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(54) Title: COMPOUND FOR USE AS A MINERAL FIBRE BINDER AND PROCESS FOR PROVIDING SUCH

(57) Abstract

Compound or salts thereof suitable for use as a binder for mineral fibres, for example glass or stone wool, said compound comprising: a carboxylic acid group, and/or, a β-hydroxyalkyl amide group.

COMPOUND FOR USE AS A MINERAL FIBRE BINDER AND PROCESS FOR PROVIDING SUCH

The invention relates to a compound or salts thereof suitable for use as a binder for mineral fibres, i.e. men made vitreous fibres (MMVF), for example glass slag or stone wool, i.e. mineral wool, in particular stone wool, a binder composition comprising such a compound, a process for providing said compound and composition, a mineral fibre product provided with such a binder and the use of said compound and composition as a mineral fibre binder.

Any discussion of the prior art throughout the specification should in no way be considered as an admission that such prior art is widely known or forms part of common general knowledge in the field.

Phenol and formaldehyde resins which are mainly used as binders for glass or stone wool are toxic.

During application and curing of the binders, after provision thereof to the mineral fibres, phenol, formaldehyde and ammonia are released. From an environmental point of view this is undesirable.

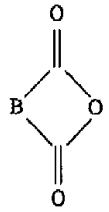
Furthermore during application, mostly by spraying, of the binder onto the spun glass or stone fibres a large amount of binder is lost, which is almost impossible to recover for re-use.

It is an object of the present invention to overcome or ameliorate at least one of the disadvantages of the prior art, or to provide a useful alternative.

According to a first aspect, the present invention provides a process for providing a water-soluble binder resin suitable for a mineral wool binder, said process comprising mixing together under reactive conditions, a cyclic anhydride and an alkanol amine to form a

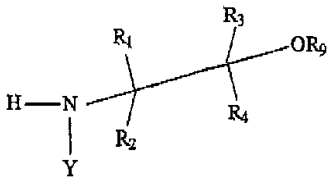
range of reaction products, said reaction products forming the components of the binder resin, wherein said process is carried out in the presence of water at a temperature in the range of 20°C to 100°C.

In one preferred embodiment, the cyclic anhydride has the following general formula (II),



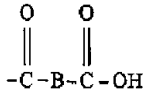
wherein B is selected from the group consisting essentially of (C_2-C_{20}) optionally substituted, aryl or (cyclo)alkyl aliphatic radical, 1,2 ethylene, 1,2-ethylidene, 4-carboxyl 1,2-phenylene, 1,3-propylene, 1,2-cyclohexyl, 1,2-phenylene, 1,3-phenylene, 1,4-phenylene and/or 1,2-cyclohex-4-enyl radical.

In another preferred embodiment, the alkanol amine has the following general formula III:



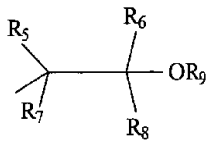
wherein R_1, R_2, R_3 and R_4 = H, (C_1 – C_8) aryl- or (cyclo)alkyl radical or CH_2-OR, in which R = H, aryl or (cyclo)alkylradical,

R_9 =



, a salt thereof or H

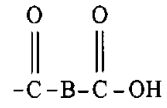
and Y =



, an alkyl group or an aryl group

wherein R_5, R_6, R_7 and R_8 = (C_1-C_8) aryl- or (cyclo)alkyl radical or CH_2-OR, in which R = H, aryl or (cyclo)alkylradical,

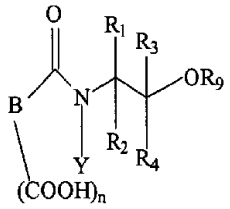
and R_9 =



, a salt thereof or H

According to a second aspect, the present invention provides a water soluble binder resin suitable for binding mineral fibres, said resin obtainable according to the process of the first aspect.

In one embodiment, the compounds have the following general formula (I):

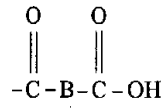


n = 1,2,3

B = (C_2-C_{20}) eventually substituted, aryl or (cyclo)alkyl aliphatic radical, 1,2-ethylene, 1,2-ethylidene, 4-carboxyl 1,2-phenylene, 1,3-propylene, 1,2-cyclohexyl, 1,2-phenylene, 1,3-phenylene, 1,4-phenylene and/or 1,2-cyclohex-4-enyl radical,

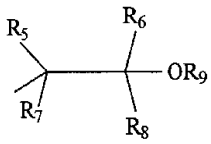
R_1, R_2, R_3, R_4, R_5, R_6, R_7 and R_8 = H, (C_1-C_8) aryl- or (cyclo)alkyl radical or CH_2-OR, in which R = H, aryl or (cyclo)alkylradical,

R_9 =



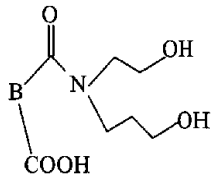
, a salt thereof or H

and Y =

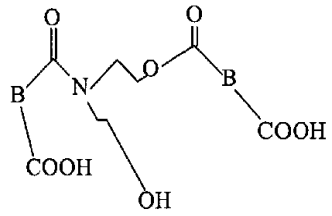


, an alkyl group or an aryl group

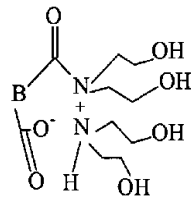
In another embodiment, the compound is selected from the group consisting of the following compounds A-I, wherein B has the same meaning as above:



(A)



(B)



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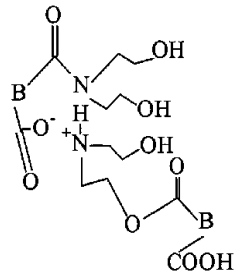

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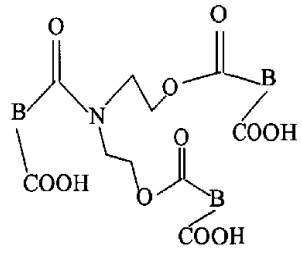

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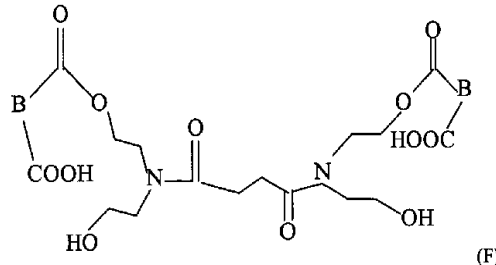
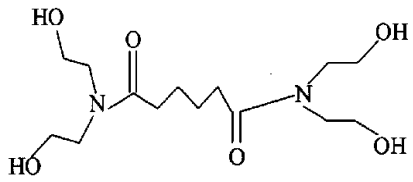
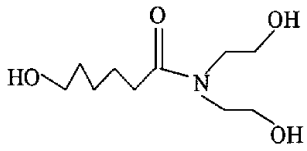


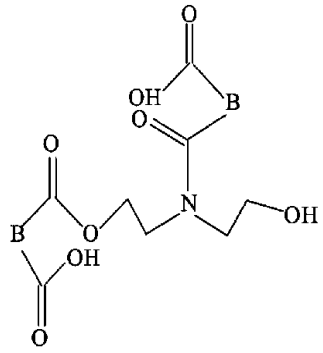
(F)



(G)



(H)



(I)

In a third aspect, the present invention provides a binder composition suitable for mineral fibres, said binder composition comprising:

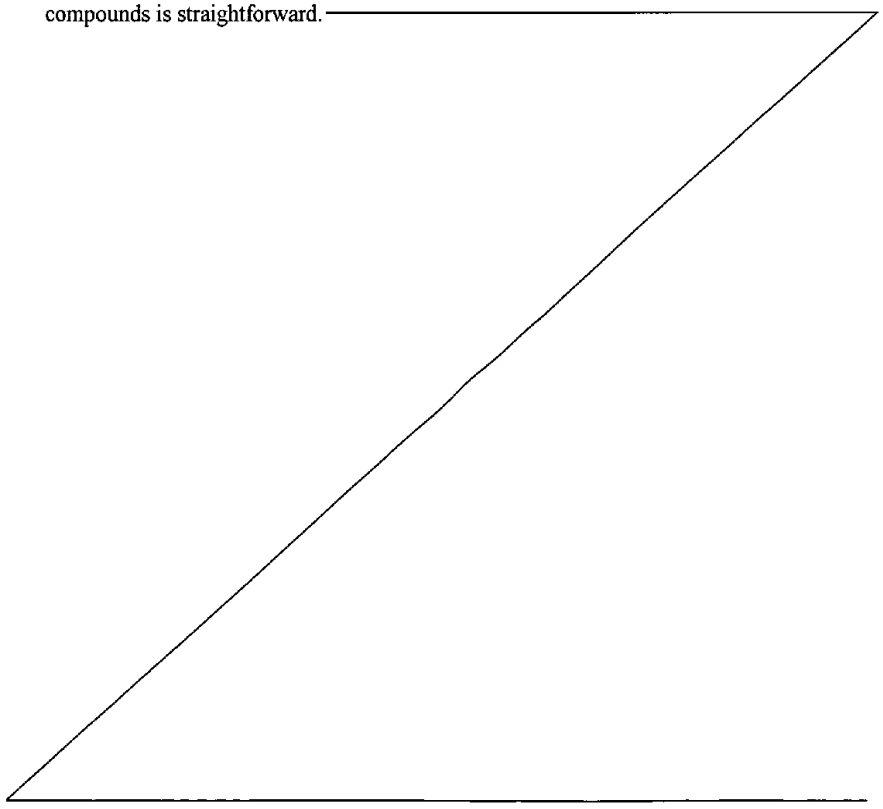
- a resin compound according to the second aspect or salts thereof, and,
- standard binding composition additives.

In a fourth aspect, the present invention provides a process for providing a bound mineral fibre product, comprising administering a binder resin or a binder composition according to the second or third aspects to the mineral fibres and curing the binder.

In a fifth aspect, the present invention provides a mineral fibre product obtainable according to the fourth aspect.

Unless the context clearly requires otherwise, throughout the description and the claims, the words 'comprise', 'comprising', and the like are to be construed in an inclusive sense as opposed to an exclusive or exhaustive sense; that is to say, in the sense of "including, but not limited to".

The inventors have found that the compounds according to the invention are particularly suitable for use as a binder for mineral wool products, whereby since the compound is preferably non-polymeric and has a low molecular weight, costs are minimized with respect to polymeric binders, and the handling of such non-polymeric compounds is straightforward.



Since the compound is soluble in water no further solublizing agents are required in order to provide a soluble binder having a desired viscocity for adhering to the mineral fibres.

Moreover on applying or curing the compound according to the present invention, no toxic materials are released into the environment.

The compounds according to the present invention also have desirable properties with respect to hardness, scratch resistance, chemical resistance, mechanical properties and adhesive properties once cured.

In formula (I) the R-groups, with the exception of R_9 can form either together or with the adjacent carbon atoms, or with the carbon atoms on B or Y a cyclo aliphatic group.

Preferably, B is a 1,2-ethylene, 1,2-ethylidene, 1,3-propylene, 1,2-cyclohexyl, 1,2-phenylene, 4-carboxyl-1,2-phenylene, 1,3-phenylene, 1,4-phenylene and/or 1,2 cyclohex-4-enyl radical.

B can be saturated or unsaturated.

B can be substituted with for instance a (C_1-C_{12}) alkyl group which is saturated or unsaturated.

B can form a part of a polymer. Such polymers can be obtained by the reaction of anhydride function polymers with a β-hydroxy alkylamine or a derivative thereof.

Anhydride functional polymers can for instance be obtained by a radical polymerisation of Maleic anhydride with styrene and with (meth)acrylate monomers.

Maleïc anhydride can also be grafted onto unsaturated compounds. A reaction between maleïc anhydride and oils, such as for instance linseed oil, results in products, which are called maleïnised oils,

which may be grafted onto unsaturated compounds, used as a comonomer or mixed into the compounds.

If B does not form part of a polymer, the molecular weight of the compounds, is less than 1000 and preferably less than 600.

Compositions according to the invention exhibit the properties as described above for the compound.

The composition may contain more than 10 wt%, for example more than 25 wt%, and preferably 50 wt% or more of the compound according to the invention.

Standard binding additives can improve the binder, examples of such additives include:

aminopropyl siloxane to improve the adhesion on glass, stabilizers to prevent thermal or UV degradation and surface-active compounds. Fillers, such as clay, silicates, magnesium sulfate and pigments, such as titanium oxide, can also be applied, as well as hydrophobising agents such as fluorine compounds, oils, minerals and silicone oil (reactive or non reactive).

The composition may also be applied in combination with other binder compositions such as for instance phenol- formaldehyde resins.

A very good binding strength is achieved when an accelerator is added to the composition, a preferred accelerator being sodium hypophosphite.

Furthermore since the binder composition is preferably composed of low molecular weight compounds, it has a viscosity at high concentrations which is lower than polyacrylic binders for example.

This is advantageous since on curing, following an initial flash evaporation, any water present usually evaporates. Before curing the composition still has a viscosity which allows it to be sprayed onto the mineral fibres and adhere thereto once sprayed.

Since the composition is intrinsically water soluble, no solublizing agents need to be provided thereto in order to enable application of the composition to the mineral fibres, the viscosity of the composition being high enough to adhere well to the mineral fibres and low enough, as stated above to enable sprayability. A decrease in the viscosity can be achieved by heating the composition to a temperature below which an eventual condensation reaction takes place.

For a schematic illustration of the reaction, for example, between tetrahydro pthalic anhydride and diethanolamine see figure 1.

The reaction between the anhydride and the alkanolamine can proceed without a solvent, in water or in an organic solvent. Preferably, the reaction starts in the presence of < 40 weight % of water compared to the reactants.

The distillation of the water can, if desired, proceed at 1 bar, under vacuum or azeotropically.

The equivalent ratio anhydride: alkanolamine lies generally between 1,8:1,0 and 1,0:1,8. Preferably, this ratio lies between 1,5:1,0 and 1:1,5.

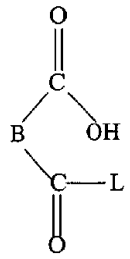
In case a high crosslink density is desired, di- or trialkanolamine or carboxylic acid functional anhydrides can be applied as starting materials.

The reaction of diethanolamine with an activated ester, such as a cyclic anhydride, can also result in an ester amine.

However, the same product can also be formed out of the β-hydroxyalkylamide because of an internal rearrangement. The inventors have measured that the β-hydroxyalkylamide and the ester-amine form an equilibrium with each other usually in a 85/15 ratio. In case the ester-amine reacts further with a cyclic anhydride another β-hydroxyalkylamide is formed, see figure 2, for example.

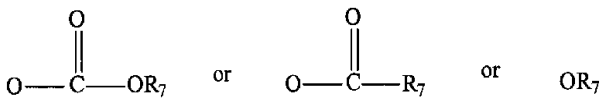
The applied compound can also be obtained by the reaction between an alkanolamine, such as for instance described above and a compound having one carboxylic acid group and an activated carboxylic acid group.

The compound having a carboxylic acid group and an activated carboxylic acid group is preferably a compound according to the following formula:



In which

- B has the meaning as referred to in the claims.
- L =



In which R^7 is a (C_1-C_{12}) branched or linear alkyl group.

Examples of appropriate compounds with one carboxylic acid group and one activated carboxylic acid groups are alkyl esters, such as for instance mono(m)ethyladipate and mono(m)ethylsebacate. Activated carboxylic acid groups are for instance anhydrides and thioesters.

The compound applied in the invention can also be obtained by reaction between a cyclic anhydride, such as described above, and an alcohol after which the obtained reaction product in situ reacts with an alkanolamine.

Examples of appropriate alcohols are (C_1-C_{10}) alcohols. Preferably methanol or ethanol are applied.

Another binder composition according to the invention can be obtained by reacting linear polyanhydrides with alkanolamines or derivatives.

It is also possible that the carboxylic acid groups and the β-hydroxy alkylamide groups are not located on the same compound.

According to a further aspect of the present invention, there is provided a composition suitable for use as a binder, said composition containing one or more compounds with carboxylic acid groups or β-hydroxyalkylamide groups. Suitable water soluble molecules having β-hydroxyalkylamides can be obtained as shown in figures 4 and 5 wherein the starting materials are dimethyl adipate and caprolactone respectively. Because no salt formation is possible with these molecules, it is known that predominantly (>70 %) the amides are formed.

This reaction can proceed in the presence of a catalyst such as for instance sodium methanolate. If no catalyst is used, the reaction should be performed at a higher temperature.

The carboxylic acid containing compounds are fully or partially water soluble compounds, such as maleic acid, glutaric acid, adipic acid, 2-methyl adipic, succinic acid, citric acid and tartaric acid.

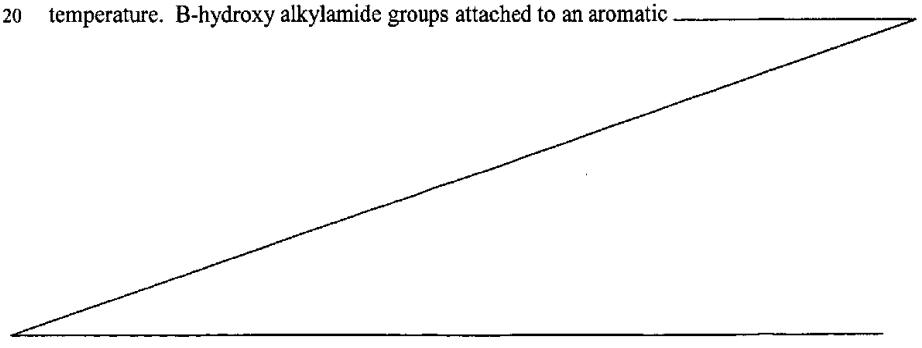
The carboxylic acid groups containing compounds can also be obtained by partial or full reaction of high functional alcohols, mono di and polysaccharides, such as sucrose or polyvinylalcohol, with cyclic anhydrides as described above.

Water soluble compounds are compounds which can be homogeneously divided in water. Eventually, emulsions or dispersions can be applied.

To further improve the water solubility of the carboxylic acid functional compounds, a base can be added for, example, a base is added which evaporates during the curing reaction. Examples of such bases are amines such as ammonia, methylamine, diethylamine and triethylamine.

In another preferred form of the invention addition products having β-hydroxyalkylamides with cyclic anhydrides are used. Figure 6 shows one of the resultant reaction products.

The binder composition according to the invention is preferably sprayed onto the fibres just after the spinning of the glass or the stonemelt. The curing of the binder composition proceeds by bringing the sprayed fibres into an oven. The curing time is mainly dependent on the components used in the binder and on the desired oven temperature. B-hydroxy alkylamide groups attached to an aromatic



group, for instance, will react slower with carboxylic acids than the ones attached on an aliphatic group and aromatic carboxylic acids will react faster with β-hydroxy alkylamides than aliphatic carboxylic acid groups. The curing temperatures lie mostly between 150°C and 400°C and preferably between 200°C and 400°C. The curing times lie mostly between 10 sec and 600 sec.

Unused binder, can, due to its low reactivity, be recycled. If water of the binder composition is evaporated during this process, water may be returned to the process to return the viscosity to the desired level, if needed.

When spraying binder composition to the fibers does not end in the wool but is collected in process water either directly or when cleaning walls and ducts in the spinning chamber system. This water may be used as dilution water for the binder, where by loss of binder is avoided/reduced.

The raw materials for fibres composition can be converted to a melt in the conventional manner, for instance in a gas heated furnace or in an electric furnace or in a shaft or cupola furnace. The melt can be converted to fibres in the conventional manner, for instance by a spinning cup process or by cascade rotor process, for instance described in WO 92/06047.

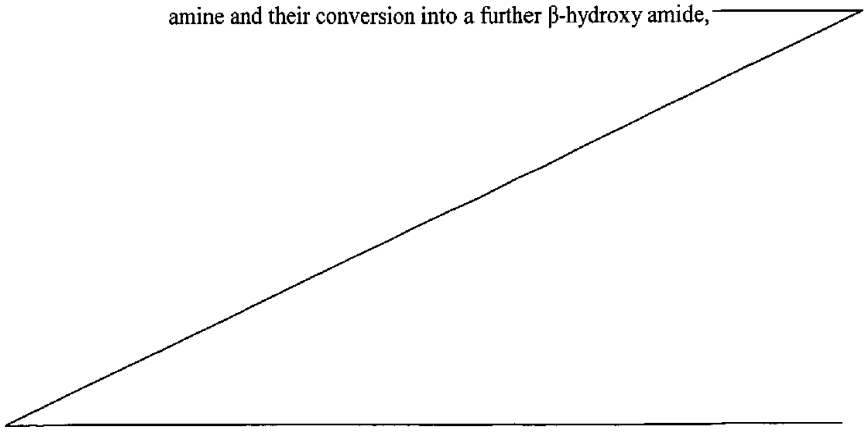
Man made vitreous fibres (MMVF) are made from vitreous melt, such as of stone, slag, glass or other melts. The melt is formed by melting in a furnace a mineral composition having the desired analysis. This composition is generally formed by blending rocks or mineral to give the desired analysis. The binder can be used on MMVF which are durable in use but which have

been shown to be biologically soluble e.g. as described in EP 791 087 and EP 596 088.

The fibres can have any convenient fibre diameter and length. Generally the average fibre diameter in below 10 μm e.g. 5 μm. Usually a mineral wool product contains 1-15 wt.% binder, preferably 2-10 wt.%. Usually the binder is added to the fibres just after fibersation of the melt. Generally the mineral wool product is in form of a slab, sheet or other shaped articles. Products according to the invention may be formulated for any of the conventional purposes of MMV fibres, for instance slabs, sheets, pipes or other shaped products that are to serve as thermal insulation, fire insulation and protection or noise reduction and regulation or as horticultural growing media. The binder can also be used to coat the surface of either the fibres or one or more of the surfaces of the mineral wool product. Silane and mineral oil are typical additives for mineral wool products. A typical phenolic binder is described in US 4710 406.

The invention will now be described by way of the following examples 1-12, tables 1, 2 and 3 and figures 1-7, wherein;

- figures 1, 2 and 7 show schematically a reaction process for providing compounds according to the present invention,
- figure 3 shows the equilibrium between a β-hydroxy amide and ester amine and their conversion into a further β-hydroxy amide,



- and figure 4 and 5 show respectively the reaction between dimethyl adipate and caprolactone with diethanolamine in order to provide compounds according to the present invention,
- figure 6 shows mixtures of different compounds according to the present invention obtained by reacting functional β-hydroxyalkyl amides partially with cyclic anhydrides.

Example 1

Preparation of the condensation product of phthalic anhydride with diethanolamine

In a double jacketed glass reactor, heated with mineral oil, provided with a mechanical stirrer and a nitrogen inlet, 300 g phthalic anhydride, 100 g water and 212 g diethanolamine were brought. The reaction mixture was steadily heated whilst stirring to ca. 70°C. After two hours the phthalic anhydride was completely dissolved and the reaction was a clear, colorless, low viscous solution.

Example 2

Preparation of the condensation product of succinic anhydride with diethanolamine

In a double jacketed glass reactor, heated with mineral oil, provided with a mechanical stirrer and a nitrogen inlet, 300 g succinic anhydride, 100 g water and 315 g diethanolamine were. The reaction mixture was steadily heated whilst stirring to ca. 70°C. After two hours the succinic anhydride was completely dissolved and the reaction product was ready. The reaction product was a clear, colorless, low viscous solution.

Investigation into the compounds obtained in Examples 1 and 2

The compounds obtained according to examples 1 and 2 were cured on a glass plate in an oven during 60 sec at 250°C.

The cured compounds had very good properties with respect to hardness, scratch resistance, chemical resistance, mechanical properties and adhesion to glass.

Example 3

Preparation of the condensation product of 1.2.3.6-tetrahydrophthalic anhydride with diethanolamine

In a double jacketed glass reactor, heated with hot water, provided with a magnetic stirrer and a nitrogen inlet, 120 g 1.2.3.6-tetrahydrophthalic anhydride, 40 g water and 84.8 g diethanolamine were brought. The reaction mixture was steadily heated, whilst stirring, to 70 °C. After 2 hours the anhydride was completely dissolved and the reaction product was ready. The reaction product was a clear, slightly yellow, low viscous solution, easily dilutable with water.

Example 4

Preparation of the condensation product of 1.2.3.6-tetrahydrophthalic anhydride with diethanolamine

In a double jacketed glass reactor, heated with hot water, provided with a magnetic stirrer and a nitrogen inlet, 170 g 1.2.3.6-tetrahydrophthalic anhydride, 120 g water and 84.8 g diethanolamine were brought. The reaction mixture was steadily heated, whilst stirring, to 70 °C. After 2 hours the anhydride was completely dissolved and the reaction product was ready. The reaction product was a clear, slightly yellow, low viscous solution, easily dilutable with water.

Example 5

Preparation of the condensation product of 1.2.3.6-tetrahydrophthalic anhydride with diethanolamine

In a double jacketed glass reactor, heated with hot water, provided with a magnetic stirrer and a nitrogen inlet, 244 g 1.2.3.6-tetrahydrophthalic anhydride, 120 g water and 84.8 g diethanolamine were brought. The reaction mixture was steadily heated whilst stirring to 70 °C. After 2 hours the anhydride was completely dissolved and the reaction product was ready. The reaction product was a clear, slightly yellow, and exhibited a low dilutability with water.

Example 6

Preparation of the condensation product of 1.2.3.6-tetrahydrophthalic anhydride with diethanolamine

In a double jacketed glass reactor, heated with hot water, provided with a magnetic stirrer, 40 g water, 84.8 g diethanolamine and 20 g 1.2.3.6-tetrahydrophthalic anhydride were brought. The reaction mixture was steadily heated while stirring to 90 °C. As soon as the anhydride dissolved another 20 g anhydride was added, followed with another 20 g again until dissolved and further until totally 120 g 1.2.3.6-tetrahydrophthalic anhydride was added. 15 minutes after the anhydride was completely dissolved the reaction product was ready. The reaction product was clear, slightly yellow, low viscous and easily dilutable with water.

Example 7

Preparation of the condensation product of succinic anhydride with diethanolamine

In a double jacketed glass reactor, heated with water and provided with a magnetic stirrer; 120 g of succinic anhydride, 80 g water and 126 g diethanolamine were brought. The reaction mixture was steadily heated while stirring to 90 °C. After two hours, the succinic anhydride was completely dissolved and the reaction

product was ready. The reaction product was a clear, colourless, low viscous solution, easily dilutable with water. Diluted with water to 41% solid content, the viscosity was 6.3 cPs. Binder analysis has given that 46.6% of the amine was bound as amide and 10.9% of the hydroxy groups were bound as ester.

Example 8

Preparation and testing of selected binder samples to evaluate the binding strength towards shots with mineral fibre composition (Grit bar test)

Shots with size between 0,25 and 0,5 mm diameter were used to make bars with dimensions 140 mm x 25 mm x 10 mm.

For making the bars 90 ml binder solution with 15% solids content and 0,2% silane coupling agent of binder solids were mixed with 450 g shots.

The coupling agent was gamma-aminopropyltriethoxysilane.

To some of the binder solutions were added NaH_2PO_2-H_2O (3% of binder solids) as curing accelerator.

Out of the 450 g shots mixed with binder solution can be made 8 bars which is cured 2 hours at 200°C in an incubator.

Four of the bars were broken directly (dry strength), the other 4 are placed 3 hours in 80°C water before they are broken (wet strength).

The binding strength was determined by breaking the bars in a measuring device, where the clamping length is 100 mm and the velocity of the compressing beam was 10 mm/min. Using the clamping length, width and thickness of the bars, the bending strength was determined in N/mm^2.

Table 1 Results obtained by Grit Bar Test

	Binding strength dry	Binding strength (wet) 80°C water	
Binder example 1	No strength		DEA:PTA 1:1
Binder example 2	11 N/mm^2	1 N/mm^2	DEA:SCA 1:1
Binder example 3	7 N/mm^2	3 N/mm^2	DEA:THPA 1:1
Binder example 3 + accelerator	9 N/mm^2	2 N/mm^2	DEA:THPA 1:1
Binder example 4	6 N/mm^2	2 N/mm^2	DEA:THPA 1:1.4
Binder example 4 + accelerator	10 N/mm^2	4 N/mm^2	DEA:THPA 1:1.4
Binder example 6	4 N/mm^2	2 N/mm^2	DEA:THPA 1:1
Binder example 6 + accelerator	8 N/mm^2	3 N/mm^2	DEA:THPA 1:1
Binder example 7 + accelerator	11 N/mm^2		DEA:SCA 1:1
Standard phenolic binder	5-6 N/mm^2	3-4 N/mm^2	

DEA = diethanolamine, SCA = succinic anhydride,
THPA = 1.2.3.6 tetrahydrophthalic anhydride,
PTA = phthalic anhydride.

Example 9

Preparation and testing of a mineral fibre product

Based on the results of the binding strength in example 8, a production trial on a standard stonewool line was performed. The binder used was as described in example 6.

It was produced a standard product with density 100 kg/m^3, 100 mm thickness and ignition loss about 2,5%.

The mechanical strengths were measured according to EN 826 (compression strength) and EN 1607 (delamination strength).

Table 2 Results (mean values of 8 samples)

Binder	Binder content	Oil content	Density	10% comp. strength	Delamination strength
DEA/THPA	2.56%	0.19%	98.8 kg/m^3	26.2 KPa	7.1 KPa
DEA/THPA +accelerator	2.61%	0.19%	101 kg/m^3	29.3 KPa	9.6 KPa
Reference Phenolic binder	2.5%	0.2%	100 kg/m^3	25 KPa	7 KPa

Example 10

Preparation of condensation product of 1.2.3.6-tetrahydrophthalic anhydride with diethanolamine

In a double jacket glass reactor, heated with hot water, provided with a magnetic stirrer, 42 g water, 84.8 g diethanolamine and 20 g 1.2.3.6-tetrahydrophthalic anhydride were brought. The reaction mixture was steadily heated whilst stirring to 90 °C. As soon as the anhydride dissolved another 20 g anhydride was added, followed with another 20 g again until dissolved, and further until totally 160 g anhydride was added. 15 minutes after the anhydride was completely dissolved the reaction product was ready. The reaction product was clear, slightly yellow, low viscous and easily dilutable with water.

The viscosity of the binder solution was measured at different solids content in water at 25 °C.

91.8% solids 400 000 cPs
78.8% solids 3 500 cPs
57.4% solids 56 cPs
10.0% solids 1.2 cPs

Binder analysis showed that 16.8% of the amine groups are bound as amide and 37.8% of the hydroxy groups were bound as ester.

Example 11

Preparation of the condensation product of 1.2.3.6-tetrahydrophthalic anhydride and phthalic anhydride with diethanolamine

In a double jacket glass reactor, heated with hot water, provided with a magnetic stirrer, 42 g water, 84.8 g diethanolamine and 20 g 1.2.3.6-tetrahydrophthalic anhydride were brought. The reaction mixture was steadily heated whilst stirring to 90 °C. As soon as the anhydride dissolved 20 g phthalic anhydride was added. When dissolved another 20 g 1.2.3.6-tetrahydrophthalic anhydride was added and further until totally 120 g 1.2.3.6-tetrahydrophthalic anhydride was added. 15 minutes after the anhydride was completely dissolved the reaction product is ready. The reaction product was clear, slightly yellow, low viscous and easily dilutable with water.

Example 12

Preparation of the condensation product of 1.2.3.6-tetrahydrophthalic anhydride with triethanolamine

In a double jacket glass reactor, heated with hot water, provided with a magnetic stirrer, 42 g water, 120 g triethanolamine and 20 g 1.2.3.6-tetrahydrophthalic anhydride were brought. The reaction mixture was steadily heated whilst stirring to 90 °C. As soon as the anhydride was dissolved another 20 g phthalic anhydride was added, followed with another 20 g again until dissolved, and further until totally 120 anhydride is added. 15 minutes after the anhydride was completely dissolved the reaction product was ready. The reaction product was clear, slightly yellowish brown, low viscous and easily dilutable with water.

Table 3

	Binding strength dry	Binding strength wet	
Binder example 4 (cured at 250°C)	4 N/mm^2	1 N/mm^2	DEA:THPA 1:1.4
Binder example 3 (cured at 250°C)	7 N/mm^2	3 N/mm^2	DEA:THPA 1:1
Binder example 11	4 N/mm^2	4 N/mm$_2$	DEA:THPA:PTA 1:1:0.15
Binder example 11 + accelerator	9 N/mm^2	5 N/mm^2	DEA:THPA:PTA 1:1:0.15
Binder example 12	4 N/mm^2	2 N/mm^2	TEA:THPA 1:1
Binder example 12 + accelerator	9 N/mm^2	3 N/mm^2	TEA:THPA 1:1

TEA = Triethanolamine

The invention is not limited to the above description and examples; the requested rights are rather determined by the following claims.

THE CLAIMS DEFINING THE INVENTION ARE AS FOLLOWS:

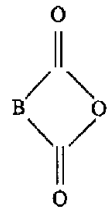
1. Process for providing a water-soluble binder resin suitable for a mineral wool binder, said process comprising mixing together under reactive conditions, a cyclic anhydride and an alkanol amine to form a range of reaction products, said reaction products forming the components of the binder resin, wherein said process is carried out in the presence of water at a temperature in the range of 20° to 100°C.

2. Process according to claim 1 wherein the alkanol amine is a secondary β-hydroxy alkylamine or an N-substituted alkanol amine.

3. Process according to claim 1 or claim 2 wherein the mixture is heated to at least about 50°C.

4. Process according to any one of claims 1 to 3, wherein the mixture is heated to at least about 70°C.

5. Process according to any one of claims 1 to 4, wherein the cyclic anhydride has the following general formula (II),



wherein B is selected from the group consisting essentially of (C_2-C_{20}) optionally substituted, aryl or (cyclo)alkyl aliphatic radical, 1,2 ethylene, 1,2-ethylidene, 4-carboxyl 1,2-phenylene, 1,3-propylene, 1,2-cyclohexyl, 1,2-phenylene, 1,3-phenylene, 1,4-phenylene and/or 1,2-cyclohex-4-enyl radical.

6. Process according to any one of the preceding claims, wherein the cyclic anhydride is selected from the group consisting essentially of:

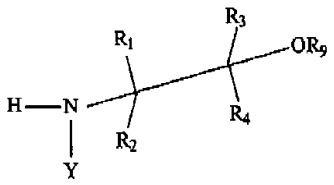
- phthalic anhydride, tetrahydro phthalic anhydride, hexahydro phthalic anyhydride, 5-norbornane-2, 3-dicarboxylic anhydride, norbornane-2, 3-dicarboxylic anhydride, 2-dodecene-1-yl-succinic anhydride, (methyl)-succinic anhydride, glutaric anhydride, 4-methylphthalic anhydride, 4-methylhexahydro phthalic anhydride, trimellitic anhydride, pyromellitic dianhydride and 4-methyltetrahydro phthalic anhydride, or mixtures thereof.

7. Process according to any one of the preceding claims wherein the cyclic anhydride is obtained by a reaction of maleic anhydride with an alkene.

8. Process according to any one of the preceding claims wherein the cyclic anhydride is provided with a carboxylic acid group.

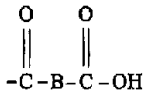
9. Process according to claim 8, wherein the anhydrides is in the form of a trimellitic anhydride.

10. Process according to any one of the preceding claims wherein the alkanol amine has the following general formula III:



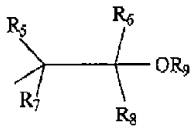
wherein R_1, R_2, R_3 and R_4 = H, ($C_1 - C_8$) aryl- or (cyclo)alkyl radical or CH_2-OR, in which R = H, aryl or (cyclo)alkylradical,

R_9 =



, a salt thereof or H

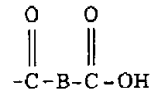
and Y =



, an alkyl group or an aryl group

wherein R_5, R_6, R_7 and R_8 = (C_1-C_8) aryl- or (cyclo)alkyl radical or CH_2-OR, in which R = H, aryl or (cyclo)alkylradical,

and R_9 =



, a salt thereof or H

11. Process according to claim 10, wherein the alkanol amine is selected from the group comprising:

- mono alkanol amines, dialkanol amines, tri alkanol amines or mixtures thereof.

12. Process according to claim 11, wherein the alkanol amine is (di) ethanolanime, 1-(m)ethylethanolamine, n-butyl-ethanolamine, 1-(m)ethylisopropanolamine, 3-amino-1,2-propanediol, 2-amino-1,3-propanediol, tris(hydroxymethyl)aminomethane.

13. Process according to claim 11, wherein the alkanol amine is diethanolamine.

14. Process according to any one of the preceding claims further carried out in the presence of a solvent.

15. Process according to any one of the preceding claims, wherein the reaction is started in the presence of less than 40 weight % of water as compared to the anhydride and alkanol amine.

16. Process according to any one of the preceding claims wherein the molar ratio of the anhydride: amine is below about 2:1.

17. Process according to claim 16, where the ratio is below about 1.5:1.

18. Process according to claim 16 or claim 17, wherein the ratio is about 1.42:1 or lower.

19. Process according to any one of the preceding claims, wherein the anhydride and amine are mixed together, substantially in the absence of a polymer.

20. A water-soluble binder resin suitable for binding mineral fibres, said resin obtainable according to the process of any one of the preceding claims.

21. A resin according to claim 20, wherein one or more of the reaction products contain one or more carboxylic acid groups which provide a cross-linking function within the binder.

22. A resin according to claim 20 comprising:

- a substantially polymer free compound having
- a carboxylic acid group, and/or,
- a β-hydroxyalkyl amide group.

23. Resin according to claim 22, wherein the ratio of the carboxylic acid groups and β-hydroxyalkyl groups lie between 1.0 : 5.0, and 5.0 : 1.0.

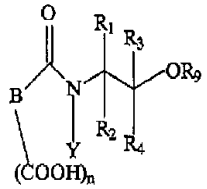
24. Resin according to claim 23, wherein the ratio of the carboxylic acid groups and the β-hydroxyalkyl groups lies between 1.0 : 3.0 and 2.0 : 1.0.

25. Resin according to any one of claim 22 to 24 wherein the functionality of the hydroxyalkyl groups lies in the range 1-250, and wherein the functionality of the carboxylic acid groups is less than about 250.

26. Resin according to claim 25, wherein the functionality of the hydroxyalkyl groups lies in the range 2 – 50.

27. Resin according to claim 25 or claim 26, wherein the functionality of the carboxylic acid groups lies in the range 1 – 50.

28. Resin according to any one of claims 22 to 27, said compound having the following general formula (I):

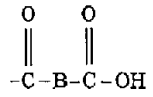


n = 1,2,3

B = (C_2-C_{20}) optionally substituted, aryl or (cyclo)alkyl aliphatic radical, 1,2-ethylene, 1,2-ethylidene, 4-carboxyl 1,2-phenylene, 1,3-propylene, 1,2-cyclohexyl, 1,2-phenylene, 1,3-phenylene, 1,4-phenylene and/or 1,2-cyclohex-4-enyl radical.

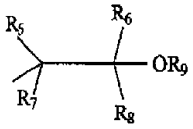
R_1, R_2, R_3, R_4, R_5, R_6, R_7 and R_8 = H, (C_1-C_8) aryl- or (cyclo)alkyl radical or CH_2-OR, in which R = H, aryl or (cyclo)alkylradical,

R_9 =



, a salt thereof or H

and Y =



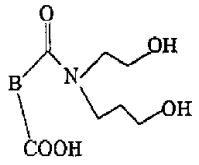
, an alkyl group or an aryl group

29. Resin according to any one of the preceding claims wherein said compound has a molecular weight of less than about 1000.

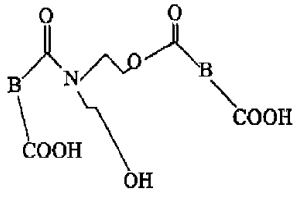
30. Resin according to claim 29, wherein said compound has a molecular weight of less than about 600.

31. Resin according to claim 29 or claim 30, wherein formula (I) as shown in claim 29 has R_1, R_2, R_3, R_4 = H.

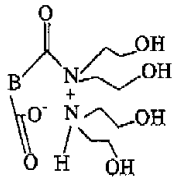
32. Resin according to any one of claims 22 to 31 wherein said compound is selected from the group consisting of the following compounds A-I, wherein B has the same meaning as in claim 28:



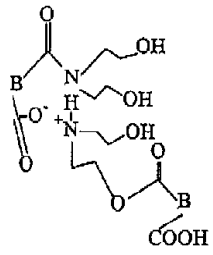
(A)



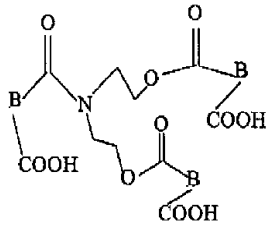
(B)



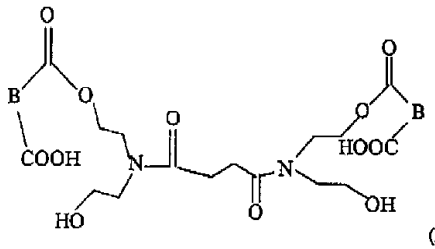
(C)



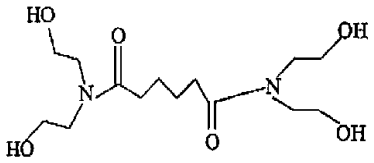
(D)



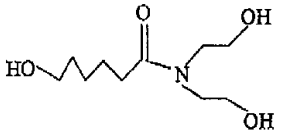
(E)



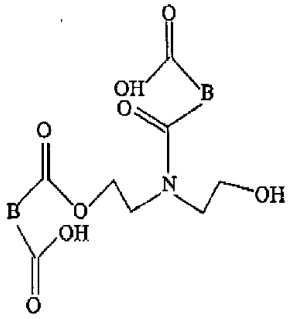
(F)



(G)



(H)



(I)

33. A binder composition suitable for mineral fibres, said binder composition comprising:

- a resin according to any one of claims 20 to 32 or salts thereof, and,
- standard binding composition additives.

34. A binder composition according to claim 33 suitable for glass wool and stone wool.

35. A binder composition according to claim 33 or claim 34 wherein said additives are selected from the following group:

Hydrophobicity agents, e.g. oil, mineral oil, silicone oil (reactive or non reactive), fluorocarbon compounds; hydrophilic surfactants as e.g. polyethylene glycols; Silanes or titanates; hydroxides and accelerators.

36. A binder composition according to claim 35 wherein the hydrophobicity agent is stearylamine.

37. A binder composition according to claim 35 wherein the hydroxides are $Mg(OH)_2$ or $Al(OH)_3$.

38. A binder composition according to claim 35 wherein the accelerator is sodium hydrophosphite.

39. A binder composition according to any one of claims 33 to 38 further comprising water.

40. A binder composition according to any one of claims 33 to 39 having a dry binding strength of at least 3 N/mm^2, and having a wet binding strength of at least 1 N/mm^2, after 3 hours in water having a temperature of 80°C.

41. A binder composition according to claim 40, having a dry binding strength of at least 6 N/mm^2.

42. A binding composition according to claim 40 or claim 41 having a dry binding strength of at least 8 N/mm^2.

43. A binder composition according to any one of claims 40 to 42 having a wet binding strength of at least 2 N/mm^2.

44. Process for providing a bound mineral fibre product, comprising administering a binder resin or a binder composition according to any one of claims 20 to 43 to the mineral fibres and curing the binder.

45. Process according to claim 44 for providing glass wool or stone wool product.

46. Process according to claim 45, wherein glass or stone wool is prepared by spraying spun glass or stone with the binder composition and subsequently curing at temperatures between 150 and 300°C.

47. Mineral fibre product obtainable according to any one of claims 44 to 46.

48. Process for providing a water-soluble binder resin suitable for a mineral wool binder, substantially as herein described with reference to any one of the embodiments

of the invention as illustrated in the accompanying drawings and/or examples but excluding comparative examples.

49. A water-soluble binder resin suitable for binding mineral fibres, substantially as herein described with reference to any one of the embodiments of the invention as illustrated in the accompanying drawings and/or examples but excluding comparative examples.

50. A binder composition suitable for mineral fibres, substantially as herein described with reference to any one of the embodiments of the invention as illustrated in the accompanying drawings and/or examples but excluding comparative examples.

51. Process for providing a bound mineral fibre product, substantially as herein described with reference to any one of the embodiments of the invention as illustrated in the accompanying drawings and/or examples but excluding comparative examples.

DATED this 30th Day of April 2003
BALDWIN SHESLTON WATERS
ATTORNEYS FOR: ROCKWOOL INTERNATIONAL A/S

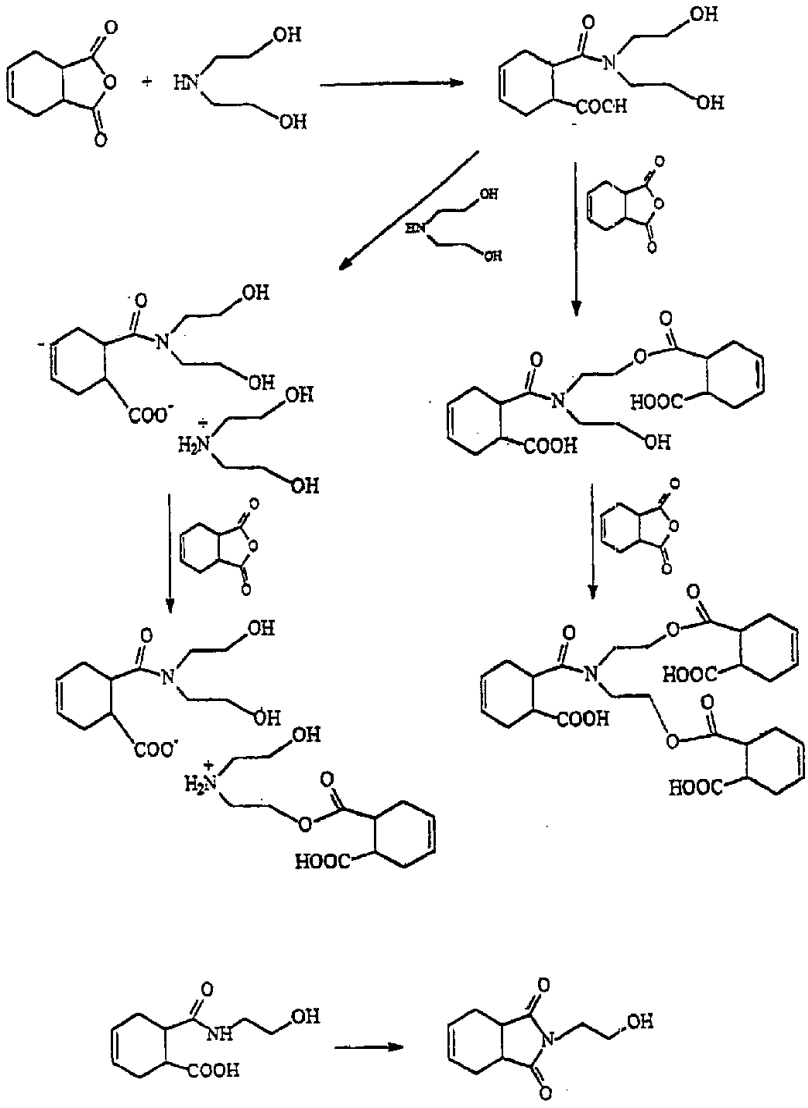


FIG. 1

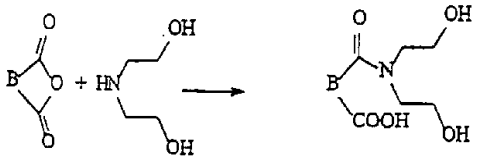


FIG. 2

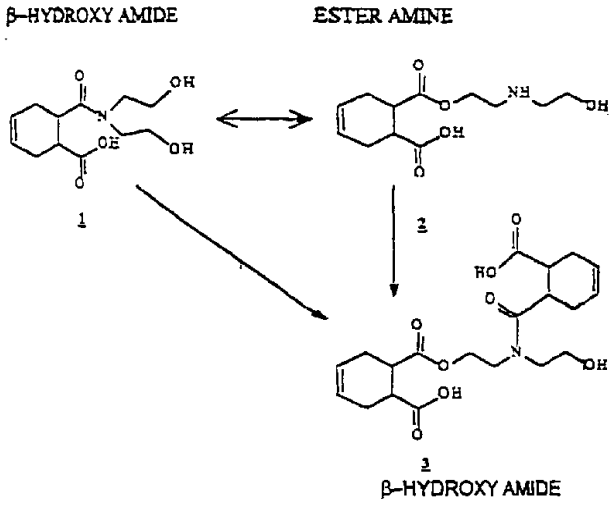


FIG. 3

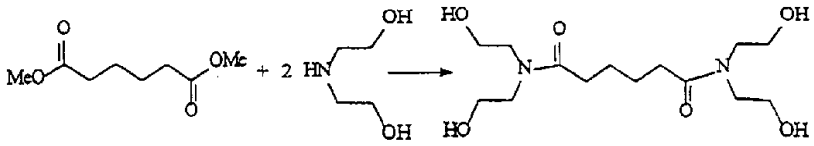


FIG. 4

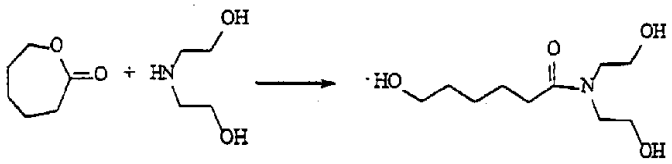


FIG. 5

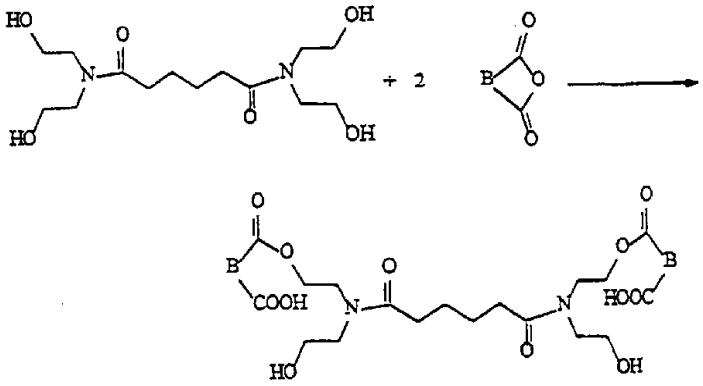


FIG. 6

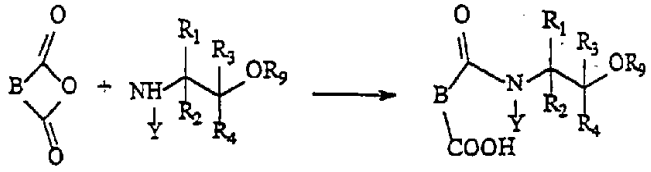


FIG. 7