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FORM 1
REGULATION 9

COMMONWEALTH OF AUSTRALIA

PATENTS ACT 1952-1973

APPLICATION FOR A PATENT

We BASF CORPORATION

of 1419 Biddle Avenue, Wandott, MICHIGAN, U.S.A.

hereby apply for the grant of a Patent for an invention entitled:

URETHANE COMPOSITION FOR STEERING WHEELS

which is described in the accompanying complete specification. This Application is a Convention Application and is based on the Application(s) numbered: 068,918 for a Patent or similar protection made in U.S.A. on 1 July 1987.

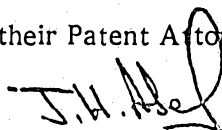
Our address for service is:

GRIFFITH HASSEL & FRAZER
71 YORK STREET
SYDNEY N.S.W. 2000
AUSTRALIA

DATED this 21st day of June, 1988.

BASF CORPORATION

By their Patent Attorneys



GRIFFITH HASSEL & FRAZER

TO: THE COMMISSIONER OF PATENTS
COMMONWEALTH OF AUSTRALIA
3648A:rk

5000471 21/06/88

APPLICATION ACCEPTED AND AMENDMENTS
15 - 8 - 90

FORM 8

COMMONWEALTH OF AUSTRALIA

PATENTS ACT 1952

DECLARATION IN SUPPORT OF AN APPLICATION FOR A PATENT

In support of an application made by: BASF CORPORATION

for a patent for an invention entitled: "URETHANE COMPOSITION FOR STEERING WHEELS"

I, William G. Conger
of BASF Corporation, 1419 Biddle, Wyandotte, Michigan 48192

do solemnly and sincerely declare as follows:

1. I am authorised by the above mentioned applicant for the patent to make this declaration on its behalf.
2. The name and address of each actual inventor of the invention is as follows:

Robert Atis MARKOVs of 8794 Manchester Lane, Grosse Ile, Michigan,
UNITED STATES OF AMERICA
Eric Robert SATTler of 13901 Greenwich Lane Apt. 205, Southgate,
Michigan, UNITED STATES OF AMERICA

and the fact upon which the applicant is entitled to make this application are as follows:

The applicant is the assignee of the invention from the inventors.

3. The basic application as defined by Section 141 of the Act was made as follows:

In UNITED STATES OF AMERICA on 1 July 1988
in the name ROBERT ATIS MARKOVs AND ERIC ROBERT SATTler

4. The basic application referred to in the preceding paragraph of this Declaration was the first applications made in a Convention country in respect of the invention the subject of this application.

Declared at Wyandotte, MI this 24th day of May 1990

Signed: William G. Conger

Position: Assistant Secretary, BASF Corporation

GRIFFITH HACK & CO, GPO BOX 4164, SYDNEY, NSW, 2001
AUSTRALIA

(12) PATENT ABRIDGMENT (11) Document No. AU-B-18221/88
(19) AUSTRALIAN PATENT OFFICE (10) Acceptance No. 603228

(54) Title
URETHANE COMPOSITION FOR STEERING WHEELS

International Patent Classification(s)
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C08G 018/63 C08L 075/04 C08L 075/06 C08L 075/08

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(71) Applicant(s)
BASF CORPORATION

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(74) Attorney or Agent
GRIFFITH HACK & CO., GPO Box 4164, SYDNEY NSW 2001

(56) Prior Art Documents
AU 39617/85 C08G 18/14 18/63
AU 38662/85 C08G 18/66 18/65

(57) Claim

1. A urethane composition consisting essentially of the reaction product of

(a) a mixture of a graft polymer dispersion in a polyol or in a polyol mixture,

(b) an organic polyisocyanate selected from the group consisting of an isocyanate prepolymer prepared by reacting 4,4'-diphenylmethane diisocyanate with a mixture of dipropylene glycol and a polyoxypropylene glycol having a molecular weight of about 420, a polymeric polymethylene polyphenylene polyisocyanate containing about 55 weight percent diphenylmethane diisocyanate, a 1:1 mixture of 2,4'- and 4,4'-diphenylmethane diisocyanate and mixtures thereof,

(c) a blowing agent,

(d) a chain extender, and

(e) a catalyst, surfactant and optionally flame retardants, fillers, ultraviolet absorbers and other additives.

5. A process for the preparation of an integral skin steering wheel which comprises pouring into a mold at a

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temperature of from 25°C. to 100°C. a urethane composition according to any one of claims 1 to 4, closing the mold and upon completion of the reaction, removing the integral skin steering wheel from said mold.

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COMMONWEALTH OF AUSTRALIA

PATENTS ACT 1952

Form 10

COMPLETE SPECIFICATION

FOR OFFICE USE

Short Title:

Int. Cl:

Application Number:
Lodged:

This document contains the
amendments made under
Section 49 and is correct for
printing.

Complete Specification-Lodged:

Accepted:

Lapsed:

Published:

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Related Art:

TO BE COMPLETED BY APPLICANT

Name of Applicant: BASF CORPORATION

Address of Applicant: 1419 Biddle Avenue, Wandott, MICHIGAN,
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Actual Inventor: Robert Atis Markovs and Eric Robert Sattler

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Complete Specification for the invention entitled:

URETHANE COMPOSITION FOR STEERING WHEELS

The following statement is a full description of this invention,
including the best method of performing it known to me/us:-

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Background of the Invention

1. Field of the Invention

The present invention relates to integral skin polyurethane steering wheels. More particularly, the invention relates to integral skin polyurethane steering wheels prepared from graft polymer dispersions. This process yields foams with improved physical properties as well as improved skin formation, surface appearance and processing characteristics.

10 2. Description of the Prior Art

The prior art generally teaches that graft polymer dispersions in polyols may be employed for a variety of polyurethane products. U.S. Patent 4,542,166 teaches that a steering wheel may be produced employing integral skin foam incorporating a polymer polyol and an aliphatic or alicyclic isocyanate in the presence of a polyol having a molecular weight of 62 to 300.



Summary of the Invention

According to the present invention there is provided
a urethane composition consisting essentially of the
reaction product of

- (a) a mixture of a graft polymer dispersion in a polyol or in a polyol mixture,
- (b) an organic polyisocyanate selected from the group consisting of an isocyanate prepolymer prepared by reacting 4,4'-diphenylmethane diisocyanate with a mixture of dipropylene glycol and a polyoxypropylene glycol having a molecular weight of about 420, a polymeric polymethylene polyphenylene polyisocyanate containing about 55 weight percent diphenylmethane diisocyanate, a 1:1 mixture of 2,4'- and 4,4'-diphenylmethane diisocyanate and mixtures thereof,
- (c) a blowing agent,
- (d) a chain extender, and
- (e) a catalyst, surfactant and optionally flame retardants, fillers, ultraviolet absorbers and other additives.

A preferred embodiment of the present invention will now be described by way of example only.

Description of the Preferred Embodiments

The urethane composition which is employed for integral skin steering wheels consists essentially of the reaction product of (a) a mixture of a graft polymer



dispersion in a polyol, or a polyol mixture (b) an organic polyisocyanate selected from the group consisting of an isocyanate prepolymer prepared by reacting 4,4'-diphenylmethane diisocyanate with a mixture of dipropylene glycol and a polyoxypropylene glycol having a molecular weight of about 420, a polymeric polymethylene polyphenylene polyisocyanate containing about 55 weight percent diphenylmethane diisocyanate, a 1:1 mixture of 2,4'- and 4,4'-diphenylmethane diisocyanate and mixtures thereof, (c) a blowing agent, (d) a chain extender, and (e) a catalyst, surfactant and optionally flame retardants, fillers U.V. absorbers and other additives.

The graft polymer dispersions which may be employed are prepared by polymerizing an ethylenically unsaturated monomer or mixture of monomers in either saturated or induced unsaturation polyols. The unsaturated polyols are preferred.

The polyols having induced unsaturation are hereinafter referred to as "macromers." Chain transfer agents may be employed as reaction moderators particularly at temperatures below 105°C. The polymerization reaction may be carried out at temperatures between 25°C and 180°C, preferably between 80°C and 135°C. The polyol mixture

contains less than 0.1 mole of unsaturation per mole of polyol mixture and ranges from 0.001 to 0.09 mole of unsaturation.

The graft polymer dispersions of this invention
5 have viscosities less than 10,000 cps at 25°C. Preferably they have viscosities ranging from 2000 to 8000 cps at 25°C.

Among those chain transfer agents which may be employed are as follows: acetic acid, bromoacetic acid, chloroacetic acid, ethyl dibromoacetate, iodoacetic acid,
10 tribromoacetic acid, ethyl tribromoacetate, trichloroacetic acid, ethyl trichloroacetate, acetone, p-bromophenylacetone, p-nitrophenylacetylene, allyl alcohol, 2,4,6-trinitroaniline, p-ethynylanisole, 2,4,6-trinitroanisole, azobenzene, benzaldehyde, p-cyanobenzaldehyde, 2-butyl-
15 benzene, bromobenzene, 1,3,5-trinitrobenzene, benzo-chrysene, ethyl trinitrobenzoate, benzoin, benzonitrile, benzopyrene, tributylborane, 1,4-butanediol, 3,4-epoxy-2-methyl-1-butene, t-butyl ether, t-butyl isocyanide, 1-phenylbutyne, p-cresol, p-bromocumene, dibenzonaphthacene, p-dioxane, pentaphenyl
20 ethane, ethanol, 1,1-diphenylethylene, ethylene glycol, ethyl ether, fluorene, N,N-dimethylformamide, 2-heptene, 2-hexene, isobutyraldehyde, diethyl bromomalonate, bromotrichloromethane, dibromoethane, diiodomethane, naphthalene, 1-

-- naphthol, 2-naphthol, methyl oleate, 2,4,4-triphenyl-1-pentene, 4-methyl-2-pentene, 2,6-diisopropylphenol, phenyl ether, phenylphosphine, diethylphosphine, dibutylphosphine, phosphorus trichloride, 1,1,1-tribromopropane, diallyl
5 phthalate, 1,2-propanediol, 3-phosphinopropionitrile, 1-propanol, pyrocatechol, pyrogallol, methyl stearate, tetraethylsilane, triethylsilane, dibromostilbene, α -bromostyrene, α -methylstyrene, tetraphenyl succinonitrile, 2,4,6-trinitrotoluene, p-toluidine, N,N-dimethyl-p-tolu-
10 idine, α -cyano-p-tolunitrile, α,α' -dibromo-p-xylene, 2,6-xylenol, diethyl zinc, dithiodiacetic acid, ethyl dithiodiacetic acid, 4,4'-dithio-bisanthranilic acid, benzenethiol, o-ethoxybenzenethiol, 2,2'-dithiobisbenzothiazole, benzyl sulfide, 1-dodecanethiol, ethanethiol, 1-hexanethiol, 1-
15 naphthalenethiol, 2-naphthalenethiol, 1-octanethiol, 1-heptanethiol, 2-octanethiol, 1-tetradecanethiol, α -toluenethiol, isopropanol, 2-butanol, carbon tetrabromide and tertiary dodecyl mercaptan.

The chain transfer agents employed will depend on
20 the particular monomers or mixtures of monomers employed and the molar ratios of such mixtures. The concentration of the chain transfer agent which is employed may range from 0.1 to 10 percent by weight based on the weight of monomer.

Representative polyols essentially free from ethylenic unsaturation which may be employed in the invention 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, equivalent weight of the polyols will vary from 100 to 10,000, preferably from 1000 to 3000.

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, 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-pentandiol, 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-
5 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
10 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
15 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
20 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

For example, combinations of polyoxypropylene and polyoxy-ethylene 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
5 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
10 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
15 thereof having equivalent weights of from 100 to 500.

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
20 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.

5 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
10 with alkylene oxides include alkanethiols containing at least two -SH groups such as 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.

15 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-
20 diaminotoluene; aliphatic amines such as methylamine, triisopropanolamine, ethylenediamine, 1,3-diaminopropane, 1,3-diaminobutane, and 1,4-diaminobutane.

Also, polyols containing ester group 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 discussion 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.

The unsaturated polyols or macromers which are employed in the present invention may be prepared by the reaction of any conventional polyol such as those described above with an organic compound having both ethylenic unsaturation and a hydroxyl, carboxyl, anhydride, isocyanate or epoxy group or they may be prepared by employing an organic compound having both ethylenic unsaturation and a hydroxyl, carboxyl, anhydride, or epoxy group as a reactant in the preparation of the conventional polyol. Representative of such organic compounds include unsaturated mono- and polycarboxylic acids and anhydrides such as maleic acid and anhydride, fumaric acid, crotonic acid and anhydride, propenyl succinic anhydride, acrylic acid, acryloyl chloride, hydroxy ethyl acrylate or methacrylate and halogenated maleic acids and anhydrides, unsaturated polyhydric alcohols

such as 2-butene-1,4-diol, glycerol allyl ether, trimethyl-
olpropane allyl ether, pentaerythritol allyl ether, penta-
erythritol vinyl ether, pentaerythritol diallyl ether, and
1-butene-3,4-diol, unsaturated epoxides such as 1-vinyl-
5 cyclohexene-3,4-epoxide, butadiene monoxide, vinyl glycidyl
ether(1-vinyloxy-2,3-epoxy propane), glycidyl methacrylate
and 3-allyloxypropylene oxide (allyl glycidyl ether). If a
polycarboxylic acid or anhydride is employed to incorporate
unsaturation into the polyols, it is preferable to react the
10 unsaturated polyol with an alkylene oxide, preferably
ethylene or propylene oxide, to replace the carboxyl groups
with hydroxyl groups prior to employment in the present
invention. The amount of alkylene oxide employed is such as
to reduce the acid number of the unsaturated polyol to about
15 5 or less.

In the event maleated macromers are employed, they
may be isomerized at temperatures ranging from 80°C to 120°C
for one-half hour to three hours in the presence of an
effective amount of an isomerization catalyst. The catalyst
20 is employed at concentrations greater than 0.01 weight
percent based on the weight of the macromer.

When preparing the polyetherester polyol employing
the catalyst selected from the group consisting of salts and

oxides of divalent metals, the concentration of catalyst which may be employed ranges from 0.005 to 0.5 weight percent based on the weight of polyol mixture. The temperatures employed range from 75°C to 175°C. The equivalent weight of the macromer may vary from 1000 to 10,000, preferably from 2000 to 6000.

Among the divalent metals which may be employed are: zinc acetate, zinc chloride, zinc oxide, zinc neodecanoate, tin chloride, calcium naphthenate, calcium chloride, calcium oxide, calcium acetate, copper naphthenate, cadmium acetate, cadmium chloride, nickel chloride, manganese chloride, and manganese acetate.

Certain of the above-mentioned catalysts such as calcium naphthenate promote the isomerization of the maleate to the fumarate structure during the preparation of the macromer, while others such as zinc chloride, which is an effective catalyst for the polymerization, inhibit this isomerization.

As mentioned above, the graft polymer dispersions of the invention are prepared by the in situ polymerization, in the above-described polyols of an ethylenically unsaturated monomer or a mixture of ethylenically unsaturated monomers. Representative ethylenically unsaturated monomers

which may be employed in the present invention include butadiene, isoprene, 1,4-pentadiene, 1,6-hexadiene, 1,7-octadiene, styrene, α -methylstyrene, 2-methylstyrene, 3-methylstyrene and 4-methylstyrene, 2,4-dimethylstyrene, 5 ethylstyrene, isopropylstyrene, butylstyrene, phenylstyrene, cyclohexylstyrene, benzylstyrene, and the like; substituted styrenes such as cyanostyrene, nitrostyrene, N,N-dimethylaminostyrene, acetoxystyrene, methyl 4-vinylbenzoate, phenoxystyrene, p-vinylphenyl oxide, and the like; the 10 acrylic and substituted acrylic monomers such as acrylonitrile, acrylic acid, methacrylic acid, methyl acrylate, 2-hydroxyethyl acrylate, methyl methacrylate, cyclohexyl methacrylate, benzyl methacrylate, isopropyl methacrylate, octyl methacrylate, methacrylonitrile, ethyl α -ethoxy- 15 acrylate, methyl α -acetaminoacrylate, butyl acrylate, 2-ethylhexyl acrylate, phenyl acrylate, phenyl methacrylate, N,N-dimethylacrylamide, N,N-dibenzylacrylamide, N-butylacrylamide, methacrylyl formamide, and the like; the vinyl esters, vinyl ethers, vinyl ketones, etc., such as vinyl 20 acetate, vinyl butyrate, isopropenyl acetate, vinyl formate, vinyl acrylate, vinyl methacrylate, vinyl methoxyacetate, vinyl benzoate, vinyltoluene, vinyl naphthalene, vinyl methyl ether, vinyl ethyl ether, vinyl propyl ethers, vinyl butyl

-ethers, vinyl 2-ethylhexyl ether, vinyl phenyl ether, vinyl 2-methoxyethyl ether, methoxybutadiene, vinyl 2-butoxyethyl ether, 3,4-dihydro-1,2-pyran, 2-butoxy-2'-vinylloxy diethyl ether, vinyl methyl ketone, vinyl ethyl ketone, vinyl
5 phosphonates such as vinyl phenyl ketone, vinyl ethyl sulfone, N-methyl-N-vinyl acetamide, N-vinyl-pyrrolidone, vinyl imidazole, divinyl sulfoxide, divinyl sulfone, sodium vinylsulfonate, methyl vinylsulfonate, N-vinyl pyrrole, and the like; dimethyl fumarate, dimethyl maleate, maleic acid,
10 crotonic acid, fumaric acid, itaconic acid, monomethyl itaconate, t-butylaminoethyl methacrylate, dimethylaminoethyl methacrylate, glycidyl acrylate, allyl alcohol, glycol monoesters of itaconic acid, vinyl pyridine, and the like.
Any of the known polymerizable monomers can be used and the
15 compounds listed above are illustrative and not restrictive of the monomers suitable for use in this invention.
Preferably, the monomer is selected from the group consisting of acrylonitrile, styrene and mixtures thereof.

The amount of ethylenically unsaturated monomer
20 employed in the polymerization reaction is generally from 25 percent to 60 percent, preferably from 30 percent to 45 percent, based on the total weight of the product. The polymerization occurs at a temperature between about 25°C

and 180°C, preferably from 80°C to 135°C.

Illustrative polymerization initiators which may be employed are the well-known free radical types of vinyl polymerization initiators such as the peroxides, per-

5 sulfates, perborates, percarbonates, azo compounds, etc. These include hydrogen peroxide, dibenzoyl peroxide, acetyl peroxide, benzoyl hydroperoxide, t-butyl hydroperoxide, di-t-butyl peroxide, lauroyl peroxide, butyryl peroxide, diisopropylbenzene hydroperoxide, cumene hydroperoxide,

10 paramenthane hydroperoxide, diacetyl peroxide, di- α -cumyl peroxide, dipropyl peroxide, diisopropyl peroxide, isopropyl-t-butyl peroxide, butyl-t-butyl peroxide, difuroyl peroxide, bis(triphenylmethyl) peroxide, bis(p-methoxybenzoyl) peroxide, p-monomethoxybenzoyl peroxide, rubene peroxide,

15 ascaridol, t-butyl peroxybenzoate, diethyl peroxyterephthalate, propyl hydroperoxide, isopropyl hydroperoxide, n-butyl hydroperoxide, t-butyl hydroperoxide, cyclohexyl hydroperoxide, trans-decalin hydroperoxide, α -methylbenzyl hydroperoxide, α -methyl- α -ethyl benzyl hydroperoxide, tetralin

20 hydroperoxide, triphenylmethyl hydroperoxide, diphenylmethyl hydroperoxide, α, α' -azobis-(2-methyl heptonitrile), 1,1'-

-azo-bis(cyclohexane carbonitrile), 4,4'-azobis(4-cyanopentanoic acid), 2,2'-azobis(isobutyronitrile), 1-t-butylazo-1-cyanocyclohexane, persuccinic acid, diisopropyl peroxy dicarbonate, 2,2'-azobis(2,4-dimethylvaleronitrile),
5 2-t-butylazo-2-cyano-4-methoxy-4-methylpentane, 2,2'-azobis-2-methylbutanenitrile, 2-t-butylazo-2-cyanobutane, 1-t-amylazo-1-cyanocyclohexane, 2,2'-azobis(2,4-dimethyl-4-methoxyvaleronitrile, 2,2'-azobis-2-methyl-butyronitrile, 2-t-butylazo-2-cyano-4-methylpentane, 2-t-butylazo-2-iso-
10 butyronitrile, to butylperoxyisopropyl carbonate and the like; a mixture of initiators may also be used. The preferred initiators are 2,2'-azobis(2-methylbutyronitrile), 2,2'-azobis(isobutyronitrile), 2,2'-azobis(2,4-dimethylvaleronitrile), 2-t-butylazo-2-cyano-4-methoxy-4-methyl-
15 pentane, 2-t-butylazo-2-cyano-4-methylpentane, 2-t-butylazo-2-cyano-butane and lauroyl peroxide. Generally, from about 0.1 percent to about 10 percent, preferably from about 1 percent to about 4 percent, by weight of initiator based on the weight of the monomer will be employed in the process of
20 the invention.

The integral skin polyurethane foams employed in the present invention are generally prepared by the reaction of a graft polymer dispersion 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
5 for the preparation of cellular polyurethane plastics 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
10 produce carbon dioxide may be used. It is possible to proceed with the preparation of the polyurethane plastics 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
15 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
20 boiling hydrocarbons such as pentane, hexane, heptane, pentene, and heptene; azo compounds such as azohexahydrobenzodinitrile; halogenated hydrocarbons such as dichlorodifluoromethane, trichlorofluoromethane, dichlorodifluoro-

ethane, 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'-dimethyl-15 diphenylmethane-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 poly-20 phenylene polyisocyanate. Especially useful due to their availability and properties are toluene diisocyanate, 4,4'-diphenylmethane diisocyanate and polymethylene polyphenylene polyisocyanate.

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
5 obtained by the phosgenation of crude diphenylmethane diamine. The preferred or crude isocyanates are disclosed in U.S. Patent No. 3,215,652.

As mentioned above, the graft polyols may be employed along with another polyhydroxyl-containing compo-
10 nent commonly employed in the art. Any of the polyhydroxyl-containing components which are described above for use in the preparation of the graft polyols may be employed in the preparation of the polyurethane foams useful in the present invention.

15 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
20 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-(methylanine), ethylenediamine, diethylenetriamine, N-(2-hydroxypropyl)-ethylenediamine, N,N'-di(2-hydroxypropyl)ethylenediamine, piperazine, and 2-methylpiperazine.

5 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
10 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.
15 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
20 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.

Among the flame retardants which may be employed are: pentabromodiphenyl oxide, dibromopropanol, tris(β -chloropropyl)phosphate, 2,2-bis(bromoethyl) 1,3-propanediol, tetrakis(2-chloroethyl)ethylene diphosphate, tris(2,3-dibromopropyl)phosphate, tris(β -chloroethyl)phosphate, tris(1,2-dichloropropyl)phosphate, bis-(2-chloroethyl) 2-chloroethylphosphonate, molybdenum trioxide, ammonium molybdate, ammonium phosphate, pentabromodiphenyloxide, tricresyl phosphate, hexabromocyclododecane and dibromoethyl- dibromocyclohexane. The concentrations of flame retardant compounds which may be employed range from 5 to 25 parts per 100 parts of polyol mixture.

Ultraviolet absorbers may also be employed. These include benzotriazoles such as Tinuvin P, Tinuvin 327 and Tinuvin 328 produced by Ciba-Geigy Corporation, benzophenols such as Tomisopl 800 produced by Yashitomi Pharmaceutical Industries, Ltd. and hindered amines such as Sanol LS-770, Sanol LS-744 and Tinuvin 144, Ciba-Geigy Corporation.

Polyol A is an ethylene oxide propylene oxide adduct of trimethylolpropane containing 13 percent ethylene oxide and having a hydroxyl number of about 35.

Polyol B is Polyol A containing a 31 weight percent 1:1 acrylonitrile:styrene polymer dispersion having a hydroxyl number of about 24.

DABCO S-25 is a 25 weight percent solution of triethylenediamine in butanediol.

Fomrez UL-1 is a metal catalyst sold by Witco Chemical Corporation.

F-11A is a fluorocarbon blowing agent sold by duPont Corporation.

I-460 is similar to DABCO S-25.

BDO is 1,4-butanediol.

Isocyanate A is 60 percent isocyanate prepolymer prepared by reacting 4,4'-diphenylmethane diisocyanate with a mixture of dipropylene glycol and a polyoxypropylene glycol, molecular weight 420, 20 percent polymeric polymethylene polyphenylene polyisocyanate containing about 55 percent two-ring and 20 percent of a 1:1 mixture of 2,4'- and 4,4'-diphenylmethane diisocyanate.

Isocyanate B is an isocyanate prepolymer prepared by reacting 4,4'-diphenylmethane diisocyanate with a mixture of dipropylene glycol and a polyoxypropylene glycol having a molecular weight of about 420.

5

Examples 1-5

Procedure

The indicated quantities of the ingredients employed were blended together at room temperature and poured into steering wheel molds. The molds were closed and
10 after the reaction had been completed, the finished steering wheels were removed from the molds. The wheels prepared employing a graft polymer dispersion had improved skin formation and improved surface appearance.

TABLE I

<u>Examples</u>		<u>1</u>	<u>2</u>	<u>3</u>
<u>Formulation, pbw</u>				
5	Polyol A	82.6	57.6	41.3
	Polyol B	-	25.0	41.3
	BDO	5.6	5.6	5.6
	Glycerin	0.4	0.4	0.4
	DABCO S-25 (I-460)	1.9	1.9	1.9
10	Fomrez UL-1	0.002	0.002	0.002
	H ₂ O	0.05	0.05	0.05
	F-11A	9.5	9.5	9.5
	Isocyanate A/100 parts resin	37.0	35.5	35.0
<u>Physical Properties</u>				
15	Tensile (kPa)	1241	1575	2135
	Elongation (%)	110	105.5	120
	Split Tear (N/M)	2802	2329	2486
	Density (Kg/M ³)	485	564	572
20	ILD (kg)			
	25%	-	17.2	24.7
	65%	-	83.2	106.6
	25% Return	-	11.3	14.5
	Sag Factor	-	4.8	4.3
25	% Recovery	-	4.8	4.3
	CLD (kPa)			
	25%	57.9	195.1	293
	50%	101.4	809.5	1126
30	25% Return	-	148.2	176
	Compression Set (%)			
30	@ 50%			
	22 hrs., 69°C	-	18.4	18.2
Shore A		47-45	50-44	57-50

TABLE I (continued)

<u>Examples</u>		<u>4</u>	<u>5</u>
<u>Formulation, pbw</u>			
	Polyol A	57.6	41.3
5	Polyol B	25.0	41.3
	BDO	5.6	5.6
	Glycerin	0.4	0.4
	DABCO S-25 (I-460)	1.9	1.9
	Fomrez UL-1	0.002	0.002
10	H ₂ O	0.05	0.05
	F-11A	9.5	9.5
	Isocyanate B/100 parts resin	40.9	40.4
<u>Physical Properties</u>			
	Density (Kg/m ³)	545	559
15	Tensile (kPa)	2661	3142
	Elongation (%)	160.5	149.0
	Split Tear (N/M)	3660	4132
	ILD (kg)		
	25%	32.0	37.9
20	65%	123.8	178.9
	25% Return	17.5	19.5
	Sag Factor	3.8	4.7
	CLD (kPa)		
	25%	324.8	400.6
25	50%	1415.5	1708.6
	25% Return	186.9	221
	Compression Set (%)		
	24 hrs., 69°C	26.5	26.6
	Shore A	64-59	73-67

The claims defining the invention are as follows:

1. A urethane composition consisting essentially of the reaction product of

(a) a mixture of a graft polymer dispersion in a polyol or in a polyol mixture,

(b) an organic polyisocyanate selected from the group consisting of an isocyanate prepolymer prepared by reacting 4,4'-diphenylmethane diisocyanate with a mixture of dipropylene glycol and a polyoxypropylene glycol having a molecular weight of about 420, a polymeric polymethylene polyphenylene polyisocyanate containing about 55 weight percent diphenylmethane diisocyanate, a 1:1 mixture of 2,4'- and 4,4'-diphenylmethane diisocyanate and mixtures thereof,

(c) a blowing agent,

(d) a chain extender, and

(e) a catalyst, surfactant and optionally flame retardants, fillers, ultraviolet absorbers and other additives.

2. A composition according to claim 1, wherein the polyol is an ethylene oxide propylene oxide adduct of trimethylolpropane containing about 13 percent ethylene oxide and having a hydroxyl number of about 35.

3. A composition according to claim 2, wherein the graft polymer dispersion consists of about a 1:1, 30 percent dispersion of acrylonitrile:styrene in the polyol.

4. A composition according to any one of the preceding claims, which has been molded in the form of a steering wheel.

5. A process for the preparation of an integral skin steering wheel which comprises pouring into a mold at a temperature of from 25°C. to 100°C. a urethane composition according to any one of claims 1 to 4, closing the mold and upon completion of the reaction, removing the integral skin steering wheel from said mold.



6. A process for the preparation of an integral skin steering wheel substantially as hereinbefore described with reference to the Examples.

Dated this 21st day of June 1988

BASF CORPORATION
By their Patent Attorney
GRIFFITH HASSEL & FRAZER