

5 INTERFACIAL POLYCARBONATE POLYMERIZATION PROCESS AND PRODUCT

FIELD OF INVENTION

The present invention relates to copolymer and a process
10 for preparing an aromatic carbonate copolymer by employing
an interfacial polymerization reaction utilizing an aliphatic diol
monomer prepared prior to the interfacial polymerization
reaction thereof with an aromatic dihydroxy compound. The
resulting copolymer incorporates the aliphatic diol in the
15 polymer chain. The copolymer consists of alternating units of
the residue of the reaction of the aromatic dihydroxy
compound and the aliphatic diol.

BACKGROUND OF THE INVENTION

It is known to prepare polymers from certain
20 cyclobutanediols and aromatic dihydroxy compounds such as
2,2-bis(4-hydroxyphenyl) propane, commonly referred as
bisphenol -A (BPA). The polymers prepared therefrom employ

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☒	Claim(s) : paged 15 ¹⁵ to paged 17 ¹⁷	
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solvents and high temperatures. U.S. Patents 3,326,855;
3,313,777; 3,546,331; 3,546,332; 3,541,200; 3,375,210 and 3,674,623
disclose polymers prepared from tertrahalobutanediol and
particularly tetramethylcyclobutanediol (TMCBD). As disclosed
5 therein, these TMCBD, for example, can be reacted with other
reactants such as aryl dihydroxy compounds usually at elevated
temperatures and in the presence of organic solvents. Several
of the references are also directed to methods of surface
crystallization of copolymers prepared from TMCBD and
10 dihydroxy co-reactants. The reactions disclosed therein do not
employ aliphatic haloformates, which in the instant invention
are prepared prior to the reaction thereof with an aryl
dihydroxy reactant. Otherwise, if an aromatic haloformate is
produced instead of forming an aliphatic haloformate, the
15 reaction with an aliphatic diol will not produce a polymer of
alternating aliphatic and aromatic species.

SUMMARY OF THE INVENTION

The process of this invention has been developed to
produce aliphatic-aromatic co-polycarbonates from their
20 respective aromatic dihydroxy and aliphatic diol monomer
components. The process is an interfacial reaction but modified
to produce the resulting co-polycarbonate. Generally, the

reaction of this invention involves the haloformate of an aliphatic diol such as 2,2,4,4-tetramethylcyclobutandiol and bisphenol -A (BPA). The reaction of this invention first requires forming an aliphatic haloformate as will be subsequently disclosed herein. Otherwise, the reaction will not result in the formation of a copolymer of alternating aliphatic and aromatic units.

DETAILED DESCRIPTION OF THE INVENTION

This invention relates to a novel copolymer comprised of aliphatic and aromatic units in the polymer chain and to a process of reacting an aliphatic haloformate and an aromatic dihydroxy reactant in water and an organic phase at ambient temperature and pressure. The aliphatic haloformate is prepared by reacting a certain aliphatic diol such as an aliphatic alkyl diol and include cycloaliphatic, straight and branched chain, diols free of substitute groups which would be reactive with other reactants employed in the reaction system of this invention. The aliphatic diol is preferably a tetraalkylcyclobutanediol. The alkyl groups are those hydrocarbon groups of up to about 8 carbon atoms such as methyl, ethyl, propyl, isopropyl, butyl, isobutyl, t-butyl, pentyl, hexyl, heptyl, 2-ethylhexyl, isooctyl, octyl and the like.

The cycloalkyl group contains preferably 4 to 6 carbon atoms although up to 12 carbon atoms can be used. The cycloalkyl group may be unsubstituted or substituted by a hydrocarbon, a halogen, or the like group, providing that the substituents are not reactive with the other reactants of the reaction system of this invention. Examples of such cycloalkyls include cyclobutyl, 2,2,4,4-tetramethylcyclobutyl, cyclopentyl, cyclohexyl, methylcyclohexyl groups and the like. The preferred aliphatic diol is 2,2,4,4-tetramethylcyclobutanediol-1,3.

10 The haloformate of the aliphatic diol is formed by reacting an aliphatic diol with a carbonate precursor, preferably a halocarbonyl precursor and more particularly a carbonyl chloride such as phosgen. When employing phosgen, the preferred intermediate reactant formed is 2,2,4,4-tetramethyl-1,3-cyclobutane bischloroformate (TMCBOCI).

A critical feature of this invention is that the haloformate of the aliphatic diol must be formed prior to the reaction thereof with an aromatic dihydroxy diol. If the haloformate of the aliphatic diol is not first formed, then the reaction may result in a copolymer that is not an alternating i.e. 1:1, aliphatic and aromatic units. In addition, another feature of this

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invention is that the reaction is carried out at a high pH about 8-14 and preferably about 10 to 14.

As stated previously, the other reactant of this process is an aromatic dihydroxy reactant such as an aromatic dihydric phenol. The dihydric phenols include, but are not limited thereto, 2,2-bis(4-hydroxyphenyl) propane; 2,4'-dihydroxydiphenylmethane; bis(2-hydroxyphenyl) methane; bis(4-hydroxyphenyl) methane; bis(4-hydroxy-5-nitrophenyl) methane; bis(4-hydroxy-2,6-dimethyl-3-methoxyphenyl) methane; 1,1-bis(4-hydroxyphenyl) ethane; 1,2-bis(4-hydroxyphenyl) ethane; 1,1-bis(4-hydroxy-2-chlorophenyl) ethane; 1,1-bis(2,5-dimethyl-4-hydroxyphenyl) ethane; 1,3-bis(3-methyl-4-hydroxyphenyl) propane; 2,2-bis(3-phenyl-4-hydroxyphenyl) propane; 2,2-bis(3-isopropyl-4-hydroxyphenyl) propane; 2,2-bis(4-hydroxynaphthyl) propane; 2,2-bis(4-hydroxyphenyl) pentane; 3,3-bis(4-hydroxyphenyl) pentane; 2,2-bis(4-hydroxyphenyl) heptane; bis(4-hydroxyphenyl) phenylmethane; bis(4-hydroxyphenyl) cyclohexylmethane; 1,2-bis(4-hydroxyphenyl)-1,2-bis(phenyl) propane; 2,2-bis(4-hydroxyphenyl)-1-phenylpropane; the bisphenol of 4-vinyl cyclohexane; spiro buildane bisphenol; and the like. Also included are dihydroxybenzenes typified by the hydroquinone

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and resorcinol, dihydroxydiphenyls such as 4,4'-dihydroxydiphenyl; 2,2'-dihydroxydiphenyl; 2,4'-dihydroxydiphenyl; dihydroxynaphthalenes such as 2,6-dihydroxynaphthalene, and the like, bis-(4-hydroxyphenyl) sulfone; 2,4'-dihydroxydiphenyl sulfone; 5'-chloro-2,4'-dihydroxydiphenyl sulfone; 5'-chloro-2,4'-dihydroxydiphenyl sulfone; 3'-chloro-4,4'-dihydroxydiphenyl sulfone; 4,4'-dihydroxytriphenyl disulfone, etc.; 4,4'-dihydroxydiphenyl ether; 4,4'-dihydroxytriphenyl ether; the 4,3'-, 4,2'-, 4,1'-, 2,2'-, 2,3'-, etc.;

10 dihydroxydiphenyl ethers; 4,4'-dihydroxy-2,6-dimethyldiphenyl ether; 4,4'-dihydroxy-2,5-dimethyldiphenyl ether; 4,4'-dihydroxy-3,3'-dissobutyldiphenyl ether; 4,4'-dihydroxy-3,3'-dissopropyldiphenyl ether; 4,4'-dihydroxy-3,2'-dinitrodiphenyl ether; 4,4'-dihydroxy-3,3'-dichlorodiphenyl ether; 4,4'-dihydroxy-

15 3,3'-difluorodiphenyl ether; 4,4'-dihydroxy-2,3-dibromodiphenyl ether; 4,4'-dihydroxydinaphthyl ether; 4,4'-dihydroxy-3,3'-dichlorodinaphthyl ether; 2,4-dihydroxytetraphenyl ether; 4,4'-dihydroxypentaphenyl ether; 4,4'-dihydroxy-2,6-dimethoxydiphenyl ether; 4,4'-dihydroxy-2,5-diethoxydiphenyl

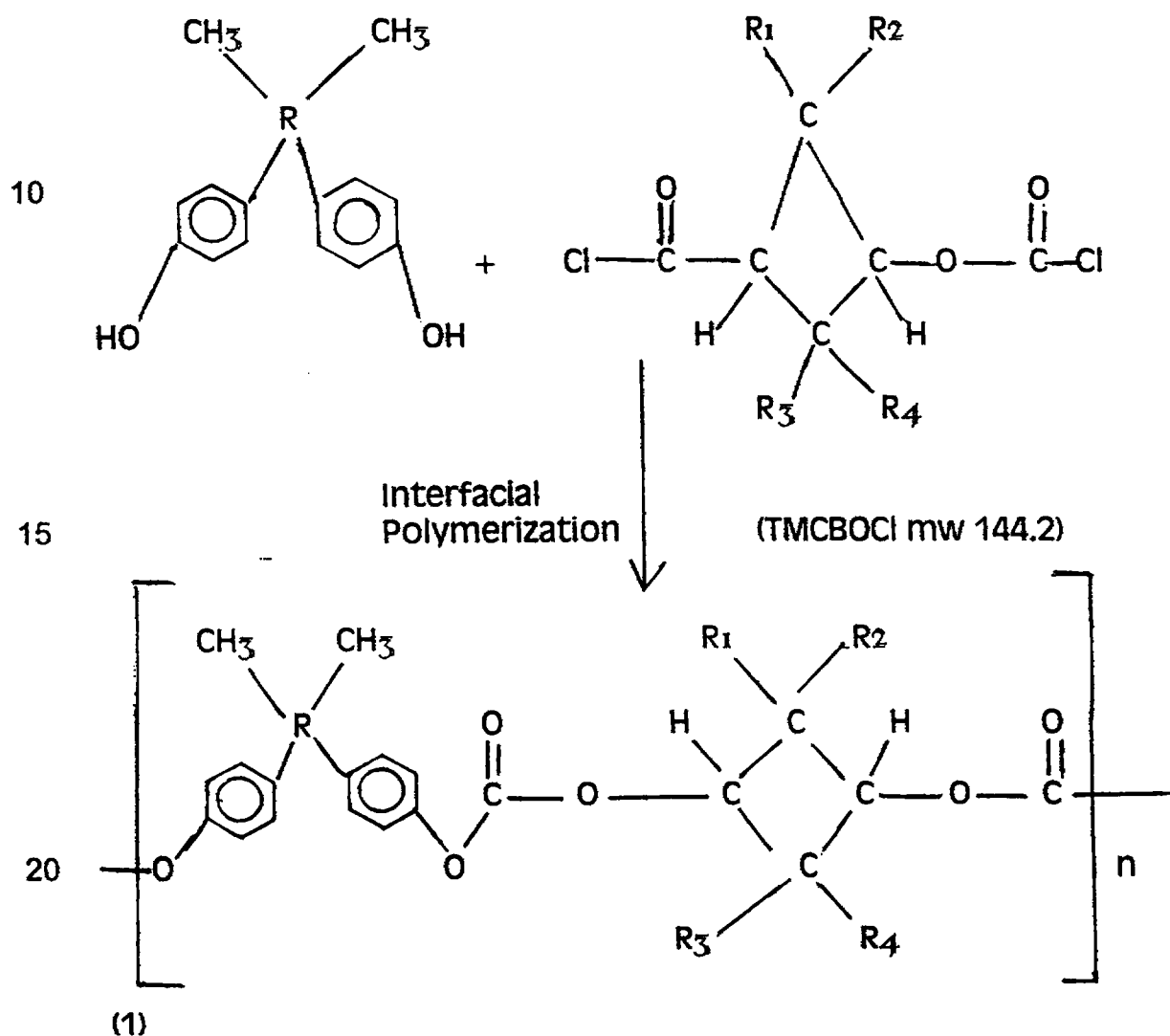
20 ether, etc. Mixtures of the dihydric phenols can also be employed and where dihydric phenol is mentioned herein, mixtures of such materials are considered to be included.

Preferably the dihydric phenol is a bis(4-hydroxyphenyl) alkane in which the central alkane group contains from 1-8 carbon atoms. Especially preferred dihydric phenols are gem-bis(4-hydroxyphenyl) alkanes in which the central alkylidene group contains from 1-8 carbon atoms. The most preferred dihydric phenol is 2,2-bis(4-hydroxyphenyl) propane (BPA).

The carbonate polymer produced by the process of this invention is a polymer consisting of the residue of the reaction of an aromatic dihydroxy and an aliphatic diol. The residue of the reaction is a copolymer having a ratio of aromatic units to aliphatic units of 1:0.5 to 1:1.5. Preferably, the carbonate polymer consists essentially of a ratio of 1:1 aromatic units to aliphatic units. Through the novel discovery of this invention, aliphatic diols, in particular tetramethylcyclobutanediol (TMCBD) can be incorporated, in aromatic polycarbonates by an interfacial polymerization process. The interfacial reaction process of this invention can also result in the formation of copolycarbonates, ter-polycarbonates and polyester-polycarbonates.

In general, the polycarbonates of this invention can be produced from the polymerization of aliphatic haloformates in the presence of an aromatic dihydroxy reactant such as BPA.

Polymerization occurs at ambient temperatures and pressure under relatively high pH condition of 8-14 and preferably 10-14. A catalyst is employed to promote the reaction and high yields may be obtained in 5 minutes of increasing the pH of the reaction. Catalysts that may be employed herein are preferably amine catalyst and particularly triethylamine (TEA). The general synthesis of the carbonate polymer may be as follows:



wherein R, R₁, R₂, R₃ and R₄ are independently selected from alkyl, aryl or alkylaryl hydrocarbon groups of 1 to 20 carbon atoms and preferably 3-10 carbons atoms, and n is an integer of 5 to 200. R is preferably isopropylidene but may also be acetophenone (bis AP) or spirobiindane (SBI). TMCBOCI is the bischloroformate of tetramethylcyclobutanediol (TMCBD). The reaction can also proceed with two or more aromatic dihydroxy compounds such as isopropylidene and bis AP or bis AP and SBI or even isopropylidene, bis AP and SBI.

10 **DETAILED DESCRIPTION OF THE EXAMPLES**

The following examples further exemplify this invention. However, the invention is not limited by these examples.

General Reaction

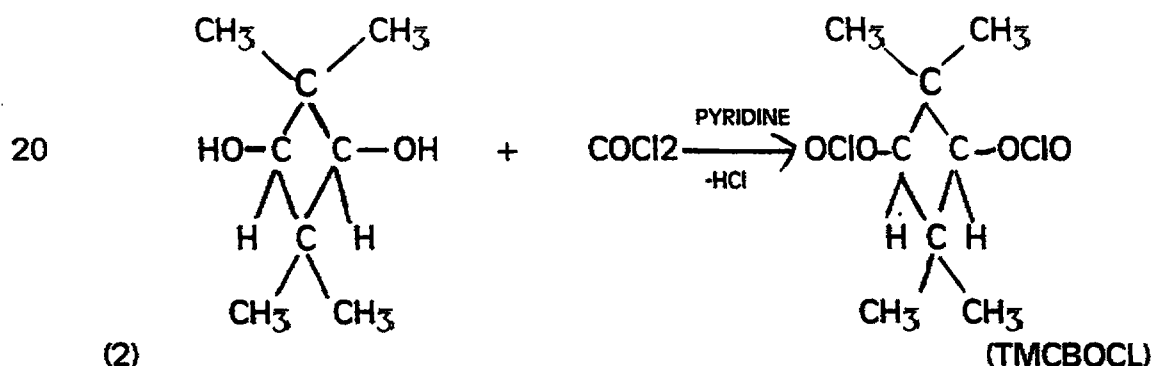
The copolymers are produced by the reaction of the bischloroformate of tetramethylcyclobutanediol (TMCBOCI) in the presence of bisphenol-A. Polymerization is carried out at ambient temperatures and pressures and at a pH of about 12. A catalytic amount of triethylamine is employed to promote the reaction.

20 **EXAMPLE 1**

Formation of 2,2,4,4-tetramethyl-1,3-cyclobutane bischloroformate (TMCBOCI)

To a 1000 ml reactor fitted with a dewer condenser with dichloromethane (DCM)/acetone bath, a nitrogen/phosgene feed, and a mechanical stirrer, there was charged 400 mls of DCM and 100 g tetramethylcyclobutanediol (TMCBD). This was placed in a dichloromethane (DCM) dry ice bath. The mixture was stirred under a nitrogen blanket to disperse the TMCBD. About 150 g of phosgene (1.5 mol) was then fed into the reactor at the rate of 2 g/min. 115 ml of pyridine (in 100 mls DCM) was dripped into the reactor at about 20 ml/min. The reaction did not appear to be exothermic. The reaction was run for about 2 hours and then was warmed to room temperature. The mixture was extracted into 5 L of hexane, evaporated down to 1 liter, placed in a freezer and crystals which formed were collected.

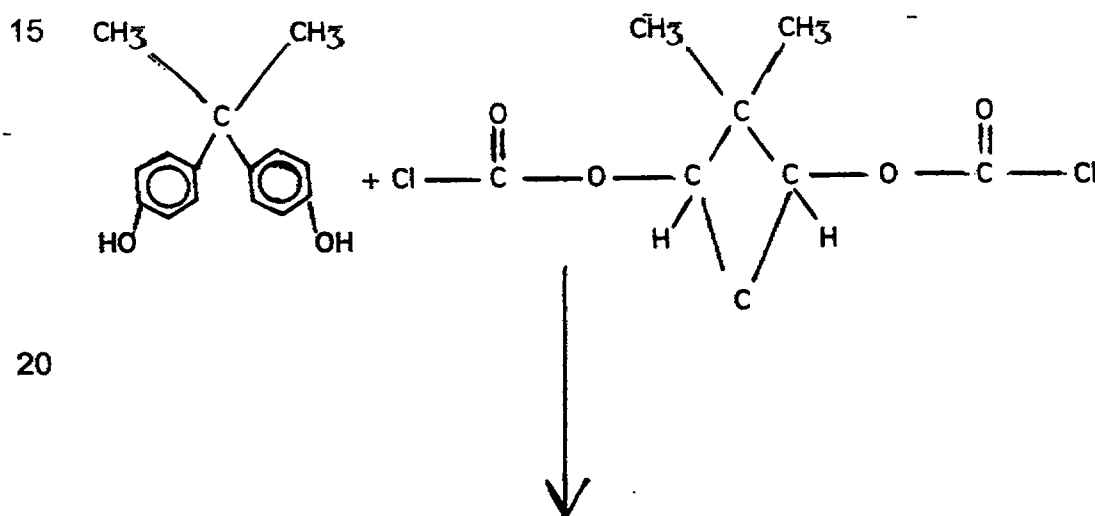
Nuclear Magnetic Resonance (NMR) confirmed the production of the bischloroformate. The yield was about 75%.

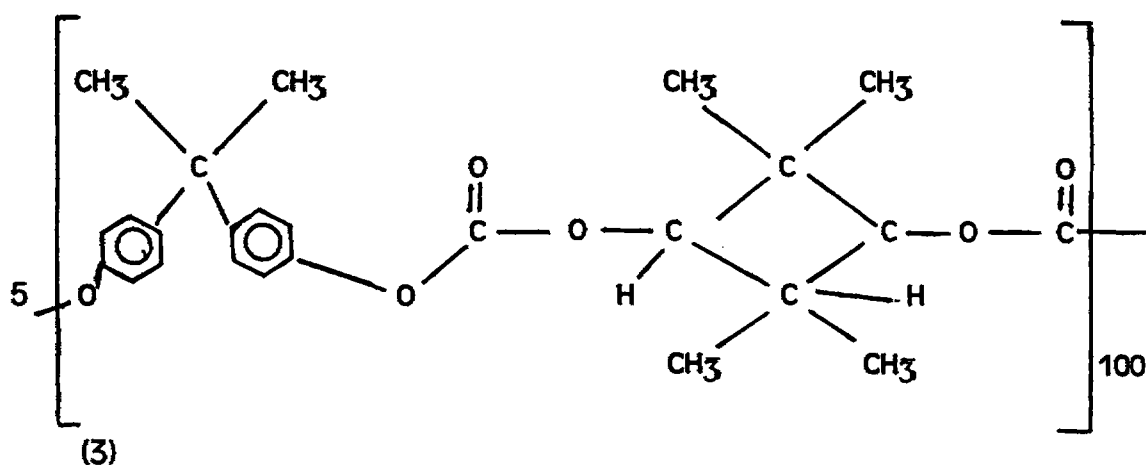


EXAMPLE 2

To a 125 ml flask reactor, 1.77 gms of TMCBOCl prepared in Example 1 above, 1.66 gms of bisphenol A, 50 ml of dichloromethane, 0.2 mls of triethylamine and 25 ml of water were charged. 25 mls of 10% aqueous sodium hydroxide (NaOH) solution was slowly added. The reaction was run for about 15 minutes. The organic layer was then decanted and washed three times in 50 mls of a 10% aqueous hydrochloride (HCl) solution. The organic layer was then poured in 500 mls of methanol and the precipitated polymer was collected and dried under a vacuum.

The yield was over 90% and the polymer was confirmed by NMR.

Reaction

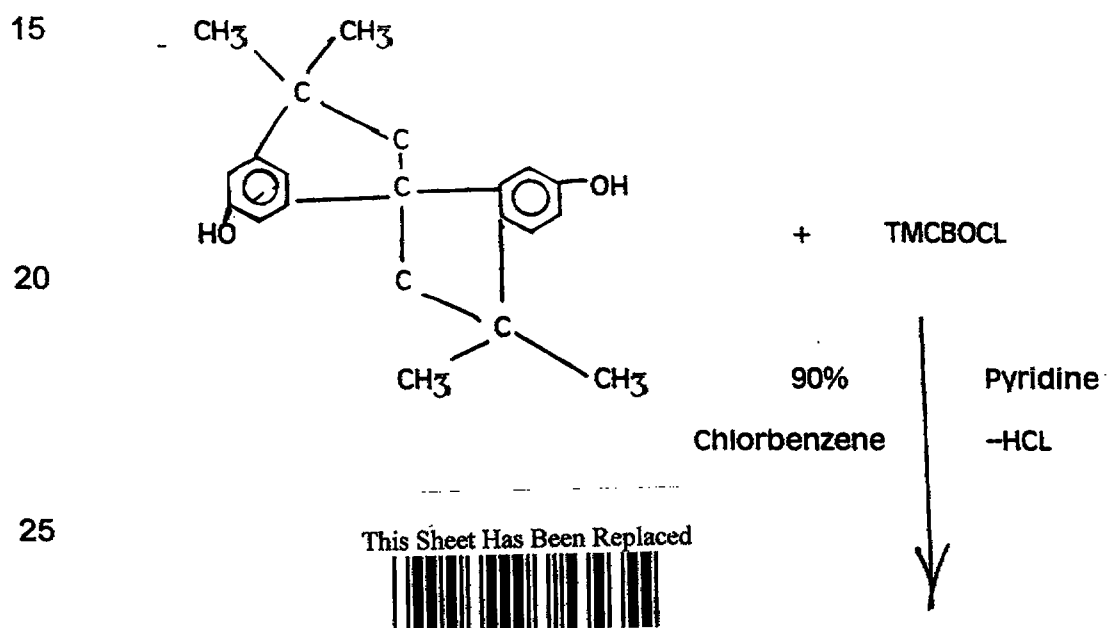


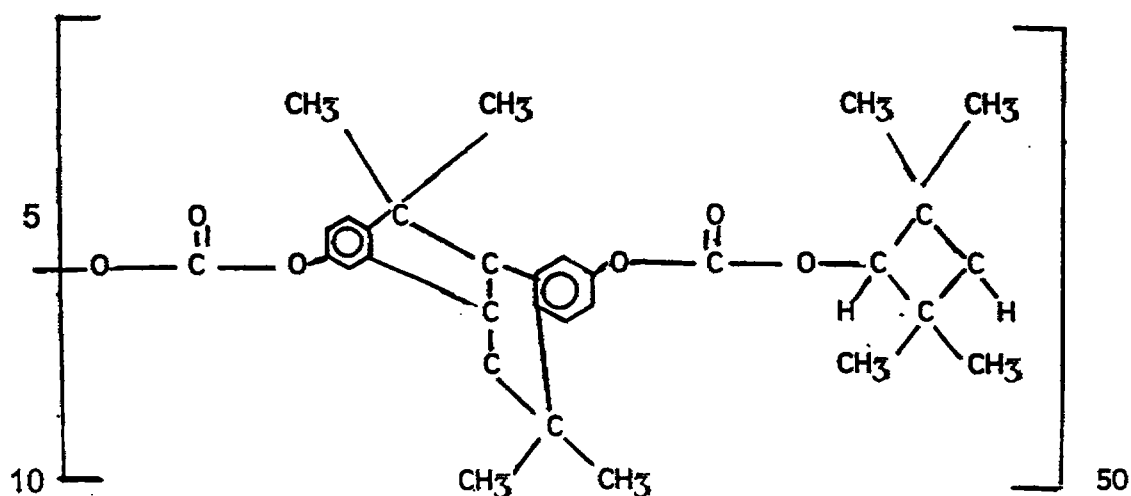
EXAMPLE 3

Example 2 was repeated except that in place of the
 10 isopropylidene the bisphenol A, spirobiindane dihydroxy phenol
 was employed herein.

The yield was greater than 90% and the polymer was
 confirmed by NMR.

Reaction





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EXAMPLE 4

Example 2 was repeated except that in place of the isopropylidene of the bisphenol A, acetophenone dihydroxy phenol (Bis AP) was employed herein. The results were essentially the same.

EXAMPLE 5

The copolymer of Example 2 was injection molded at 350°C into IZOD test bar, which bars were translucent with good dimensional stability.

EXAMPLE 6

The copolymer of Examples 2, 3 and 4 were each separately cast into thin films in a glass dish from a 10 weight % solution of each copolymer in methylene chloride. The films



were then removed and dried at 85°C for about 24 hours. The results were as follows:

	<u>MW</u>	<u>MWD</u>	<u>Td (°C)</u>	<u>Tg(°C)</u>	<u>NMR*</u>
Example 2	65000	3.3	383	144	alternating
Example 3	24000	6.3	320	125	alternating
Example 4	26000	3.1	370	170	alternating

* Structure confirmed by NMR

Td - decomposition temperature

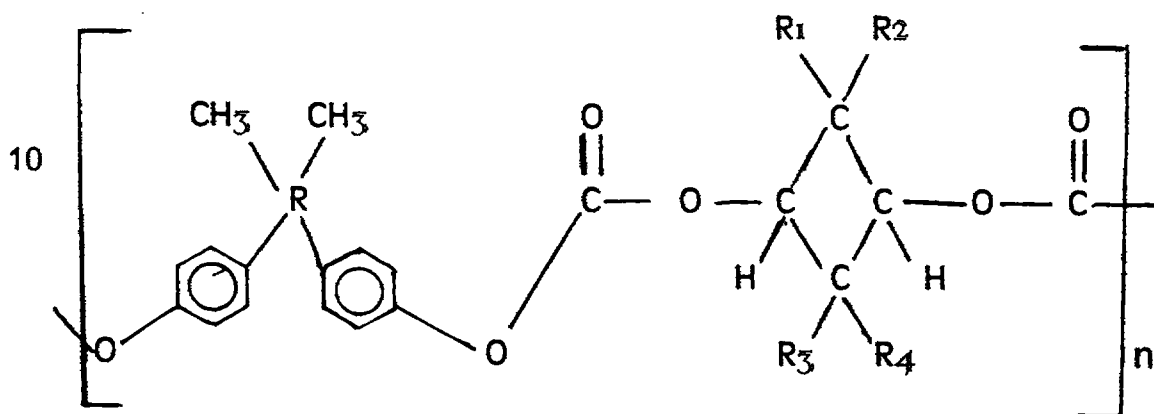
5 Tg - glass transition temperature

While many modifications and variations of the present invention are possible in view of the foregoing specification, it is understood that they would fall within the scope of the appended claims.

**CLAIMS**

What is claimed is:

1. A carbonate polymer consisting essentially of the
5 residue of the polymerization reaction of an aromatic dihydroxy
compound and an aliphatic haloformate, said carbonate
polymer is represented by the following formula:



- 15 wherein R, R₁, R₂, R₃ and R₄ are independently selected from
the group consisting of alkyl, aryl, and alkylaryl hydrocarbons of
1 to 20 carbon atoms and n is a integer of 5-200, and wherein
the ratio of aromatic species to aliphatic species is 1:05 to 1:1.5.

2. The carbonate polymer of claim 1 wherein the
aromatic dihydroxy compound is a mixture of at least two
different aromatic dihydroxy compounds.

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3. The carbonate polymer of claim 1 where the aromatic dihydroxy compound is selected from the group consisting of bisphenol A, acetophenone, spirobiindane and mixtures thereof.
4. The carbonate polymer of claim 1 wherein R, R₁, R₂, R₃ and R₄ are hydrocarbons of 3-10 carbon atoms.
5. The carbonate polymer of claim 1 wherein the aromatic dihydroxy compound is an aromatic dihydroxy phenol and the aliphatic haloformate is a bishaloformate.
6. The carbonate polymer of claim 5 wherein the bishaloformate is bischloroformate.
7. The carbonate polymer of claim 5 wherein the aromatic dihydroxy phenol is 2,2-bis(4-hydroxyphenyl)propane.
8. The carbonate polymer of claim 6 wherein the bischloroformate is tetramethylcyclobutane bischloroformate.
9. A process for preparing a carbonate polymer by an interfacial polymerization reaction comprising the steps of (1) reacting an aliphatic diol with a halogen containing carbonate precursor to form an aliphatic haloformate, (2) reacting the
5 residue of (1) with an aromatic dihydroxy compound in an interfacial polymerization reaction in the presence of an amine

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catalyst, (3) carrying out the reaction at essentially about ambient temperatures and pressure and at a pH of 8-14 and (4) recovering the carbonate polymer consisting essentially of the residue of an aliphatic haloformate and an aromatic dihydroxy
5 compound wherein the ratio of aromatic species to aliphatic species in the recovered polymer is 1:05 to 1:1.5.

10. The process of claim 9 wherein the carbonate precursor is a carbonyl halide.

11. The process of claim 9 wherein the aromatic dihydroxy compound is an aromatic dihydroxy phenol.

12. The process of claim 9 wherein the aliphatic diol is a tetraalkylcyclobutanadiol.

13. The process of claim 10 wherein the carbonyl halide precursor is carbonyl chloride.

14. The process of claim 9 wherein the aliphatic haloformate is an aliphatic bischloroformate.

15. The process of claim 9 wherein the aromatic dihydroxy phenol is 2,2-bis(4-hydroxyphenyl) propane and the aliphatic haloformate is tetramethylcyclobutane bischloroformate.

16. The process of claim 9 wherein the pH is 10-14 and the catalyst is triethylamine.

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**INTERFACIAL POLYCARBONATE POLYMERIZATION PROCESS
AND PRODUCT**

ABSTRACT OF THE INVENTION

A process for preparing an aromatic carbonate polymer by an interfacial polymerization reaction by first forming an aliphatic chloroformate and then reacting the bischloroformate
5 with a dihydroxy phenol such as bisphenol-A. The resulting polymer consists of alternating units of the reaction residue of the dihydroxy phenol and the aliphatic chloroformate.

The cycloalkyl group contains preferably 4 to 6 carbon atoms although up to 12 carbon atoms can be used. The cycloalkyl group may be unsubstituted or substituted by a hydrocarbon, a halogen, or the like group, providing that the substituents are not reactive with the other reactants of the reaction system of this invention. Examples of such cycloalkyls include cyclobutyl, 2,2,4,4-tetramethylcyclobutyl, cyclopentyl, cyclohexyl, methylcyclohexyl groups. The preferred aliphatic diol is 2,2,4,4-tetramethylcyclobutanediol-1,3.

The haloformate of the aliphatic diol is formed by reacting an aliphatic diol with a carbonate precursor, preferably a halocarbonyl precursor and more particularly a carbonyl chloride such as phosgen. When employing phosgen, the preferred intermediate reactant formed is 2,2,4,4-tetramethyl-1,3-cyclobutane bischloroformate (TMCBOCI).

A critical feature of this invention is that the haloformate of the aliphatic diol must be formed prior to the reaction thereof with an aromatic dihydroxy diol. If the haloformate of the aliphatic diol is not first formed, then the reaction may result in a copolymer that is not alternating, i.e. 1:1, aliphatic and aromatic units. In addition, another feature of this

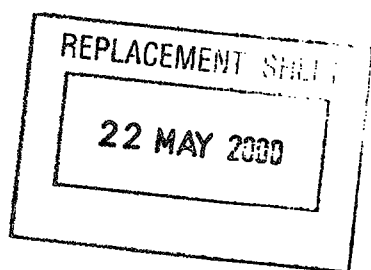
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invention is that the reaction is carried out at a high pH about 8-14 and preferably about 10 to 14.

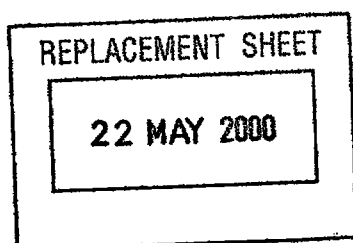
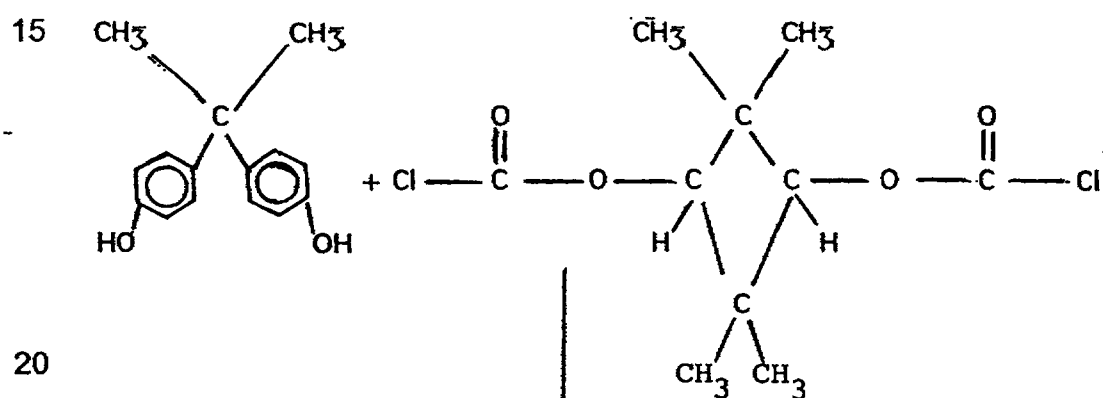
As stated previously, the other reactant of this process is an aromatic dihydroxy reactant such as an aromatic dihydric phenol. The dihydric phenols include, but are not limited thereto, 2,2-bis(4-hydroxyphenyl) propane; 2,4'-dihydroxydiphenylmethane; bis(2-hydroxyphenyl) methane; bis(4-hydroxyphenyl) methane; bis(4-hydroxy-5-nitrophenyl) methane; bis(4-hydroxy-2,6-dimethyl-3-methoxyphenyl) methane; 1,1-bis(4-hydroxyphenyl) ethane; 1,2-bis(4-hydroxyphenyl) ethane; 1,1-bis(4-hydroxy-2-chlorophenyl) ethane; 1,1-bis(2,5-dimethyl-4-hydroxyphenyl) ethane; 1,3-bis(3-methyl-4-hydroxyphenyl) propane; 2,2-bis(3-phenyl-4-hydroxyphenyl) propane; 2,2-bis(3-isopropyl-4-hydroxyphenyl) propane; 2,2-bis(4-hydroxynaphthyl) propane; 2,2-bis(4-hydroxyphenyl) pentane; 3,3-bis(4-hydroxyphenyl) pentane; 2,2-bis(4-hydroxyphenyl) heptane; bis(4-hydroxyphenyl) phenylmethane; bis(4-hydroxyphenyl) cyclohexylmethane; 1,2-bis(4-hydroxyphenyl)-1,2-bis(phenyl) propane; 2,2-bis(4-hydroxyphenyl)-1-phenylpropane; the bisphenol of 4-vinyl cyclohexane; spiro biindane bisphenol; and the like. Also included are dihydroxybenzenes typified by the hydroquinone

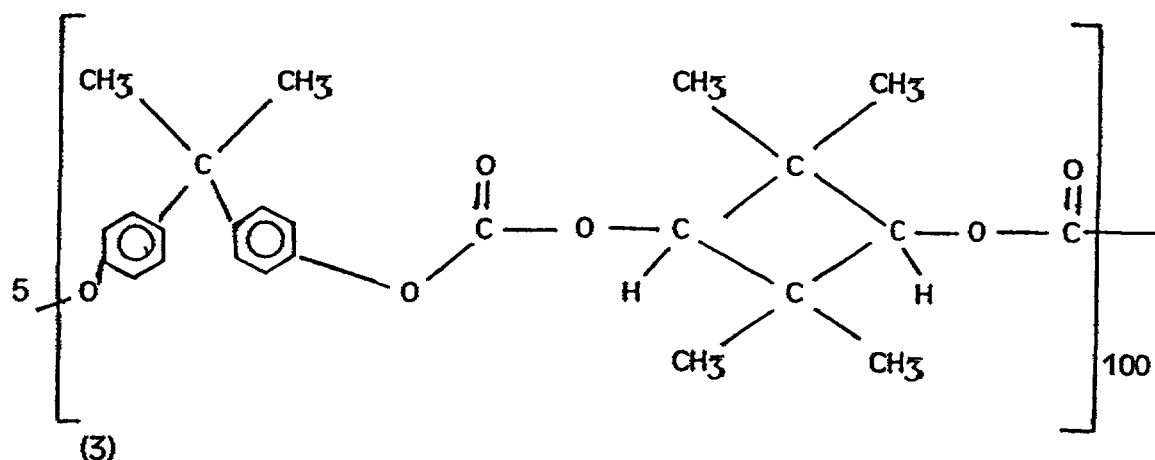


EXAMPLE 2

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The yield was over 90% and the polymer was confirmed by NMR.

Reaction

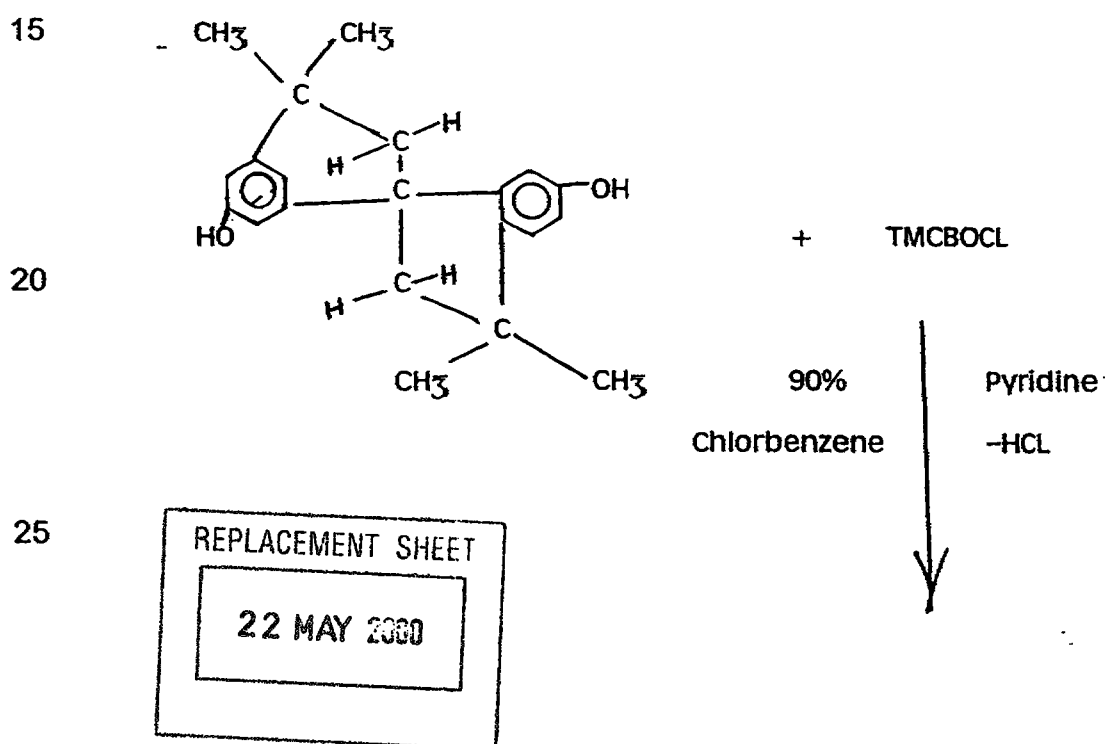


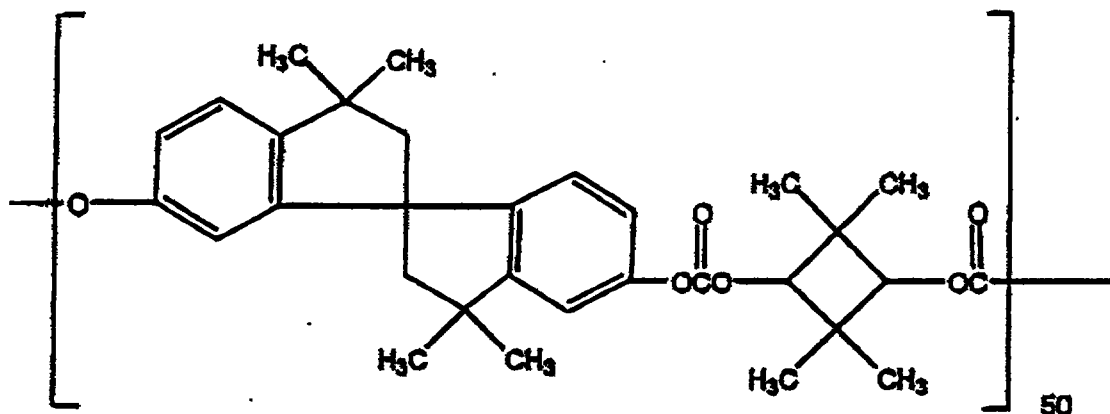
EXAMPLE 3

Example 2 was repeated except that in place of the isopropylidene the bisphenol A, spirobiindane dihydroxy phenol was employed herein.

The yield was greater than 90% and the polymer was confirmed by NMR.

Reaction





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EXAMPLE 4

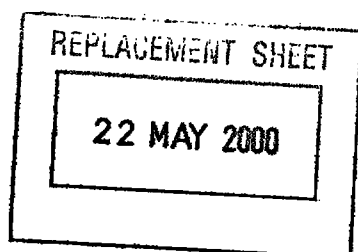
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EXAMPLE 5

The copolymer of Example 2 was injection molded at
 20 350°C into IZOD test bar, which bars were translucent with good
 dimensional stability.

EXAMPLE 6

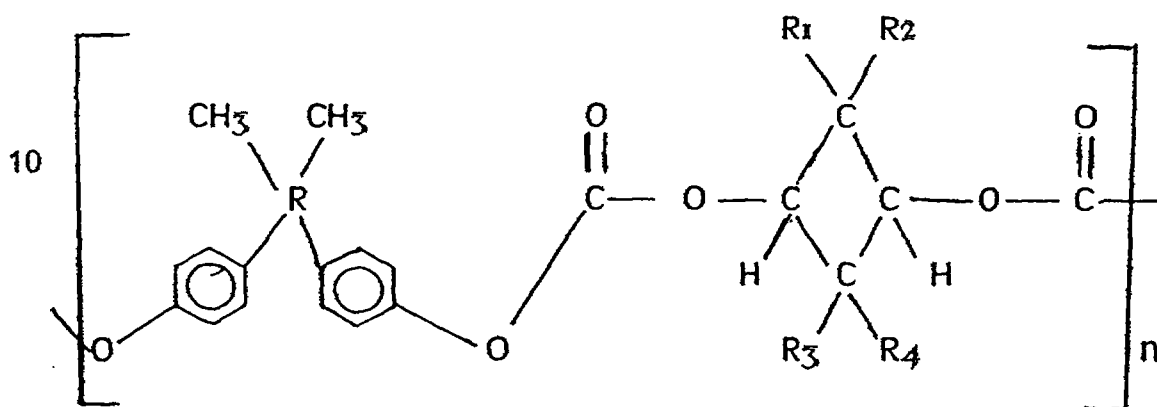
The copolymer of Examples 2, 3 and 4 were each
 separately cast into thin films in a glass dish from a 10 weight %
 25 solution of each copolymer in methylene chloride. The films



**CLAIMS**

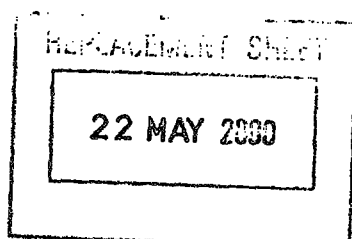
What is claimed is:

1. A carbonate polymer consisting essentially of the residue of the polymerization reaction of an aromatic dihydroxy compound and an aliphatic haloformate, said carbonate polymer is represented by the following formula:



- 15 wherein R, R_1, R_2, R_3 and R_4 are independently selected from the group consisting of alkyl, aryl, and alkylaryl hydrocarbons of 3 to 10 carbon atoms and n is a integer of 5-200, and wherein the ratio of aromatic species to aliphatic species is 1:0.5 to 1:1.5.

2. The carbonate polymer of claim 1 wherein the aromatic dihydroxy compound is a mixture of at least two different aromatic dihydroxy compounds.





3. The carbonate polymer of claim 1 where the aromatic dihydroxy compound is selected from the group consisting of bisphenol A, acetophenone, spirobiindane and mixtures thereof.

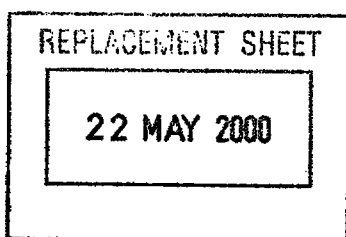
4. The carbonate polymer of claim 1 wherein the aromatic dihydroxy compound is an aromatic dihydroxy phenol and the aliphatic haloformate is a bishaloformate.

5. The carbonate polymer of claim 4 wherein the bishaloformate is bischloroformate.

6. The carbonate polymer of claim 4 wherein the aromatic dihydroxy phenol is 2,2-bis(4-hydroxyphenyl)propane.

7. The carbonate polymer of claim 5 wherein the bischloroformate is tetramethylcyclobutane bischloroformate.

8. A process for preparing a carbonate polymer by an interfacial polymerization reaction comprising the steps of (1) reacting an aliphatic diol with a halogen containing carbonate precursor to form an aliphatic haloformate, (2) reacting the residue of (1) with an aromatic dihydroxy compound in an interfacial polymerization reaction in the presence of an amine





catalyst, (3) carrying out the reaction at essentially about ambient temperatures and pressure and at a pH of 8-14 and (4) recovering the carbonate polymer consisting essentially of the residue of an aliphatic haloformate and an aromatic dihydroxy
5 compound wherein the ratio of aromatic species to aliphatic species in the recovered polymer is 1:0.5 to 1:1.5.

9. The process of claim 8 wherein the carbonate precursor is a carbonyl halide.

10. The process of claim 8 wherein the aromatic dihydroxy compound is an aromatic dihydroxy phenol.

11. The process of claim 8 wherein the aliphatic diol is a tetraalkylcyclobutanadiol.

12. The process of claim 9 wherein the carbonyl halide precursor is carbonyl chloride.

13. The process of claim 8 wherein the aliphatic haloformate is an aliphatic bischloroformate.

14. The process of claim 8 wherein the aromatic dihydroxy phenol is 2,2-bis(4-hydroxyphenyl) propane and the aliphatic haloformate is tetramethylcyclobutane bischloroformate.

15. The process of claim 8 wherein the pH is 10-14 and the catalyst is triethylamine.

