foam while the lactone-polyester polyol has not been usable in the hitherto known one-shot process for the production of polyurethane foam.

Dimethylbenzene ω, ω' -diisocyanate is represented by the formula

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which encompasses 1,2-dimethylbenzene ω,ω' -diisocyanate, 1,3 - dimethylbenzene ω,ω' -diisocyanate, 1,4-dimethylbenzene ω,ω' -diisocyanate and mixtures thereof. From the practical point of view, a mixture of the 1,3-isomer and 1,4-isomer, especially a mixture of 60 to 90 weight percent of the 1,3-isomer and 40 to 10 weight percent of the 1,4-isomer, is advantageously employed.

15 Polyol employable in the present invention is an isocyanate-modified polyether polyol or polyester polyol, each having two or more terminal hydroxyl groups and having a hydroxyl number of 25 to 75, preferably about 30 to about 65.

The isocyanate-modified polyether polyols may be those obtained by reacting an excess amount of such polyether polyol as mentioned below with a low molecular weight polyisocyanate such as tolylene diisocyanate, diphenylmethane diisocyanate or dimethylbenzene ω,ω' -diisocyanate by heating in the presence of the catalyst for urethane linkage formation.

As the polyether polyol employable in producing the isocyanate-modified polyether polyol, there may be used products obtained by addition polymerization of alkylene oxides (e.g. ethylene oxide, propylene oxide, butylene oxide, styrene oxide and mixtures thereof) to low molecular weight polyhydric alcohols employed as an initiator (e.g. ethylene glycol, diethylene glycol, propylene glycol, glycerol, trimethylol propane, tetramethylol, pentaerythritol, sorbitol, sucrose, mannitol, sorbitol, mannitan, sorbitan and mixtures thereof) or to amines (e.g. ethylenediamine, propylenediamine, ethanolamine and mixtures thereof) under alkaline or acid conditions.

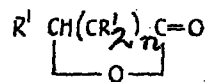
40 These polyetherpolyols may be prepared in a known manner such as described in "High Polymer Vol. XIII. Polyethers Part 1" (1963) by Norman G. Gaylord published by Interscience Publishers.

45 Among the isocyanate-modified polyether
polyols, the most desirable one is the poly-
ether polyol modified by dimethylbenzene
50 ω, ω' -diisocyanate, which is prepared by react-
ing under heating the above-mentioned poly-
ether polyol in excess with dimethylbenzene
 ω, ω' -diisocyanate in the presence of the octa-
alkyl-stannoxane of the formula (II) as shown
below.

As polyester polyols employable in this invention there may be counted such a product as obtained by the reaction of low molecular weight polyhydric alcohols (e.g. ethylene glycol, propylene glycol, diethylene glycol, triethylene glycol, butylene glycol, triethylol propane, glycerol, hexanetriol, pentaerythritol

and mixtures thereof) with polycarboxylic acids (e.g. succinic acid, glutaric acid, pimelic acid, suberic acid, azelaic acid, sebacic acid, maleic acid, fumaric acid, itaconic acid, phthalic acid, iso-phthalic acid, terephthalic acid, their acid anhydrides and mixtures thereof) and may also be counted a polyactone polyester polyol such as obtained by reacting a lactone with a polyfunctional initiator by heating in the presence or absence of a catalyst, the initiator being exemplified by diols (e.g. ethylene glycol, propylene glycol, diethylene glycol, 1,4-butanediol, xylenediol) diamines (e.g. ethylenediamine, phenyldiamine, xylylenediamine), aminoalcohols (e.g. ethanolamine, p-aminophenethyl alcohol etc.), polyols (e.g. glycerol, trimethylol propane, pentaerythritol, sorbitol, sucrose, mannitol, sorbitol, oxyalkylene polyol) or polyamines (e.g. diethanolamine, diisopropanolamine).

The lactones employable are represented by, for example, the general formula:

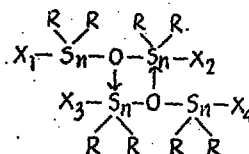


(I)

wherein n is an integer not less than 3, at least n + 2 R's are hydrogen, the remainder of the R's are alkyl, cycloalkyl, alkoxy or single ring aromatic hydrocarbon radicals, none of the substituents containing more than about twelve carbon atoms, and the total number of carbon atoms in the substituents on a lactone ring not exceeding twelve. As typical lactones there are exemplified δ -valerolactone, ϵ -caprolactone, γ -butyrolactone, γ -caprolactone, β -ethyl- δ -valerolactone or dimethyl capro-lactone.

The polyester polyol may be prepared in a known manner such as described in, for example, "Polyesters and their Application" third edition, April, 1959, published by 100 Bjorksten Research Lab. Inc: Inc. New York, U.S.A., and U.S.P. No. 2,890,208, No. 2,977,885 and 2,933,478.

In the present invention, the -octaalkyl stannoxane shown by the general formula (II) 105 is used as a catalyst.



(II)

wherein X_1 , X_2 , R_3 , and X_4 may be the same or different and represent halogen (i.e. Cl, Br, I or F) OH, alkoxy, formyloxy, alkyl-

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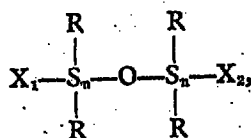
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carbonyloxy, NCO or NCS, and R represents alkyl, "alkyl" and "alkoxy" containing not more than 4 carbon atoms and "alkylcarbonyloxy" containing 2 to 17 carbon atoms, inclusive.

This compound was hertofore simply called tetraalkyl-distannoxane of the formula



but it has been confirmed that the compound exists under normal conditions in a form of dimer, namely the octaalkyl-stannoxane of the formula (II). Therefore, in the present specification, the compound is described and explained as the dimer, the octaalkyl-stannoxane of the formula (II).

The stannoxane of the formula (II) may be prepared, for example, by the procedure as described in "Journal of Organometallic Chemistry" Volume 1, No. 1, pages 81-88 (1963).

In the present invention, the most typical and desirable octaalkyl-stannoxanes for the practical use are exemplified as follows:—

- 1) Octamethyl-tetrachloro-stannoxane
- 2) Octabutyl-tetrachloro-stannoxane
- 3) Octabutyl-tetrabromo-stannoxane
- 4) Octabutyl - dichloro - dihydroxy - stannoxane
- 5) Octabutyl-tetraacetoxy-stannoxane
- 6) Octaethyl-tetraformyloxy-stannoxane
- 7) Octabutyl - diacetoxy - dihydroxy - stannoxane
- 8) Octabutyl-tetrauroxyloxy-stannoxane
- 9) Octapropyl - tetrastearoyloxy - stannoxane
- 10) Octabutyl-tetraisocyanato-stannoxane
- 11) Octaethyl - diisocyanato - dihydroxy-stannoxane
- 12) Octamethyl - diisocyanato - dimethoxy-stannoxane
- 13) Octabutyl - tetraisoithiocyanato - stannoxane
- 14) Octabutyl - diisoithiocyanato - diethoxy-stannoxane
- 15) Octamethyl - dichloro - dimethoxy-stannoxane
- 16) Octaethyl - dibromo - diethoxy - stannoxane
- 17) Octabutyl - diacetoxy - diethoxy - stannoxane.

According to the present invention, a suitable amount of monoaminoalcohol having 1 to 4 carbon atoms is present in the reaction system, whereby the polyurethane foam produced is free from cracking.

As the monoaminoalcohol, there may be counted, for example, 2-aminoethanol, 2-

aminopropan-1-ol, 1-aminopropan-2-ol, 2-aminobutan-1-ol, 1-aminopropan-3-ol and 3-aminobutan-1-ol among which 2-aminoethanol is most advantageous from a view point of practical use.

As the blowing agent, there may be used for example, water, compounds which produce water during the reaction, halogenated hydrocarbons having a relatively low boiling point such as dichlorodifluoromethane, trichloromonofluoromethane, or compounds which generate nitrogen gas during the reaction such as azo compounds.

The amount of the compound of the general formula (II) used as a catalyst in this invention is preferably about 0.05 to about 2.0, more preferably about 0.1 to about 1.5 parts by weight per 100 parts by weight of the polyol component.

The amount of the monoaminoalcohol which is used varies with the polyol employed and with reaction conditions, but it is preferably from about 0.1 to about 5, especially from about 0.3 to about 3.0 parts by weight per 100 parts by weight of the polyol component.

In carrying out the method of this invention, the sole use of the compound shown by the general formula (II) as a catalyst may be enough for giving polyurethane foam with desirable properties, but tertiary amines may if desired also be employed together with the compound of the general formula (II).

Suitable tertiary amines are for example, triethylamine and N,N,N',N'-tetramethyl-1,3-butanediamine.

In the method of this invention, the reaction may be carried out in the presence of a foam stabilizer, (e.g. silicone oil-polydimethyl siloxane, alkylsilane-poly-oxyalkylene copolymer) non-ionic surface active agents (e.g. sorbitan monostearate, glycerol monooleate, castor oil-ethylene oxide adduct), paints, additives, reinforcing materials, dyes, antioxidants or fireproofing agents.

The precise procedures for the production of the polyurethane foam in this invention may be similar to known methods for example, those described in "High Polymers, Vol. XVI Polyurethanes: Chemistry and Technology I, Chemistry" by Sanders Frisch (1962) published by Interscience Publishers.

The following examples illustrate the invention. In the Examples, "parts" means parts by weight unless other specified.

Parts by weight bear the same relation to parts by volume as do grams to milliliters.

Throughout the specification, the abbreviations "kg", "g", "cps", "m" and "cm" represent "kilograms", "grams", "centipoises", "meters" and "centimeters", respectively.

EXAMPLE 1

To 100 parts of poly caprolactone polyester polyol (OH number 55.5, acid number 0.6, white wax-like substance), which was obtained

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by reacting epsilon-caprolactone with trimethylol propane after the manner disclosed in U.S. Patent No. 2,890,208, were added, under vigorous stirring and heating at 45°C, 2.0 parts of castor oil-ethylene oxide adduct obtained by reacting ethylene oxide with castor oil (commercially available under the trade name "Newpole CZ 300" which is sold by Sanyo Chemical Industries Ltd., Japan), 0.5 part of 2,5-di-tert-amyl-hydroquinone, 3.0 parts of water, 1.5 part of 2-aminoethanol and 0.16 part of octabutyl-tetrauroxyloxy-stannoxane in that order.

The resulting mixture was then admixed with 46.5 parts of dimethylbenzene ω,ω' -diisocyanate (a mixture of 75 parts of 1,3-isomer and 25 parts of 1,4-isomer) under vigorous stirring, whereupon a reaction took place which yielded white flexible polyurethane foam.

Control 1

The same procedure as Example 1 was carried out without using 2-aminoethanol, using triethylenediamine (0.3 part) and stannous-2-ethylhexoate (0.7 part) in place of octabutyl - tetrauroxyloxy stannoxane (0.16 part) and using 42 parts of dimethylbenzene ω,ω' -diisocyanate. However, during the foaming reaction, the foam completely collapsed after being formed so that no foam product was obtained.

EXAMPLE 2

To 100 parts of polycaprolactone polyester polyol having the same properties as in Example 1 were added under vigorous stirring and heating at 45°C, 2.0 parts of "Newpole CZ 300", 0.5 part of 2,5-di-tert-amyl hydroquinone, 2.0 parts of water, 0.5 part of 2-aminoethanol and 2.0 parts of octabutyl-tetraacetoxy-stannoxane in that order.

The mixture was then admixed with 33.5 parts of dimethylbenzene ω,ω' -diisocyanate (a mixture of 70 parts of 1,3-isomer and 30 parts of 1,4-isomer) under vigorous stirring, whereupon a reaction took place which yielded white flexible polyurethane foam.

Control 2

The same procedure as in Example 2 was carried out without using 2-aminoethanol.

Polyurethane foam thus produced had large cracks therein.

EXAMPLE 3

To 100 parts of polycaprolactone polyester polyol (OH number 55.5, acid number 0.6, white wax-like substance), which was obtained by reacting epsilon-caprolactone and glycerol after the manner disclosed in U.S. Patent No. 2,890,208, were added, under vigorous stirring and heating at 45°C, 2.0 parts of "Newpole CZ 300", 0.5 part of 2,5-di-tert-amylhydroquinone, 2.5 parts of water, 1.2 part of 2-aminoethanol and 0.15 part of octabutyl-di-

chloro-dihydroxy-stannoxane in that order. The mixture was then admixed with 41.0 parts of dimethylbenzene ω,ω' -diisocyanate (a mixture of 78 parts of 1,3-isomer and 22 parts of 1,4-isomer) under vigorous stirring, whereupon a reaction took place which yielded white flexible polyurethane foam.

EXAMPLE 4

To 100 parts of polycaprolactone polyester polyol (OH number 56.0, acid number 0.47, white wax-like substance), which was obtained by reacting epsilon-caprolactone and a polyether polyol (product from glycerol and propylene oxide, OH number 160) after the manner disclosed in U.S. Patent No. 2,890,208, were added under vigorous stirring and heating at 45°C, 2.0 parts "Newpole CZ 300", 0.5 part of 2,5-di-tert-amylhydroquinone, 2.5 parts of water, 1.4 part of 2-aminoethanol and 0.17 part of octabutyl-tetraacetoxy-stannoxane in that order.

The mixture was then admixed with 42.0 parts of dimethylbenzene ω,ω' -diisocyanate (a mixture of 78 parts of 1,3-isomer and 22 parts of 1,4-isomer) under vigorous stirring, whereupon a reaction took place which yielded white flexible polyurethane foam.

EXAMPLE 5

100 Parts of polyester polyol (OH number 61, acid number 0.9, water content 0.02%, viscosity 1000 cps (75°C)), which was obtained by reacting adipic acid, diethylene glycol and trimethylol propane, were mixed homogeneously with 3.0 parts of water, 2.0 parts of castor oil-ethylene oxide adduct obtained by reacting ethylene oxide with castor oil (commercially available under the trade name "Nikkol CO 40" which is sold by Nikko Shokai Kabushiki Kaisha in Japan) and 0.5 part of 2,5-di-tert-amylhydroquinone. After being heated at 50°C, the mixture was admixed under vigorous stirring with 0.12 part of octabutyl - tetraacetoxy - stannoxane, 0.6 part of 2-aminoethanol and then with 43.8 parts dimethylbenzene ω,ω' -diisocyanate (a mixture of 75 parts of 1,3-isomer and 25 parts of 1,4-isomer), whereupon a reaction took place which yielded white flexible polyurethane foam.

EXAMPLE 6

100 parts of polyester polyol having the same properties as that used in Example 5 were homogeneously admixed with 2.5 parts of water, 2.0 parts of "Nikkol CO 40" and 0.5 part of 2,5-di-tert-amylhydroquinone.

After being heated at 50°C, the mixture was admixed homogeneously with 0.12 part of octabutyl-tetrauroxyloxy-stannoxane, 0.5 part of 2-aminoethanol and then with 38.5 parts dimethylbenzene ω,ω' -diisocyanate (a mixture of 75 parts of 1,3-isomer and 25 parts of 1,4-isomer), whereupon a reaction took

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place which yielded white flexible polyurethane foam.

EXAMPLE 7

- 5 To 100 parts of polycaprolactone polyester polyol having the same properties as one used in Example 3 where added, under vigorous stirring and heating at 45°C, 2.0 parts of "Newpole CZ 300", 0.5 part of 2,5-di-tert-amyl hydroquinone, 2.5 parts of water, 1.2
- 10 part of 2-aminoethanol and 0.15 part of octabutyl-tetraiscyanato-stannoxane in that order. The mixture was then admixed with 41.0 parts of dimethylbenzene ω, ω' -diisocyanate (a mixture of 78 parts of 1,3-isomer and 22
- 15 parts of 1,4-isomer) under vigorous stirring, whereupon a reaction took place which yielded white flexible polyurethane foam.

EXAMPLE 8

- 20 To 100 parts of polycaprolactone polyester polyol having the same properties as that used in Example 4 were added, under vigorous stirring and heating at 45°C, 2.0 parts of "Newpole CZ 300", 0.5 part of 2,5-di-tert-amylhydroquinone, 2.5 parts of water, 1.4
- 25 part of 2-aminoethanol and 0.17 part of octabutyl - diisothiocyano - diethoxy - stannoxane in that order. The mixture was then admixed with 42.0 parts of dimethylbenzene ω, ω' -diisocyanate (a mixture of 78 parts of
- 30 1,3-isomer and 22 parts of 1,4-isomer), under vigorous stirring, whereupon a reaction took place which yielded white flexible polyurethane foam.

EXAMPLE 9

- 35 100 parts of polyester polyol having the same properties as that in Example 5 were homogeneously admixed with 3.0 parts of water, 2.0 parts of "Nikkol CO 40" and 0.5 part of 2,5-di-tert-amylhydroquinone. After being heated at 45°C, the mixture was admixed homogeneously with 0.12 part of octa-
- 40 propyl-tetrabromo-stannoxane, 0.4 part of 2-aminoethanol, and then with 43.2 parts dimethylbenzene ω, ω' -diisocyanate (a mixture of 78 parts of 1,3-isomer and 22 parts of
- 45 1,4-isomer), whereupon a reaction took place which yielded white flexible polyurethane foam.

EXAMPLE 10

- 50 To 100 parts of polycaprolactone polyester polyol having the same properties as that used in Example 1 were added under vigorous stirring and heating at 45°C, with 2.0 parts of "Newpole CZ 300", 0.5 part of 2,5-di-tert-amylhydroquinone, 3.0 parts of water, 1.5
- 55 parts of 2-aminoethanol and 0.16 part of octabutyl-tetrachloro-stannoxane in this order. The mixture was then admixed with 45.9 parts of dimethylbenzene ω, ω' -diisocyanate (a mixture of 1,3-isomer and 25 parts of 1,4-isomer) under vigorous stirring, whereupon a reaction took place which yielded white flexible polyurethane foam.

EXAMPLE 11

- 65 100 parts of polyether triol (OH number 56) which was produced by addition polymerization of propylene oxide and glycerol, were admixed with 3.3 parts of dimethylbenzene ω, ω' -diisocyanate (a mixture of 70
- 70 parts of 1,3-isomer and 30 parts of 1,4-isomer) and 0.02 part of octabutyl-tetrauroxy-stannoxane, followed by heating at 70°C for about 1 hour to give isocyanate-modified polyether polyol which had OH number 35 and viscosity 3500 cps (25°C).
- 75 100 parts of the above prepared polyether polyol heated at 42°C were homogeneously admixed under vigorous stirring with 1.5 part of silicone oil, 0.5 part of 2,5-di-tert-amylhydroquinone, 1.0 part of polymeric phenol type antioxidant (commercially available under the trade mark "Topanol C.A." which is sold by Imperial Chemical Industries Ltd., in England), 3.0 parts of water, 1.2 part of 2-aminoethanol and 0.8 part of octabutyl-tetrauroxy-stannoxane.
- 80 The resulting mixture was then admixed under vigorous stirring with 40.5 parts of dimethylbenzene ω, ω' -diisocyanate (a mixture of 70 parts of 1,3-isomer and 30 parts of 1,4-isomer), whereupon a reaction took place which yielded white flexible polyurethane foam.
- 85 The properties of polyurethane foams obtained in the foregoing Examples and Controls were measured and the results are shown in Table 1.
- 90
- 95

TABLE 1
The mechanical properties of polyurethane foam

Example No.		*4 1	2	3	4	5	6	7	8	9	10	11	Control No.	
		1	2											
Density (kg/m ³)		28.4	45.0	34.6	34.1	28.0	33.0	34.4	34.2	28.5	28.2	33.5	foam product is not produced.	Cracking *occurs
Tensile strength (kg/cm ²)		1.18	1.40	1.17	1.15	1.0	1.05	1.16	1.15	1.05	1.20	0.85		
Elongation (%)		260	260	250	250	280	720	250	250	280	280	240		
Tear Strength (kg/m)		0.30	0.40	0.30	0.35	0.33	0.35	0.35	0.33	0.35	0.33	0.25		
Compression set (%)		14.8	3.7	4.8	4.0	10.0	12.5	4.8	4.2	10.0	10.0	7.5		
Indentation load deflection (kg) (JIS*1 K—6401 25% compress).		9.2	15.8	11.3	10.6	10.8	13.2	10.0	9.6	10.5	9.5	9.1		
Compression load (kg/cm ²)	25%	25.0	42.0	32.6	30.5	33.0	32.0	31.6	41.0	26.0	26.5	26.0		
	50%	30.4	60.2	46.8	41.2	40.0	39.8	45.4	58.8	31.5	32.1	32.0		
	65%	43.4	95.0	70.6	65.4	53.0	58.9	68.8	93.5	44.5	44.8	48.0		
Flex fatigue loss of height (%) (ASTM*2 D—1564 After 30 Min.)		2.1	1.6	1.7	1.5	1.7	1.5	1.6	1.6	2.0	2.0	2.3		
Chemical	against tri-chlorethylene	good												
Resistance	against 1,1,1-trichlorethane	good												
Heat resistance (120°C., 22 hours)		good												
Weathering resistance (after exposure to atmosphere for 3 months)		non-yellow												

*1 Japanese Industrial Standard

*2 American Society for Testing Materials

*3 Measurement of properties is impossible because of remarkable "cracking" occurring in foams.

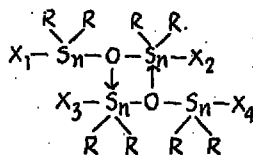
*4 Substantially same result is obtained by using 1.0 part of 3-aminobutan-1-ol in place of 1.5 part of 2-aminoethanol in the Example 1.

WHAT
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A process for the production of polyurethane foam comprising reacting dimethylbenzene ω, ω' -diisocyanate with an isocyanate-modified polyether polyol having a hydroxyl number of 25 to 75 or a polyester polyol having a hydroxyl number of 25 to 75 in the presence of a blowing agent, a compound of the formula:



wherein X_1 , X_2 , X_3 , and X_4 may be the same or different and represent halogen, —OH, alkoxy having not more than 4 carbon atoms, formyloxy, alkylcarbonyloxy having not more than 17 carbon atoms, —NCO and

2. A process as claimed in claim 1, wherein the monoaminoalcohol is 2-aminoethanol.

4. A process as claimed in any preceding claims, wherein the isocyanate-modified polyether polyol is one modified by dimethylbenzene ω,ω' -diisocyanate.

5. A process as claimed in any of claims 1 to 3, wherein the polyester polyol is polycaprolactone polyester polyol.

6. A process for the production of polyurethane foam substantially as described herein with reference to the Examples.

7. Polyurethane foam prepared by the process as claimed in any preceding claim.

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* C I M S *