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(54) Titre : ETHER AKLYLAMINO-AKLYLOXY (ALCOOL) MONOALKYLE POUR PROCEDE DE LAVAGE DE GAZ ACIDE  
(54) Title: ALKYLAMINO ALKYLOXY (ALCOHOL) MONOALKYL ETHER FOR ACID GAS SCRUBBING PROCESS

(57) **Abrégé/Abstract:**

An acid gas absorbent comprising an alkylamino alkyloxy (alcohol) monoalkyl ether and a process for the selective removal Of H<sub>2</sub>S from gaseous mixtures containing H<sub>2</sub>S and CO<sub>2</sub> using an absorbent solution comprising an alkylamino alkyloxy alcohol monoalkyl ether.



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(54) Title: ALKYLAMINO ALKYLOXY (ALCOHOL) MONOALKYL ETHER FOR ACID GAS SCRUBBING PROCESS

(57) Abstract: An acid gas absorbent comprising an alkylamino alkyloxy (alcohol) monoalkyl ether and a process for the selective removal Of H<sub>2</sub>S from gaseous mixtures containing H<sub>2</sub>S and CO<sub>2</sub> using an absorbent solution comprising an alkylamino alkyloxy alcohol monoalkyl ether.



**WO 2007/021462 A3**

ALKYLAMINO ALKYLOXY (ALCOHOL)  
MONOALKYL ETHER FOR ACID GAS SCRUBBING PROCESS

BACKGROUND OF THE INVENTION

FIELD OF THE INVENTION

[0001] The present invention relates to an absorbent composition and to a process for the selective absorption of one or more gaseous acidic component(s) from a normally gaseous mixture containing said gaseous acidic component(s) and gaseous non-acidic components and CO<sub>2</sub>- using the absorbent composition.

DESCRIPTION OF THE RELATED ART

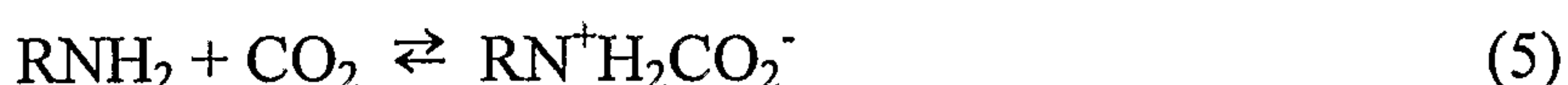
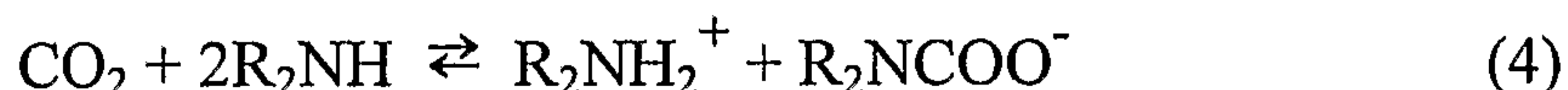
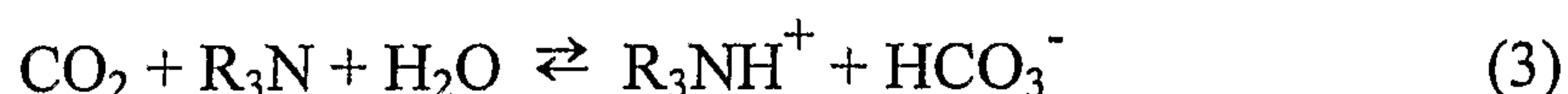
[0002] It is well known in the art to treat gases and liquids, such as mixtures containing acidic gases including CO<sub>2</sub>, H<sub>2</sub>S, CS<sub>2</sub>, HCN, COS and oxygen and sulfur derivatives of C<sub>1</sub> to C<sub>4</sub> hydrocarbons with amine solutions to remove these acidic gases. The amine usually contacts the acidic gases and the liquids as an aqueous solution containing the amine in an absorber tower with the aqueous amine solution contacting the acidic fluid countercurrently.

[0003] The treatment of acid gas mixtures containing, inter alia, CO<sub>2</sub> and H<sub>2</sub>S with amine solutions typically results in the simultaneous removal of substantial amounts of both the CO<sub>2</sub> and H<sub>2</sub>S. For example, in one such process generally referred to as the "aqueous amine process", relatively concentrated amine solutions are employed. A recent improvement of this process involves the use of sterically hindered amines as described in USP 4,112,052, to obtain nearly complete removal of acid gases such as CO<sub>2</sub> and H<sub>2</sub>S. This type of process may be used where the partial pressures of the CO<sub>2</sub> and related gases are low. Another process often used for specialized applications where the partial pressure of CO<sub>2</sub> is extremely high and/or where many acid gases are present, e.g., H<sub>2</sub>S, COS, CH<sub>3</sub>SH and CS<sub>2</sub> involves the use of an amine in combination with a physical absorbent, generally referred to as the "nonaqueous solvent

process". An improvement on this process involves the use of sterically hindered amines and organic solvents as the physical absorbent such as described in USP 4,112,051.

[0004] It is often desirable, however, to treat acid gas mixtures containing both CO<sub>2</sub> and H<sub>2</sub>S so as to remove the H<sub>2</sub>S selectively from the mixture, thereby minimizing removal of the CO<sub>2</sub>. Selective removal of H<sub>2</sub>S results in a relatively high H<sub>2</sub>S/CO<sub>2</sub> ratio in the separated acid gas which simplifies the conversion of H<sub>2</sub>S to elemental sulfur using the Claus process.

[0005] The typical reactions of aqueous secondary and tertiary amines with CO<sub>2</sub> and H<sub>2</sub>S can be represented as follows:



wherein each R is an organic radical which may be the same or different and may be substituted with an hydroxy group. The above reactions are reversible, and the partial pressures of both CO<sub>2</sub> and H<sub>2</sub>S are thus important in determining the degree to which the above reactions occur.

[0006] While selective H<sub>2</sub>S removal is applicable to a number of gas treating operations including treatment of hydrocarbon gases from shale pyrolysis, refinery gas and natural gas having a low H<sub>2</sub>S/CO<sub>2</sub> ratio, it is particularly desirable in the treatment of gases wherein the partial pressure of H<sub>2</sub>S is relatively low compared to that of CO<sub>2</sub> because the capacity of an amine to

absorb  $\text{H}_2\text{S}$  from the latter type gases is very low. Examples of gases with relatively low partial pressures of  $\text{H}_2\text{S}$  include synthetic gases made by coal gasification, sulfur plant tail gas and low-Joule fuel gases encountered in refineries where heavy residual oil is being thermally converted to lower molecular weight liquids and gases.

[0007] Although it is known that solutions of primary and secondary amines such as monoethanolamine (MEA), diethanolamine (DEA), dipropanolamine (DPA), and hydroxyethoxyethylamine (DGA) absorb both  $\text{H}_2\text{S}$  and  $\text{CO}_2$  gas, they have not proven especially satisfactory for preferential absorption of  $\text{H}_2\text{S}$  to the exclusion of  $\text{CO}_2$  because the amines undergo a facile reaction with  $\text{CO}_2$  to form carbamates as shown in Equations 5 and 6.

[0008] Diisopropanolamine (DIPA) is relatively unique among secondary aminoalcohols in that it has been used industrially, alone or with a physical solvent such as sulfolane, for selective removal of  $\text{H}_2\text{S}$  from gases containing  $\text{H}_2\text{S}$  and  $\text{CO}_2$ , but contact times must be kept relatively short to take advantage of the faster reaction of  $\text{H}_2\text{S}$  with the amine compared to the rate of  $\text{CO}_2$  reaction shown in Equations 2 and 4 hereinabove.

[0009] In 1950, Frazier and Kohl, Ind. and Eng. Chem., 42, 2288 (1950) showed that the tertiary amine, methyldiethanolamine (MDEA), has a high degree of selectivity toward  $\text{H}_2\text{S}$  absorption over  $\text{CO}_2$ . This greater selectivity was attributed to the relatively slow chemical reaction of  $\text{CO}_2$  with tertiary amines as compared to the rapid chemical reaction of  $\text{H}_2\text{S}$ . The commercial usefulness of MDEA, however, is limited because of its restricted capacity for  $\text{H}_2\text{S}$  loading and its limited ability to reduce the  $\text{H}_2\text{S}$  content to the level at low pressures which is necessary for treating, for example, synthetic gases made by coal gasification.

[0010] Recently, U.K. Patent Publication No. 2,017,524A to Shell disclosed that aqueous solutions of dialkylmonoalkanolamines, and particularly diethylmonoethanolamine (DEAE), have higher selectivity and capacity for H<sub>2</sub>S removal at higher loading levels than MDEA solutions. Nevertheless, even DEAE is not very effective for the low H<sub>2</sub>S loading frequency encountered in the industry. Also, DEAE has a boiling point of 161°C, and as such, it is characterized as being a low-boiling, relatively highly volatile amino alcohol. Such high volatilities under most gas scrubbing conditions result in large material losses with consequent losses in economic advantages.

[0011] U.S. Pat. Nos. 4,405,581; 4,405,583 and 4,405,585 disclose the use of severely sterically hindered amine compounds for the selective removal of H<sub>2</sub>S in the presence of CO<sub>2</sub>. Compared to aqueous methyldiethanolamine (MDEA) severely sterically hindered amines lead to much higher selectivity at high H<sub>2</sub>S loadings.

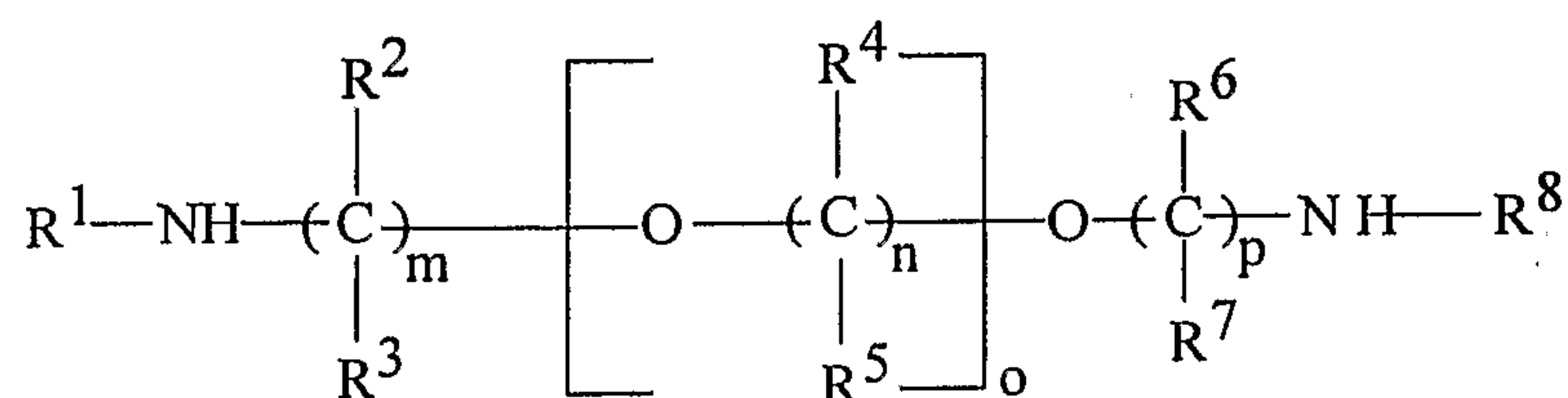
[0012] USP 4,487,967 discloses a catalytic synthesis process for selectively preparing severely sterically hindered secondary aminoether alcohols by reacting a primary amino compound with a polyalkenyl ether glycol in the presence of a hydrogenation catalyst at elevated temperatures and pressures.

[0013] USP 4,665,195 discloses a catalytic synthesis process for producing di-amino-polyalkenyl ethers by reacting (a) one or more acyclic or heterocyclic amino compounds with (b) one or more polyalkenyl ether glycols or polyalkenyl amino ether alcohols, in the presence of a hydrogenation catalyst at elevated temperatures and pressures.

[0014] The composition of BTEE has been disclosed in USP 4,405,583 and synthesized from tertiary-butylamine and bis-(2-chloroethoxy)-ethane. However, an aqueous BTEE solution suffered from phase separation under regeneration conditions (about 110°C). EEETB is disclosed as a new composi-

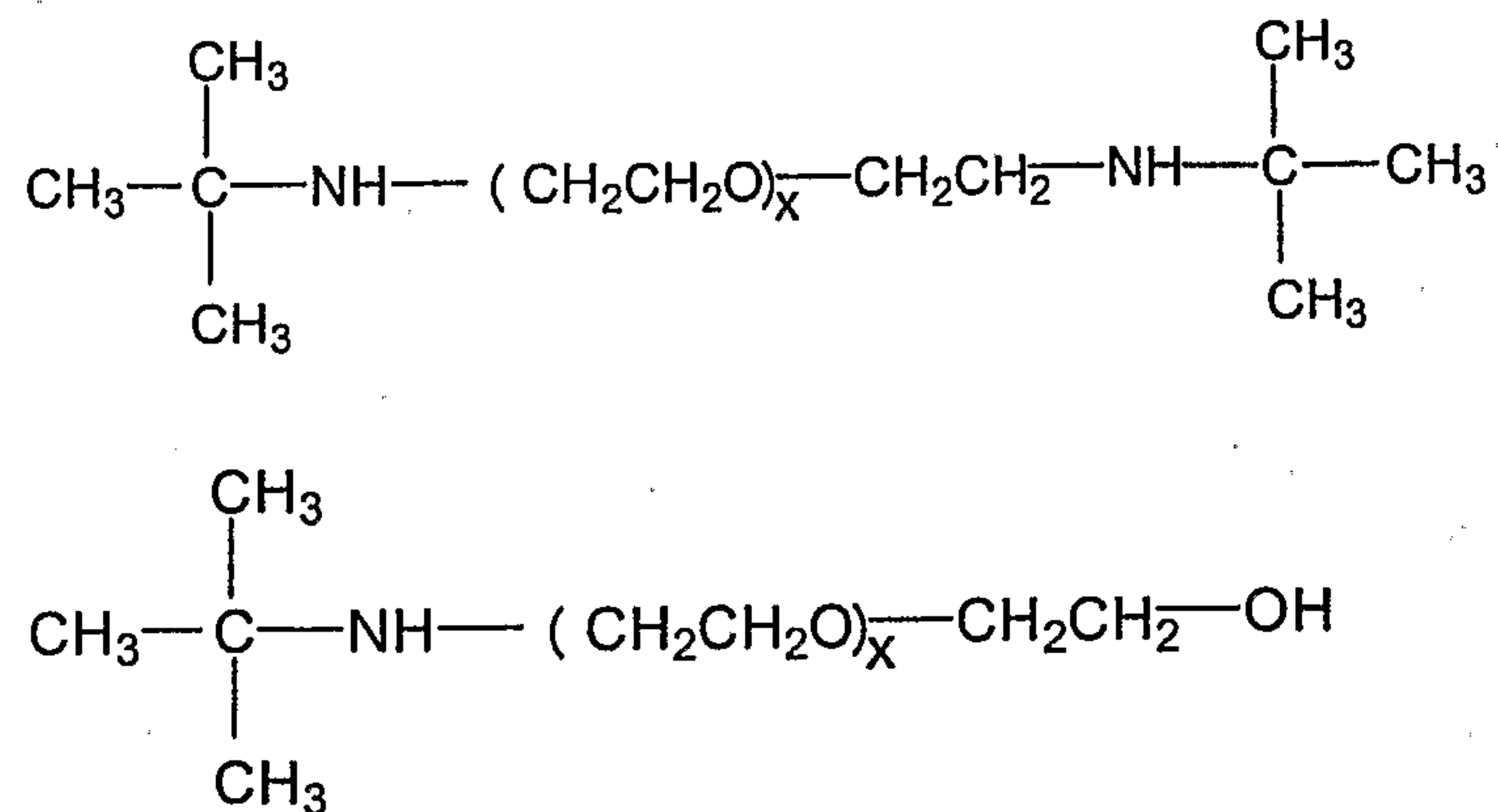
tion of matter in USP 4,471,138 and can be prepared from tertiary-butylamine and chloroethoxyethoxyethanol. EEETB in aqueous solution can be used for the selective removal of H<sub>2</sub>S in the presence of CO<sub>2</sub>. However, the BTEE/EEETB mixture gives a better selectivity and a higher capacity for H<sub>2</sub>S than EEETB. The mixture does not have phase separation under regeneration conditions, i.e., this mixture overcomes the phase separation problem of BTEE. The BTEE/EEETB mixture also gives higher selectivities for H<sub>2</sub>S than observed with the severely sterically hindered amines, e.g., ethoxyethanol-tertiary-butylamine (EETB), described in U.S. Pat. Nos. 4,405,581 and 4,405,585.

[0015] USP 4,417,075 teaches a class of di-secondary amino ethers of the formula



wherein R<sup>1</sup> and R<sup>8</sup> are each independently selected from the group consisting of primary alkyl having 1-8 carbon atoms, and primary hydroxy alkyl having 2-8 carbon atoms, secondary alkyl and secondary hydroxy alkyl radicals having 3-8 carbon atoms, tertiary alkyl and tertiary hydroxy alkyl radicals having 4 to 8 carbon atoms, R<sup>2</sup>, R<sup>3</sup>, R<sup>4</sup>, R<sup>5</sup>, R<sup>6</sup> and R<sup>7</sup> are each independently selected from the group consisting of hydrogen, C<sub>1</sub>-C<sub>3</sub> alkyl and hydroxyalkyl radicals, with that proviso that R<sup>2</sup>, R<sup>3</sup>, R<sup>6</sup> and R<sup>7</sup> are C<sub>1</sub>-C<sub>4</sub> alkyl or hydroxy alkyl radicals when R<sup>1</sup> and R<sup>8</sup> are primary alkyl or hydroxy alkyl radicals and at least one of R<sup>2</sup> or R<sup>3</sup> and R<sup>6</sup> and R<sup>7</sup> are C<sub>1</sub> to C<sub>3</sub> alkyl or hydroxyalkyl radicals when R<sup>1</sup> and R<sup>8</sup> are secondary alkyl radicals, m, n and p are positive integers ranging from 2 to 4 and a is either zero or a positive integer ranging from 1 to 10. These compounds are useful in the selective removal of H<sub>2</sub>S from gaseous mixtures containing H<sub>2</sub>S and CO<sub>2</sub>.

[0016] USP 4,894,178 teaches a mixture of two severely hindered amines with the following formula:



with x being an integer ranging from 2 to 6, and the weight ratio of the first amine to the second amine ranging from 0.43:1 to 2.3:1. This mixture can be prepared in the one-step synthesis, by the catalytic tertiary-butylamination of the polyalkenyl ether glycol,  $\text{HO}-(\text{CH}_2\text{CH}_2\text{O})_x-\text{CH}_2\text{CH}_2-\text{OH}$ . For example, a mixture of bis(tertiary-butylaminoethoxy)ethane (BTEE) and ethoxyethoxyethanol-tertiary-butylamine (EEETB) can be obtained by the catalytic tertiarybutylamination of triethylene glycol.

#### DESCRIPTION OF THE FIGURES

[0017] Figure 1 is a diagrammatic flow sheet illustrating an absorption - regeneration unit for selective removal of  $\text{H}_2\text{S}$  from gaseous streams containing  $\text{H}_2\text{S}$  and  $\text{CO}_2$ .

[0018] Figure 2 presents the effect of temperature on the synthesis of methoxy ethoxyethanol-t-butyl amine from t-butyl amine and diethyleneglycol monomethyl ether.

[0019] Figure 3 compares the level of product production from the catalytic reaction of methyl, ethyl and butyl capped diethylene glycol and mono-methyl capped triethylene glycol with t-butyl amine at  $225^\circ\text{C}$ .

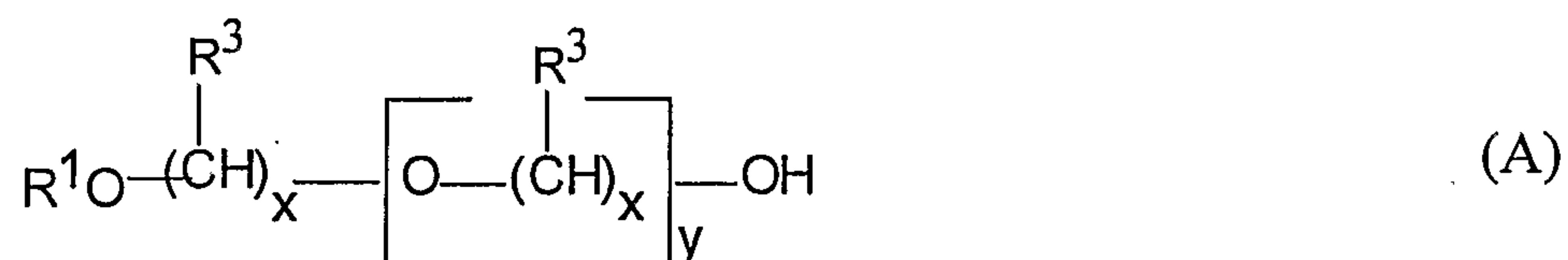
[0020] Figure 4 compares the level of product production from the catalytic reaction of triethylene glycol and mono-methyl capped triethylene glycol with t-butyl amine at 225°C.

### SUMMARY OF THE INVENTION

[0021] The present invention is directed to an absorbent made by the catalytic alkylation of an end capped, short chain alkyloxy glycol, i.e., a short chain alkyloxy alcohol monoalkylether, or a long chain polyalkylene glycol, both of which resist cyclization, to the absorbent per se, and to the use of such absorbent for the selective absorption of one or more gaseous acidic component(s) (e.g., H<sub>2</sub>S) from gaseous mixtures containing said gaseous acidic component(s) and gaseous non-acidic components and CO<sub>2</sub>.

### DETAILED DESCRIPTION OF THE INVENTION

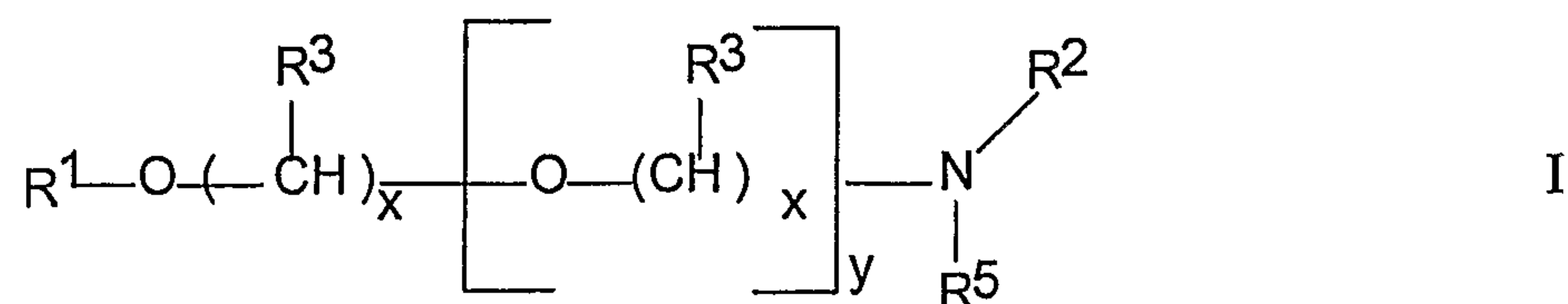
[0022] An alkylamine alkyloxy (alcohol) monoalkyl ether is produced by catalytically reacting an alkyloxy alcohol ether of the formula:



with an alkyl amine of the formula



to yield a product of the formula:



wherein

x is 1, 2, 3 or 4;

y is 1, 2, 3, 4 or 5, preferably 1 or 2; and

$R^1$  is a  $C_1$ - $C_9$  alkyl,  $C_3$ - $C_9$  branched alkyl,  $C_5$ - $C_9$  cycloalkyl,  $C_6$ - $C_{10}$  aryl, alkylaryl, or aryl alkyl, preferably  $C_1$ - $C_4$  alkyl,  $C_3$ - $C_4$  branched alkyl, more preferably  $C_1$ - $C_2$  alkyl, so that the short chain alkyloxy ether is an end capped alkyloxy alcohol monoalkyl ether and resists cyclization;

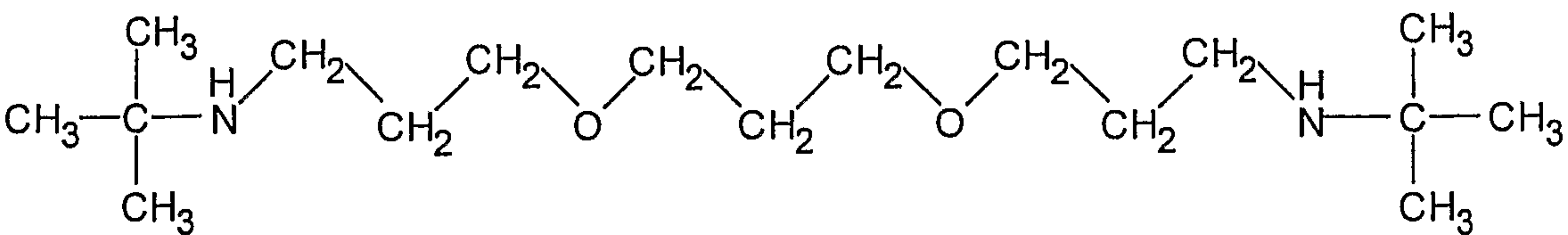
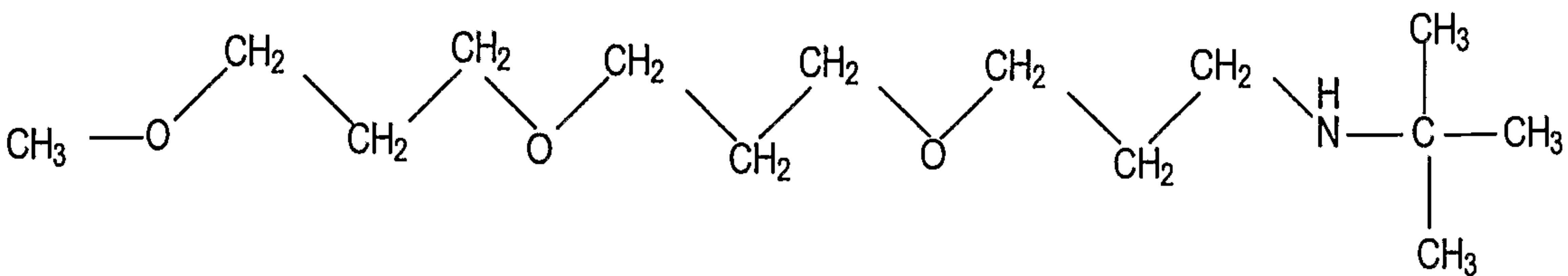
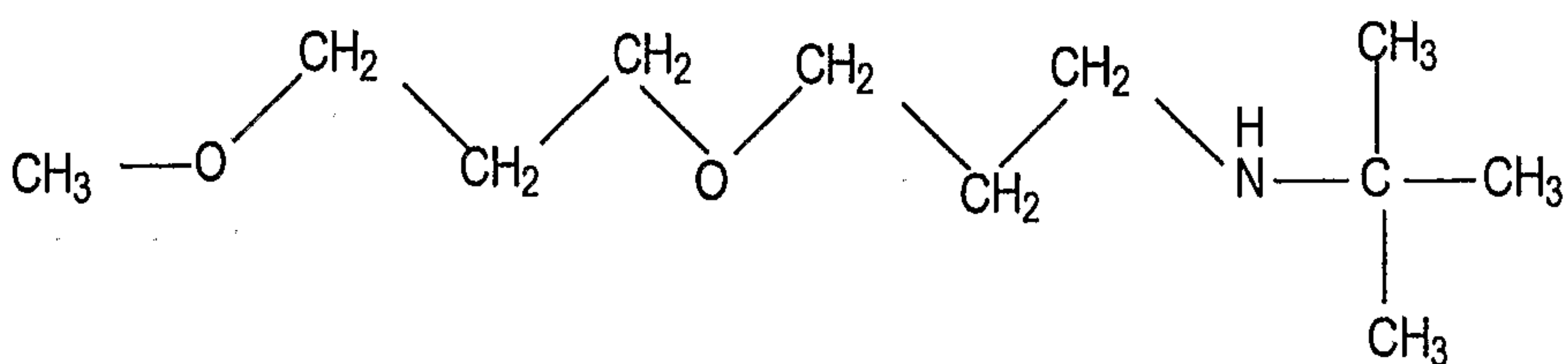
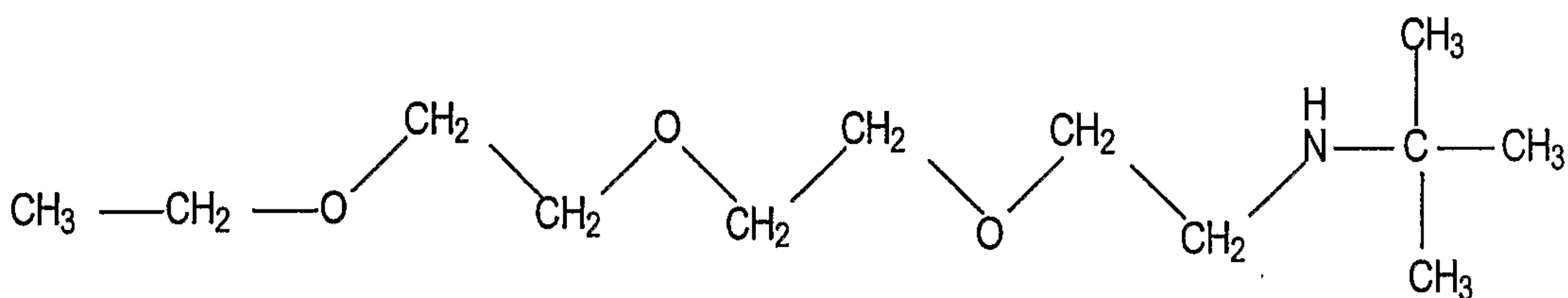
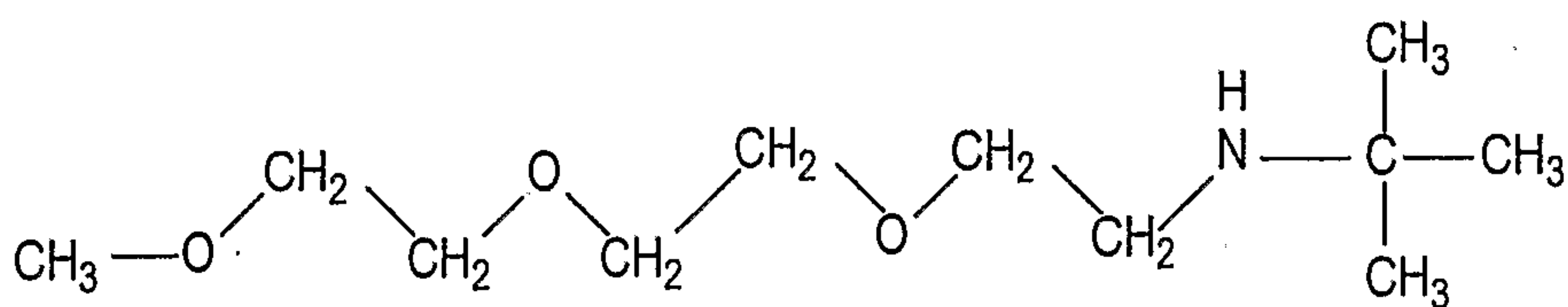
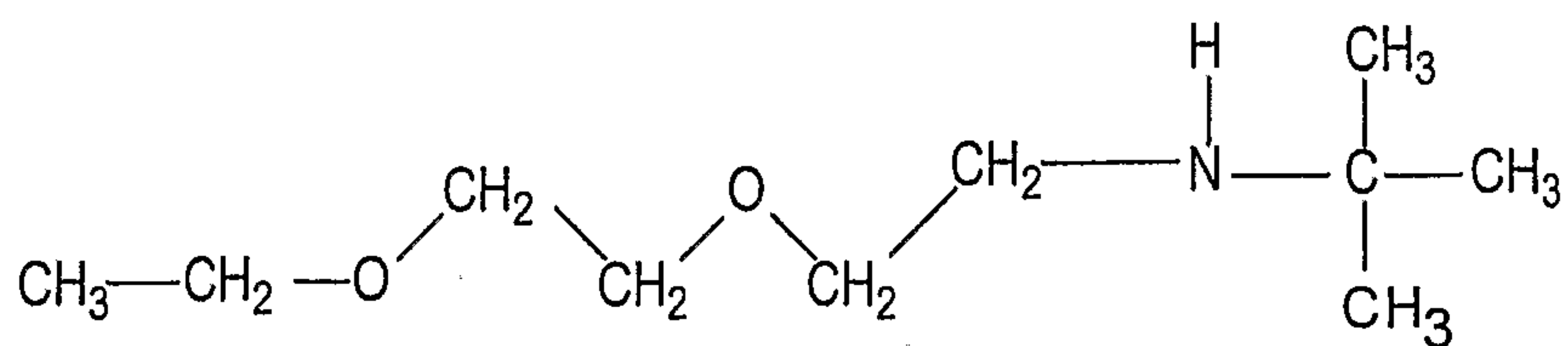
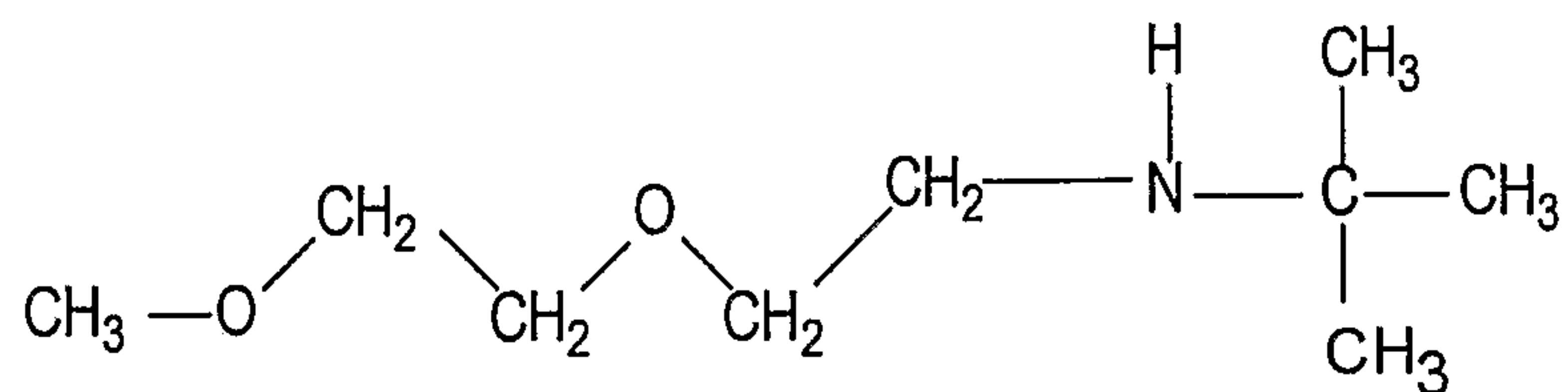
$R^2$  is a  $C_3$ - $C_9$  straight or branched alkyl, preferably a  $C_3$ - $C_6$  straight or branched alkyl, more preferably a  $C_3$ - $C_4$  straight or branched alkyl, preferably wherein N is attached to  $R^2$  through a secondary or tertiary carbon atom, preferably a tertiary carbon atom, most preferably the tertiary carbon atom of tert-butyl radical;

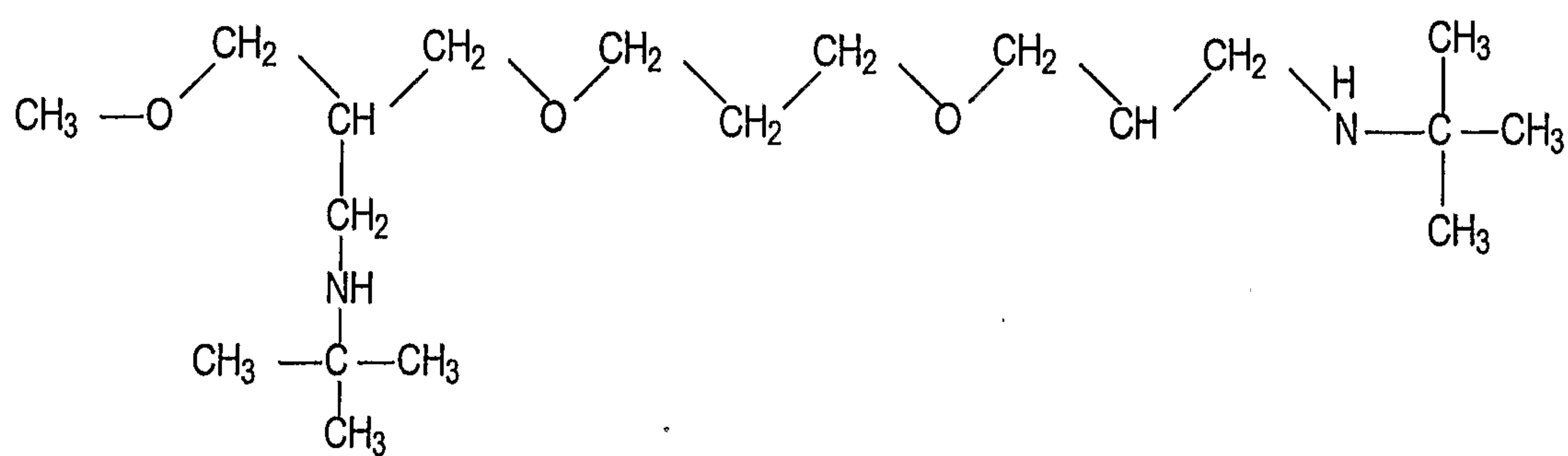
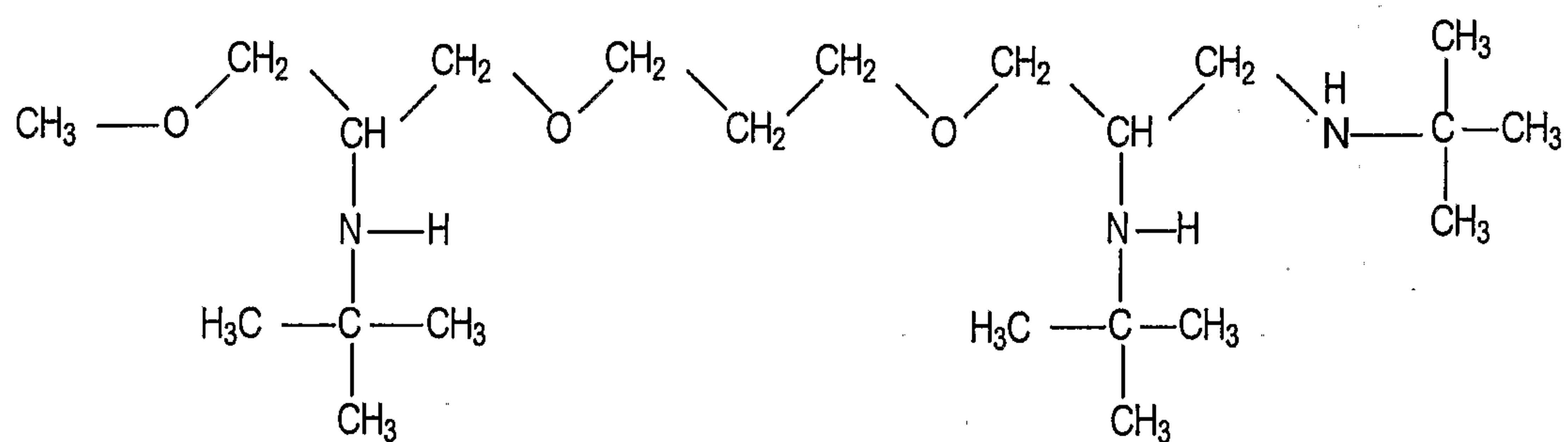
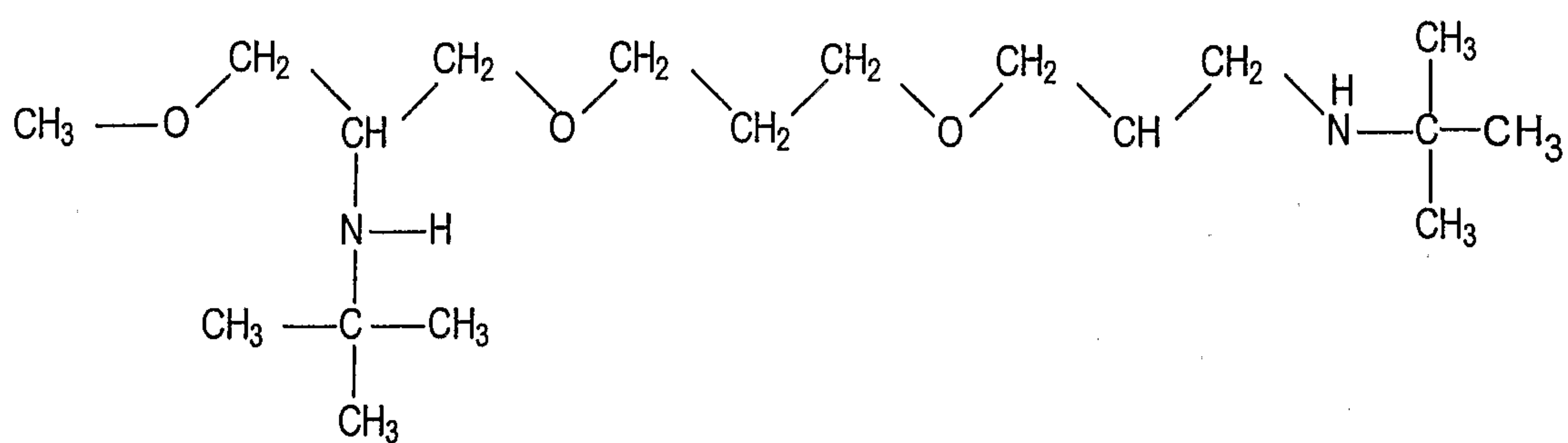
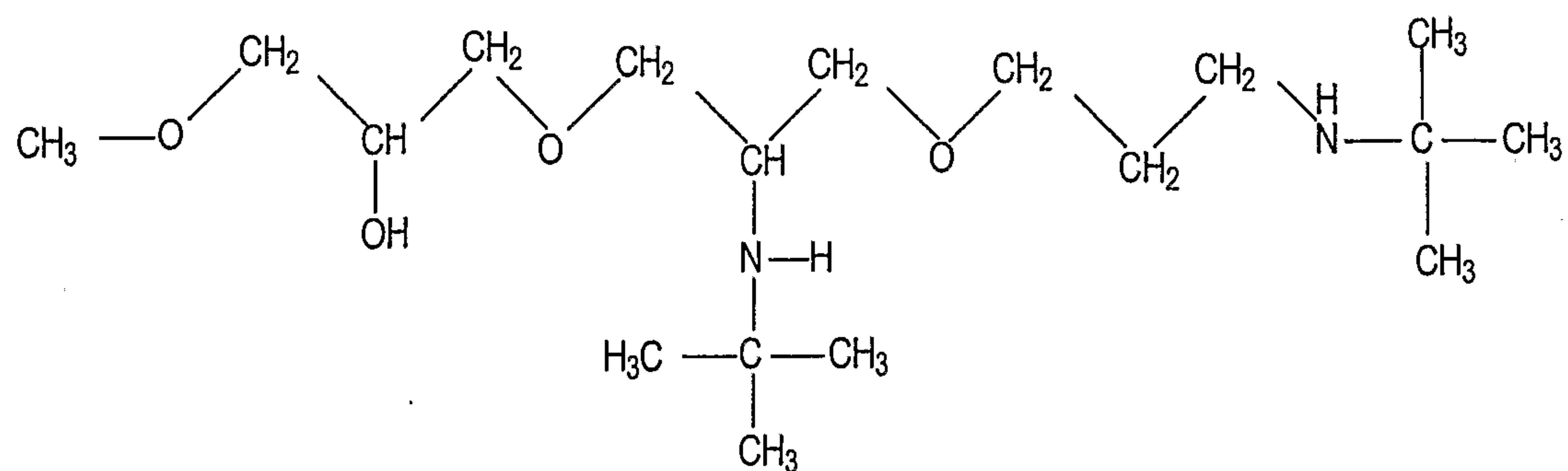
$R^5$  is hydrogen or a  $C_1$ - $C_6$  alkyl, preferably hydrogen or a  $C_1$ - $C_4$  alkyl, more preferably hydrogen or  $C_1$ - $C_2$  alkyl, most preferably hydrogen;

$R^3$  is H or a  $C_1$ - $C_5$  alkyl.

**[0023]** By end capping the short chain alkyloxy glycol, the reactant is an alkyloxy alcohol monoalkyl ether which is resistant to cyclization during the catalytic alkylamination step.

**[0024]** Desirable products include by way of example and not limitation:





[0025] The catalytic amination process of the one-step synthesis hereof is carried out under hydrogen pressure at a temperature ranging from about 160°C to about 425°C, preferably from about 180°C to about 400°C, and most preferably from about 190°C to about 250°C. The pressure in the reactor may range from about 50 to about 3000 psig, preferably from about 100 to about 1000 psig, and most preferably from about 150 to about 750 psig.

[0026] The reactor used may include any suitable vessel capable of withstanding the pressures necessary to carry out the amination process. Preferably, in the amination the reactants are passed over a fixed bed of the catalyst, either concurrently or counter-currently. Other reactors suitable for use herein include moving bed reactors and continuous stirred reactors. For example, in a continuous stirred reactor the catalyst is circulated and the reactants and reaction product are passed through the reaction vessel at a controlled rate.

[0027] The hydrogenation catalyst used in the amination process herein may include any of the known hydrogenation catalysts. Illustrative hydrogenation catalysts include platinum, palladium and other noble metals such as ruthenium, rhodium, osmium and iridium deposited on inert supports such as carbon, silica, alumina or other refractory oxides, Raney nickel, nickel-on-kieselguhr, nickel on inert support, massive nickel or nickel-cobalt or nickel-cobalt-copper coprecipitated with silicate and/or aluminum salts having alumina or kieselguhr supports. Preferred catalysts include coprecipitated nickel, nickel-cobalt, and

nickel-cobalt-copper supported on silica, alumina or a mixture thereof. Also preferred is platinum supported on alumina. Still more preferred are catalysts having increasing concentrations of nickel, about 40% to 70% nickel, by weight. Since preferred catalysts include those massive-metal coprecipitated hydrogenation catalysts described in U.S. Pat. Nos. 3,697,445; 4,251,394; 4,251,672; 4,263,173; 4,263,225; 4,273,680; 4,273,939; 4,307,248; 4,318,829; and the metal coprecipitated catalysts containing aluminum and silica disclosed and claimed in U.S. Ser. Nos. 388,966 and 388,967. It is preferred that the catalyst be reduced or activated by a reluctant, such as hydrogen prior to use in the amination reaction. This reduction or activation is typically carried out by passing hydrogen over the catalyst at temperatures ranging from 175°C to about 400°C, preferably 200°C to about 350°C.

[0028] The concentration of the hydrogenation catalyst is that which is catalytically effective and that amount will generally range from about 0.1 to about 10 wt%, based on the weight of the reactant charge. The normal pretreatment conditions and handling of the hydrogenation catalyst should be practiced as known to those skilled in the hydrogenation catalyst art. The mole ratio of amino compound to polyalkenyl ether glycol employed herein ranges from 1.2:1 to 4:1.

[0029] For purposes of this invention it may be desirable to include an inert solvent in the reaction medium. Preferably the solvent is a solvent such as a cyclic or linear ether or a hydrocarbon containing compound in which the reactants will dissolve. The solvent should be of relatively low molecular weight to facilitate its removal from the product of the reaction. The amount of the solvent may vary, but will generally range from about 10 to 50 wt%, preferably from 15 to 30 wt% based on the weight of the reactants used. Preferred solvents include tetrahydrofuran, dimethylether of ethylene glycol and toluene.

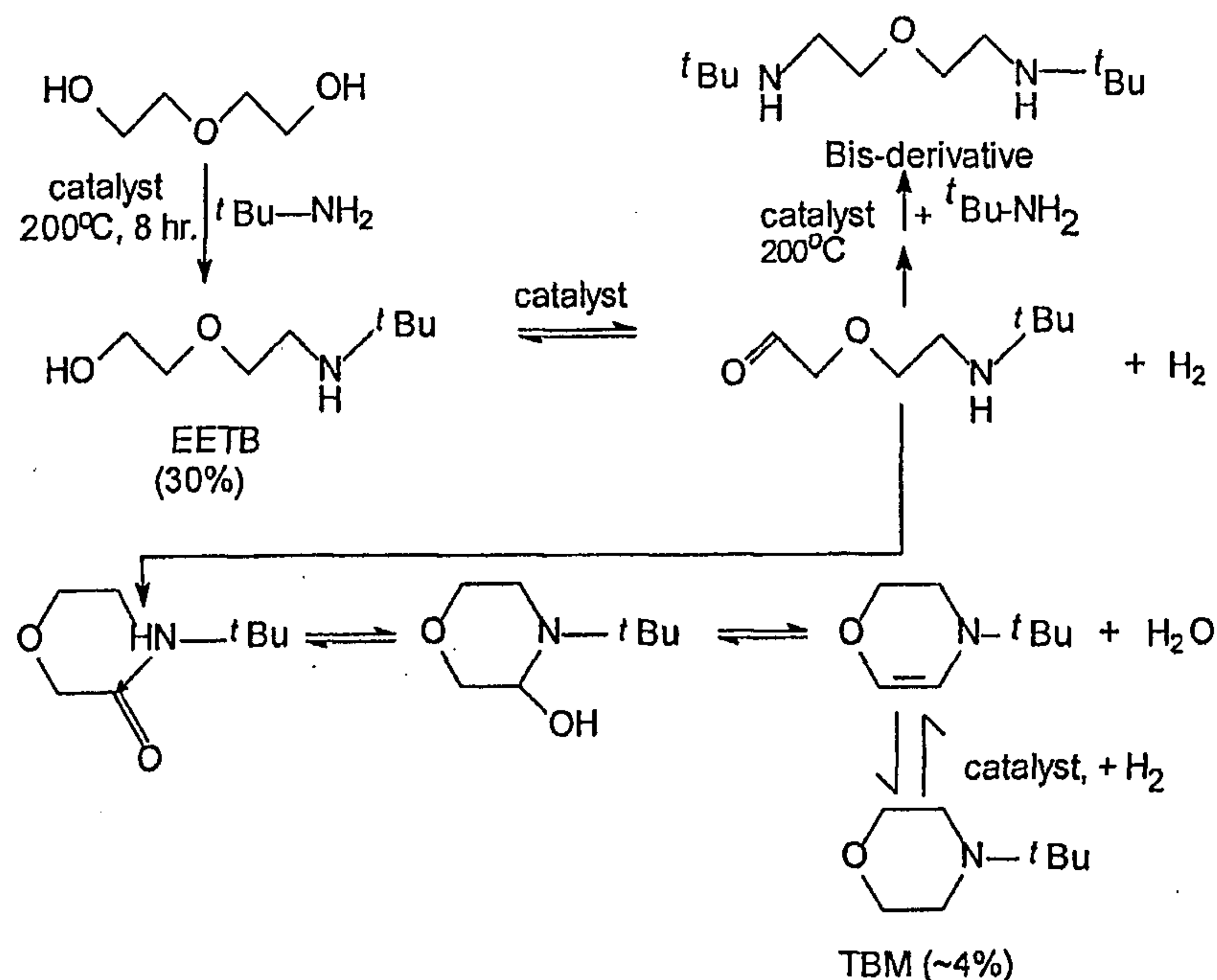
[0030] Reduction of the catalyst may be carried out in situ while conducting the process by the presence of hydrogen.

[0031] Once the reaction has been completed, the reaction product mixture can be conveniently recovered by known techniques such as solvent evaporation, the evaporation of volatile components, distillation and the like.

[0032] A preferred synthesis utilizes the catalyst as described in WO 05/81778 wherein the catalyst comprises a catalytically active metal on a support, the metal loaded catalyst characterized by having a BET surface area in the range of about 50 to about 1200 m<sup>2</sup>/g, the support characterized by a micropore volume above about 0.05 cm<sup>3</sup>/g, the metal loaded catalyst exhibiting a pore size distribution when normalized for pores of 19.99 nm or less of about 30% or more pores of up to 4.99 nm and the pores of 5 to up to 19.99 nm constituting the balance. The process utilizes an initial hydrogen pressure at room temperature of from zero to 300 psig, a temperature of about 150°C to about 350°C, a total reactor pressure at operating temperature of from 50 to 1500 psig, a reactive time of from 0.5 to 24 hours, and a molar ratio of reactant of formula B to reactant of formula (A) of from 2:1 to 5:1, preferably 2:1 to 4:1, more preferably 2:1 to 3:1. Preferably the catalyst metal is nickel, the catalyst comprising at about 2.5 to about 80% reduced metal based on the whole reduced catalyst.

[0033] The advantage of the alkylaminoalkyloxy alkyl ethers is that their synthesis is more efficient than that of ethoxyethanol-t-butyl amine (EETB) because undesirable byproducts are not formed when starting with, e.g., diethylene glycol monoalkyl ethers versus the synthesis of ethoxyethanol-t-butyl amine (EETB) which starts with diethylene glycol (DEG). In the synthesis of EETB from DEG and t-butyl amine in the presence of a catalyst, after t-butylamination of the first hydroxyl group of the DEG, dehydrogenation of the

second hydroxyl group to the aldehyde results in rapid cyclization with formation of the Schiff-Base/azomethine that is hydrogenated to t-butyl morpholine (TBM) which is an undesirable side product and to a small quantity of bis derivative (di-t-butyl aminoethyl ether). This is outlined below:



**[0034]** In the synthesis of EETB using a nickel catalyst conversion is limited to about 30% because of the negative economics associated with TBM formation. When the nickel catalyst of WO 05/81778 is used conversion to TBM is reduced and EETB production reaches about 60%.

**[0035]** By starting with the material of formula A the undesired cyclization side product TBM cannot form and bis-derivative also cannot form, therefore 90%+ conversion to desired product can be achieved in six hours versus the eight hours previously employed to reach only 30-60% product yield. Further, when starting with the material of Formula A it is not necessary to carry out the heretofore utilized distillation/purification procedure needed to recover the desired products from the undesired side products.

[0036] The absorbents described above exhibit high selectivity for gaseous acidic component, e.g., H<sub>2</sub>S removal and retain their high selectivity and loading capacity even after regeneration.

[0037] The absorbents are utilized for the selective absorption of one or more gaseous acidic components (e.g., H<sub>2</sub>S) from a normally gaseous mixture containing such gaseous acidic components (e.g., H<sub>2</sub>S) and gaseous non-acidic component and CO<sub>2</sub> comprising:

- (a) contacting said normally gaseous mixture with an absorbent solution characterized as capable of selectively absorbing gaseous acidic component (e.g., H<sub>2</sub>S) from said mixture;
- (b) regenerating, at least partially, said absorbent solution containing gaseous acidic component (e.g., H<sub>2</sub>S); and
- (c) recycling the regenerated solution for the selective absorption of gaseous acidic components (e.g., H<sub>2</sub>S) by contacting as in step (a).

Preferably, the regeneration step is carried out by heating and stripping and more preferably heating and stripping with steam.

[0038] The term "absorbent solution" as used herein includes but is not limited to solutions wherein the amino compound is dissolved in a solvent selected from water or a physical absorbent or mixtures thereof. Solvents which are physical absorbents (as opposed to the amino compounds which are chemical absorbents) are described, for example, in USP 4,112,051, and include, e.g., aliphatic acid amides, N-alkylated pyrrolidones, sulfones, sulfoxides, glycols and the mono- and diethers thereof. The preferred physical absorbents herein are sulfones, and most particularly, sulfolane. The preferred liquid medium comprises water.

[0039] The absorbent solution ordinarily has a concentration of amino compound of about 0.1 to 6 moles per liter of the total solution, and preferably 1

to 4 moles per liter, depending primarily on the specific amino compound employed and the solvent system utilized. If the solvent system is a mixture of water and a physical absorbent, the typical effective amount of the physical absorbent employed may vary from 0.1 to 5 moles per liter of total solution, and preferably from 0.5 to 3 moles per liter, depending mainly on the type of amino compound being utilized. The dependence of the concentration of amino compound on the particular compound employed is significant because increasing the concentration of amino compound may reduce the basicity of the absorbent solution, thereby adversely affecting its selectivity for H<sub>2</sub>S removal, particularly if the amino compound has a specific aqueous solubility limit which will determine maximum concentration levels within the range given above. It is important, therefore, that the proper concentration level appropriate for each particular amino compound be maintained to insure satisfactory results.

[0040] The solution of this invention may include a variety of additives typically employed in selective gas removal processes, e.g., antifoaming agents, antioxidants, corrosion inhibitors, and the like. The amount of these additives will typically be in the range that they are effective, i.e., an effective amount.

[0041] Also, the amino compounds described herein may be admixed with other amino compounds as a blend. The ratio of the respective amino compounds may vary widely, for example, from 1 to 99 wt% of the amino compounds described herein.

[0042] Three characteristics which are of ultimate importance in determining the effectiveness of the amino compounds herein for H<sub>2</sub>S removal are "selectivity", "loading" and "capacity". The term "selectivity" as used throughout the specification is defined as the following mole ratio fraction:

$$\frac{(\text{moles of H}_2\text{S}/\text{moles of CO}_2) \text{ in liquid phase}}{(\text{moles of H}_2\text{S}/\text{moles of CO}_2) \text{ in gaseous phase}}$$

The higher this fraction, the greater the selectivity of the absorbent solution for the  $\text{H}_2\text{S}$  in the gas mixture.

[0043] By the term “loading” is meant the concentration of the  $\text{H}_2\text{S}$  and  $\text{CO}_2$  gases physically dissolved and chemically combined in the absorbent solution as expressed in moles of gas per moles of the amine. The best amino compounds are those which exhibit good selectivity up to a relatively high loading level. The amino compounds used in the practice of the present invention typically have a “selectivity” of not substantially less than 10 at a “loading” of 0.1 moles, preferably, a “selectivity” of not substantially less than 10 at a loading of 0.2 or more moles of  $\text{H}_2\text{S}$  and  $\text{CO}_2$  per moles of the amino compound.

[0044] “Capacity” is defined as the moles of  $\text{H}_2\text{S}$  loaded in the absorbent solution at the end of the absorption step minus the moles of  $\text{H}_2\text{S}$  loaded in the absorbent solution at the end of the desorption step. High capacity enables one to reduce the amount of amine solution to be circulated and use less heat or steam during regeneration.

[0045] The acid gas mixture herein necessarily includes  $\text{H}_2\text{S}$ , and may optionally include other gases such as  $\text{CO}_2$ ,  $\text{N}_2$ ,  $\text{CH}_4$ ,  $\text{H}_2$ ,  $\text{CO}$ ,  $\text{H}_2\text{O}$ ,  $\text{COS}$ ,  $\text{HCN}$ ,  $\text{C}_2\text{H}_4$ ,  $\text{NH}_3$ , and the like. Often such gas mixtures are found in combustion gases, refinery gases, town gas, natural gas syn gas, water gas, propane, propylene, heavy hydrocarbon gases, etc. The absorbent solution herein is particularly effective when the gaseous mixture is a gas, obtained, for example, from shale oil retort, coal liquefaction or gasification, gasification of heavy oil with steam, air/steam or oxygen/steam, thermal conversion of heavy residual oil to lower molecular weight liquids and gases, e.g., fluid coker, Flexicoker, or delayed coker or in sulfur plant tail gas cleanup operations.

[0046] The absorption step of this invention generally involves contacting the normally gaseous stream with the absorbent solution in any suitable contacting

vessel. In such processes, the normally gaseous mixture containing  $\text{H}_2\text{S}$  and  $\text{CO}_2$  from which the  $\text{H}_2\text{S}$  is to be selectively removed may be brought into intimate contact with the absorbent solution using conventional means, such as a tower or vessel packed with, for example, rings or with sieve plates, or a bubble reactor. Other acidic gaseous components will also be removed preferentially as compared to the  $\text{CO}_2$ .

[0047] In a typical mode of practicing the invention, the absorption step is conducted by feeding the normally gaseous mixture into the lower portion of the absorption tower while fresh absorbent solution is fed into the upper region of the tower. The gaseous mixture, freed largely from the  $\text{H}_2\text{S}$ , emerges from the upper portion of the tower, and the loaded absorbent solution, which contains the selectively absorbed  $\text{H}_2\text{S}$ , leaves the tower near or at its bottom. Preferably, the inlet temperature of the absorbent solution during the absorption step is in the range of from about  $20^\circ\text{C}$  to about  $100^\circ\text{C}$ , and more preferably from  $30^\circ\text{C}$  to about  $60^\circ\text{C}$ . Pressures may vary widely; acceptable pressures are between 5 and 2000 psia, preferably 20 to 1500 psia, and most preferably 25 to 1000 psia in the absorber. The contacting takes place under conditions such that the  $\text{H}_2\text{S}$  is selectively absorbed by the solution. The absorption conditions and apparatus are designed so as to minimize the residence time of the liquid in the absorber to reduce  $\text{CO}_2$  pickup while at the same time maintaining sufficient residence time of gas mixture with liquid to absorb a maximum amount of the  $\text{H}_2\text{S}$  gas. The amount of liquid required to be circulated to obtain a given degree of  $\text{H}_2\text{S}$  removal will depend on the chemical structure and basicity of the amino compound and on the partial pressure of  $\text{H}_2\text{S}$  in the feed gas. Gas mixtures with low partial pressures such as those encountered in thermal conversion processes will require more liquid under the same absorption conditions than gases with higher partial pressures such as shale oil retort gases.

[0048] A typical procedure for the selective H<sub>2</sub>S removal phase of the process comprises selectively absorbing H<sub>2</sub>S via countercurrent contact of the gaseous mixture containing H<sub>2</sub>S and CO<sub>2</sub> with the solution of the amino compound in a column containing a plurality of trays at a low temperature, e.g., below 45°C, and at a gas velocity of at least about 0.3 ft/sec (based on "active" or aerated tray surface), depending on the operating pressure of gas, said tray column having fewer than 20 contacting trays, with, e.g., 4-16 trays being typically employed.

[0049] After contacting the normally gaseous mixture with the absorbent solution, which becomes saturated or partially saturated with H<sub>2</sub>S, the solution may be at least partially regenerated so that it may be recycled back to the absorber. As with absorption, the regeneration may take place in a single liquid phase. Regeneration or desorption of the absorbent solution may be accomplished by conventional means such as pressure reduction of the solution or increase of temperature to a point at which the absorbed H<sub>2</sub>S flashes off, or bypassing the solution into a vessel of similar construction to that used in the absorption step, at the upper portion of the vessel, and passing an inert gas such as air or nitrogen or preferably steam upwardly through the vessel. The temperature of the solution during the regeneration step should be in the range from about 50°C to about 170°C, and preferably from about 80°C to 120°C, and the pressure of the solution on regeneration should range from about 0.5 to about 100 psia, preferably 1 to about 50 psia. The absorbent solution, after being cleansed of at least a portion of the H<sub>2</sub>S gas, may be recycled back to the absorbing vessel. Makeup absorbent may be added as needed.

[0050] In the preferred regeneration technique, the H<sub>2</sub>S-rich solution is sent to the regenerator wherein the absorbed components are stripped by the steam which is generated by re-boiling the solution. Pressure in the flash drum and stripper is usually 1 to about 50 psia, preferably 15 to about 30 psia, and the temperature is typically in the range from about 50°C to 170°C, preferably about

80°C to 120°C. Stripper and flash temperatures will, of course, depend on stripper pressure, thus at about 15 to 30 psia stripper pressures, the temperature will be about 80°C to about 120°C during desorption. Heating of the solution to be regenerated may very suitably be effected by means of indirect heating with low-pressure steam. It is also possible, however, to use direct injection of steam.

[0051] In one embodiment for practicing the entire process herein, as illustrated in Figure 1, the gas mixture to be purified is introduced through line 1 into the lower portion of a gas-liquid countercurrent contacting column 2, said contacting column having a lower section 3 and an upper section 4. The upper and lower sections may be segregated by one or a plurality of packed beds as desired. The absorbent solution as described above is introduced into the upper portion of the column through a pipe 5. The solution flowing to the bottom of the column encounters the gas flowing countercurrently and dissolves the H<sub>2</sub>S preferentially. The gas freed from most of the H<sub>2</sub>S exits through a pipe 6, for final use. The solution, containing mainly H<sub>2</sub>S and some CO<sub>2</sub>, flow toward the bottom portion of the column, from which it is discharged through pipe 7. The solution is then pumped via optional pump 8 through an optional heat exchanger and cooler 9 disposed in pipe 7, which allows the hot solution from the regenerator 12 to exchange heat with the cooler solution from the absorber column 2 for energy conservation. The solution is entered via pipe 7 to a flash drum 10 equipped with a line (not shown) which vents to line 13 and then introduced by pipe 11 into the upper portion of the regenerator 12, which is equipped with several plates and effects the desorption of the H<sub>2</sub>S and CO<sub>2</sub> gases carried along in the solution. This acid gas is passed through a pipe 13 into a condenser 14 wherein cooling and condensation of water and amine solution from the gas occur. The gas then enters a separator 15 where further condensation is effected. The condensed solution is returned through pipe 16 to the upper portion of the regenerator 12. The gas remaining from the condensation, which contains H<sub>2</sub>S and some CO<sub>2</sub>, is removed through pipe 17 for final

disposal (e.g., to a vent or incinerator or to an apparatus which converts the  $\text{H}_2\text{S}$  to sulfur, such as a Claus unit or a Stretford conversion unit (not shown)).

[0052] The solution is liberated from most of the gas which it contains while flowing downward through the regenerator 12 and exits through pipe 18 at the bottom of the regenerator for transfer to a reboiler 19. Reboiler 19, equipped with an external source of heat (e.g., steam injected through pipe 20 and the condensate exits through a second pipe (not shown)), vaporizes a portion of this solution (mainly water) to drive further  $\text{H}_2\text{S}$  therefrom. The  $\text{H}_2\text{S}$  and steam driven off are returned via pipe 21 to the lower section of the regenerator 12 and exited through pipe 13 for entry into the condensation stages of gas treatment. The solution remaining in the reboiler 19 is drawn through pipe 22, cooled in heat exchanger 9, and introduced via the action of pump 23 (optional if pressure is sufficiently high) through pipe 5 into the absorber column 2.

[0053] Typically, a gaseous stream to be treated having a 1:10 mole ratio of  $\text{H}_2\text{S}:\text{CO}_2$  from an apparatus for thermal conversion of heavy residual oil, or a Lurgi coal gas having a mole ratio of  $\text{H}_2\text{S}:\text{CO}_2$  of less than 1:10 will yield an acid gas having a mole ratio of  $\text{H}_2\text{S}:\text{CO}_2$  of about 1:1 after treatment by the process of the present invention. The process herein may be used in conjunction with another  $\text{H}_2\text{S}$  selective removal process; however, it is preferred to carry out the process of this invention by itself, since the amino compounds are extremely effective by themselves in preferential absorption of  $\text{H}_2\text{S}$ .

#### EXPERIMENTAL PROCEDURE

1. Absorption tests were carried out at  $35^\circ\text{C}$  on 0.15 M aqueous solutions of absorbent using a test gas mixture of nitrogen:carbon dioxide:hydrogen sulfide of 89:10:1, for 2 hours.
2. Desorption was run at  $85^\circ\text{C}$  in flowing  $\text{N}_2$  for 2 hours at the same flow rate as the test gas mixture.

Legend for Table and Figures

|                                               |                                             |
|-----------------------------------------------|---------------------------------------------|
| DEG = Diethylene glycol                       | EETB = Ethoxyethanol-t-butylamine           |
| TEG = Triethylene glycol                      | MEETB = MethoxyEETB                         |
| DEGM = Diethylene glycol<br>monomethyl ether  | EEETB = EthoxyEETB                          |
| TEGM = Triethylene glycol<br>monomethyl ether | BEETB = ButyloxyEETB                        |
| DEGE = Diethylene glycol<br>monoethyl ether   | MEEETB = MethoxyTEGTB                       |
| TBM = t-Butylmorpholine                       | TEGTB = Triethylene glycol-t-<br>butylamine |
| TBA = t-Butyl amine                           | Bis-SE = Bis-t-butylaminoDEG                |
|                                               | BIS-TEGTB = Bis-t-butylaminoTEG             |

ABSORPTION/REGENERATION EXAMPLES

TABLE 1. SUMMARY OF ABSORPTION TESTING RESULTS

| Compound                                                | Molecular Weight   | Selectivity | Loading (%) | Capacity (%) | Selectivity - Reabsorption |
|---------------------------------------------------------|--------------------|-------------|-------------|--------------|----------------------------|
| EETB (USP 4,405,585)                                    | 161.24             | 15.4        | 16.3        | 60           | 13.3                       |
| Bis-SE (USP 4,405,583)                                  | 216.36             | 16.7        | 28.2        | 80           | 25.2                       |
| MEETB                                                   | 175                | 56.01       | 21.57       | 67.4         | 59.6                       |
| EEETB                                                   | 189.30             | 75.4        | 13.1        | 89.3         | 86.7                       |
| BEETB                                                   | 217.35             | 90.2        | 16.8        | 92.5         | 197.6                      |
| MEEETB                                                  | 219.32             | 64.4        | 24.2        | 98.4         | 69.7                       |
| TEGTB (32.2%):<br>[Bis-TEGTB](67.4%)<br>(USP 4,894,178) | 205.26 /<br>260.42 | 128.2       | 45.4        | 82.6         | 231.2                      |
| TBA-NH-(CH <sub>2</sub> ) <sub>2</sub> -HN-TBA          | 172.31             | 26.2        | 9.8         | 46           | 20.2                       |

Selectivity = (H<sub>2</sub>S/CO<sub>2</sub>) in solution / (H<sub>2</sub>S/CO<sub>2</sub>) in feed gas

Loading = Moles of H<sub>2</sub>S / Moles of Compound

Capacity = 
$$\frac{\text{Moles of H}_2\text{S absorbed by solution} - \text{Moles of H}_2\text{S after desorption from solution}}{\text{Moles of H}_2\text{S absorbed by solution}}$$

[0054] As can be seen, the methyl, ethyl and butyl substituted diethylene and triethylene glycol-t-butyl amines have higher degrees of selectivity as compared

against the EETB and Bis-derivative of the literature, have at least equivalent and in most cases superior capacity and superior selectivity after regeneration than the EETB and Bis-derivative of the literature.

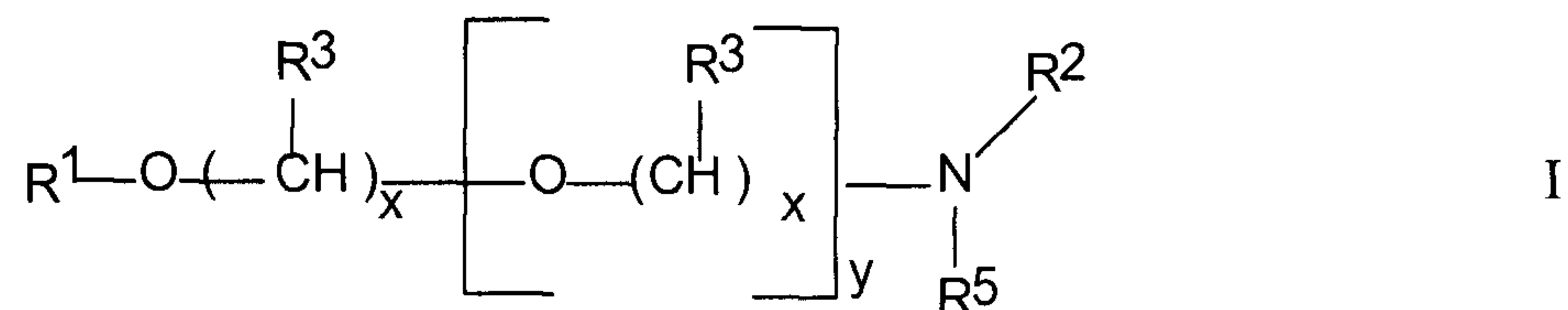
#### SYNTHESIS EXAMPLES

- a. Tertiary butyl amine (TBA) in an amount of 109.9 g was combined with monomethoxydiethylene glycol in an amount of 91.2 g (2:1 mole ratio) in an autoclave under nitrogen at room temperature. 0.503 g (0.25 wt%) of fresh nickel loaded catalyst meeting the requirements recited in WO 05/81778 was employed and added to the autoclave. The catalyst contains 64% nickel with a degree of reduction of 0.35 (Example 1 (b) of WO 05/81778). Starting hydrogen pressure at room temperature was 100 psi. Three separate runs were performed at 200°C, 220°C and 225°C with stirring at 1800 rpm, samples taken every hour. The result are presented in Figure 2 showing that yield improved with increasing temperature. When product yield achieves at least about 80% as adjusted to take into account unreacted tertiary-butyl amine, the effluent from the reactor can be utilized in the H<sub>2</sub>S sorption reaction without purification or separation, the product being essentially devoid of undesirable byproduct.
- b. Tertiary-butyl amine (TBA) was reacted separately with portions of mono methoxy diethylene glycol, mono ethoxy diethylene glycol, monobutoxy diethylene glycol and mono methoxy triethylene glycol (2:1 mole ratio) using the procedure of Example (a) above, but at 225°C. In each instance product yield (as adjusted to take into account unreacted TBA), achieved yields of 90% or more in from 5 to 7 hours as presented in Figure 3. By achieving this level of production the need for separate, time consuming purification steps are eliminated.

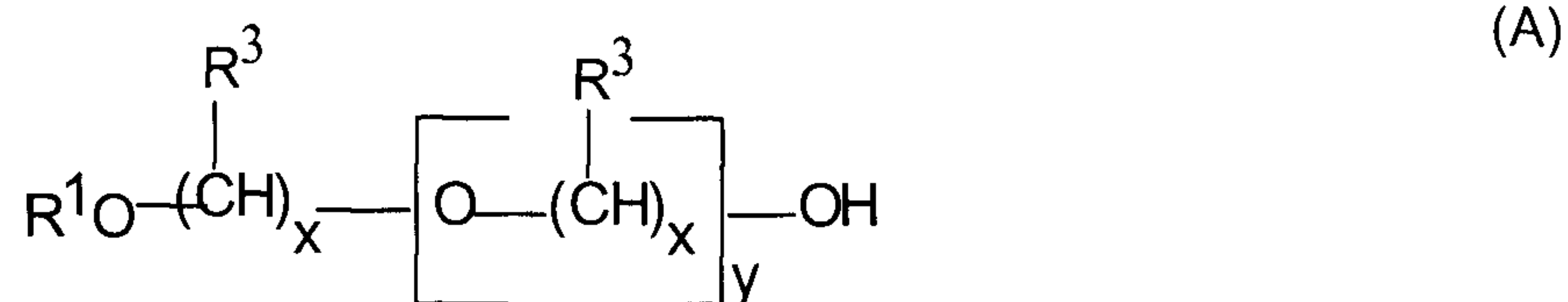
- c. Tertiary-butyl amine was reacted individually with triethylene glycol and monomethoxy triethylene glycol (2:1 mole ratio) over 0.25 wt% of the catalyst of Example (a) at 225°C. After 5 hours about 95% yield of the mono methoxy triethylene glycol-t-butyl amine was produced. Over the same 5 hours the yield of product from the triethylene glycol was about 55% triethylene glycol-t-butyl amine and 10-12% bis-(t-butylamino) triethylene glycol (for a total of about 65-67% yield). However, over time, as the run duration with the triethylene glycol was extended, e.g., 22 hours, the yield of the triethylene glycol-t-butyl amine decreased as it is converted into bis-(t-butylamino) triethylene glycol the yield of which increased to about 63% yield for a combined yield of about 95% or more (Figure 4).

**CLAIMS:**

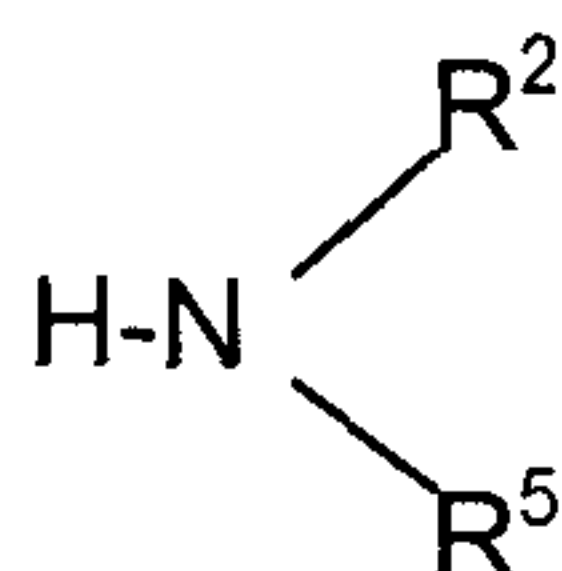
1. A process for producing absorbent materials of the formula



by catalytically reacting an alkyloxy alcohol of the formula



with an alkyl amine of the formula



wherein

x is 2, 3 or 4;

y is 1, 2 or 3;

R<sup>1</sup> is C<sub>1</sub>-C<sub>9</sub> alkyl, C<sub>3</sub>-C<sub>9</sub> branched alkyl, C<sub>5</sub>-C<sub>9</sub> cycloalkyl, C<sub>6</sub>-C<sub>10</sub> aryl, alkylaryl, or aryl alkyl;

R<sup>2</sup> is a C<sub>3</sub>-C<sub>9</sub> straight or branched alkyl wherein N is attached to R<sup>2</sup> through a secondary or tertiary carbon atom of R<sup>2</sup>;

R<sup>5</sup> is hydrogen, or C<sub>1</sub>-C<sub>6</sub> alkyl; and

R<sup>3</sup> is hydrogen or C<sub>1</sub>-C<sub>5</sub> alkyl.

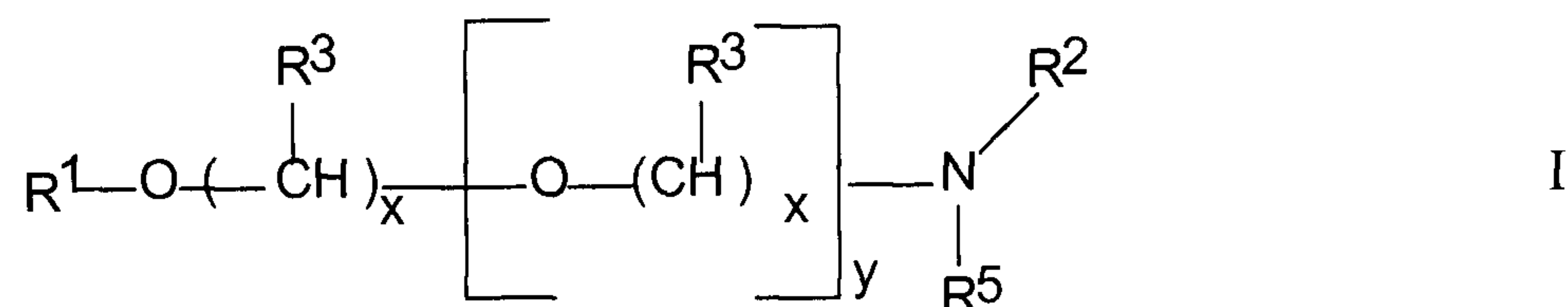
2. The process of claim 1 wherein

R<sup>1</sup> is C<sub>1</sub>-C<sub>4</sub> alkyl, or C<sub>3</sub>-C<sub>4</sub> branched alkyl,

R<sup>2</sup> is C<sub>3</sub>-C<sub>6</sub> straight or branched alkyl, and

R<sup>5</sup> is hydrogen or C<sub>1</sub>-C<sub>4</sub> alkyl.

3. The process of claim 1 wherein x is 2 and y is 1, 2 or 3.
4. The process of claim 2 wherein x is 2 and y is 1 or 2.
5. The process of claim 3 wherein  $R^3$  is hydrogen and  $R^2$  is a C<sub>4</sub>-tertiary alkyl wherein N is attached to  $R^2$  through the tertiary carbon atom of  $R^2$ .
6. The process of claim 4 wherein  $R^3$  is hydrogen and  $R^2$  is a C<sub>4</sub>-tertiary alkyl wherein N is attached to  $R^2$  through the tertiary carbon atom of  $R^2$ .
7. The process of claim 1 wherein the catalyst comprises a catalytically active metal on a support the metal loaded catalyst characterized by having a BET surface area in the range of about 50 to about 1200 m<sup>2</sup>/g, the support characterized by a micropore volume above about 0.05 cm<sup>3</sup>/g, the metal loaded catalyst exhibiting a pore size distribution when normalized for pores of 19.99 nm or less of about 30% or more pores of up to 4.99 nm and the pores of 5 to up to 19.99 nm constituting the balance.
8. An absorbent for the selective removal of H<sub>2</sub>S from a gaseous mixture containing H<sub>2</sub>S, CO<sub>2</sub> and gaseous non-acidic components, the absorbent comprising a material of the formula

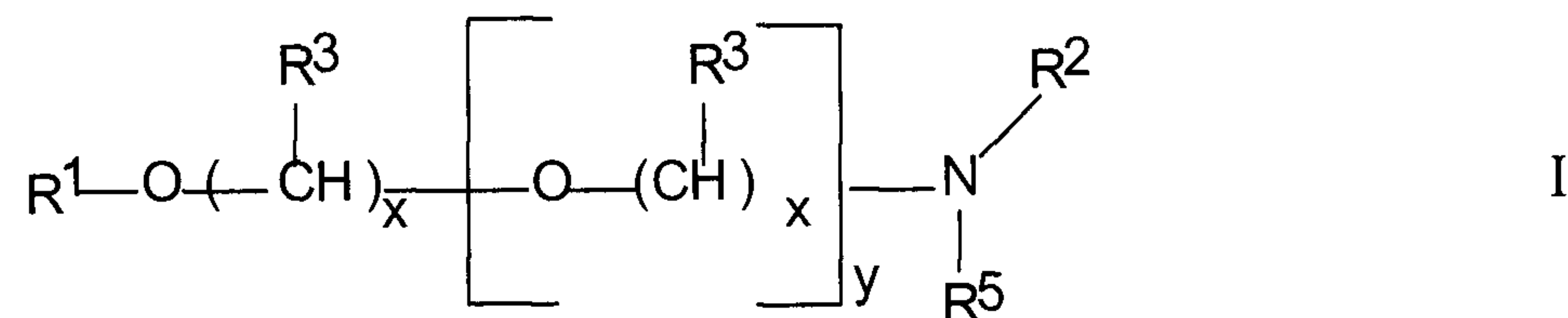


wherein:

x, y,  $R^1$ ,  $R^2$ ,  $R^3$  and  $R^5$  are as defined in claim 1.

9. The absorbent of claim 8 wherein
  - $R^1$  is C<sub>1</sub>-C<sub>4</sub> alkyl, or C<sub>3</sub>-C<sub>4</sub> branched alkyl,
  - $R^2$  is C<sub>3</sub>-C<sub>6</sub> straight or branched alkyl, and
  - $R^5$  is hydrogen or C<sub>1</sub>-C<sub>4</sub> alkyl.

10. The absorbent of claim 8 wherein x is 2 and y is 1, 2 or 3.
11. The absorbent of claim 9 wherein x is 2 and y is 1 or 2.
12. The absorbent of claim 8 wherein R<sup>3</sup> is hydrogen and R<sup>2</sup> is a C<sub>4</sub>-tertiary alkyl wherein N is attached to R<sup>2</sup> through the tertiary carbon atom of R<sup>2</sup>.
13. The absorbent of claim 9 wherein R<sup>3</sup> is hydrogen and R<sup>2</sup> is a C<sub>4</sub>-tertiary alkyl wherein N is attached to R<sup>2</sup> through the tertiary carbon atom of R<sup>2</sup>.
14. A process for the selective removal of H<sub>2</sub>S from a gaseous mixture containing H<sub>2</sub>S and CO<sub>2</sub> and gaseous non-acidic components by contacting said mixture with an absorbent solution comprising an absorbent material of the formula



where x, y, R<sup>1</sup>, R<sup>2</sup>, R<sup>3</sup> and R<sup>5</sup> are as defined in claim 1,  
under conditions whereby the H<sub>2</sub>S is selectively absorbed from the mixture.

15. The process of claim 14 wherein  
R<sup>1</sup> is C<sub>1</sub>-C<sub>4</sub> alkyl, C<sub>3</sub>-C<sub>4</sub> branched alkyl,  
R<sup>2</sup> is C<sub>3</sub>-C<sub>6</sub> straight or branched alkyl, and  
R<sup>5</sup> is hydrogen or C<sub>1</sub>-C<sub>4</sub> alkyl.
16. The process of claim 14 wherein x is 2 and y is 1, 2 or 3.
17. The process of claim 15 wherein x is 2 and y is 1 or 2.
18. The process of claim 14 wherein R<sup>3</sup> is hydrogen and R<sup>2</sup> is a C<sub>4</sub>-tertiary alkyl wherein N is attached to R<sup>2</sup> through the tertiary carbon atom of R<sup>2</sup>.

19. The process of claim 15 wherein  $R^3$  is hydrogen and  $R^2$  is a C4-tertiary alkyl wherein N is attached to  $R^2$  through the tertiary carbon atom of  $R^2$ .
20. The process of claim 1 in which the alkoxy alcohol is methoxytriethylene glycol and the alkylamine is tert.-butylamine.

Figure 1  
Adsorption-Regeneration Unit for Selective H<sub>2</sub>S Removal

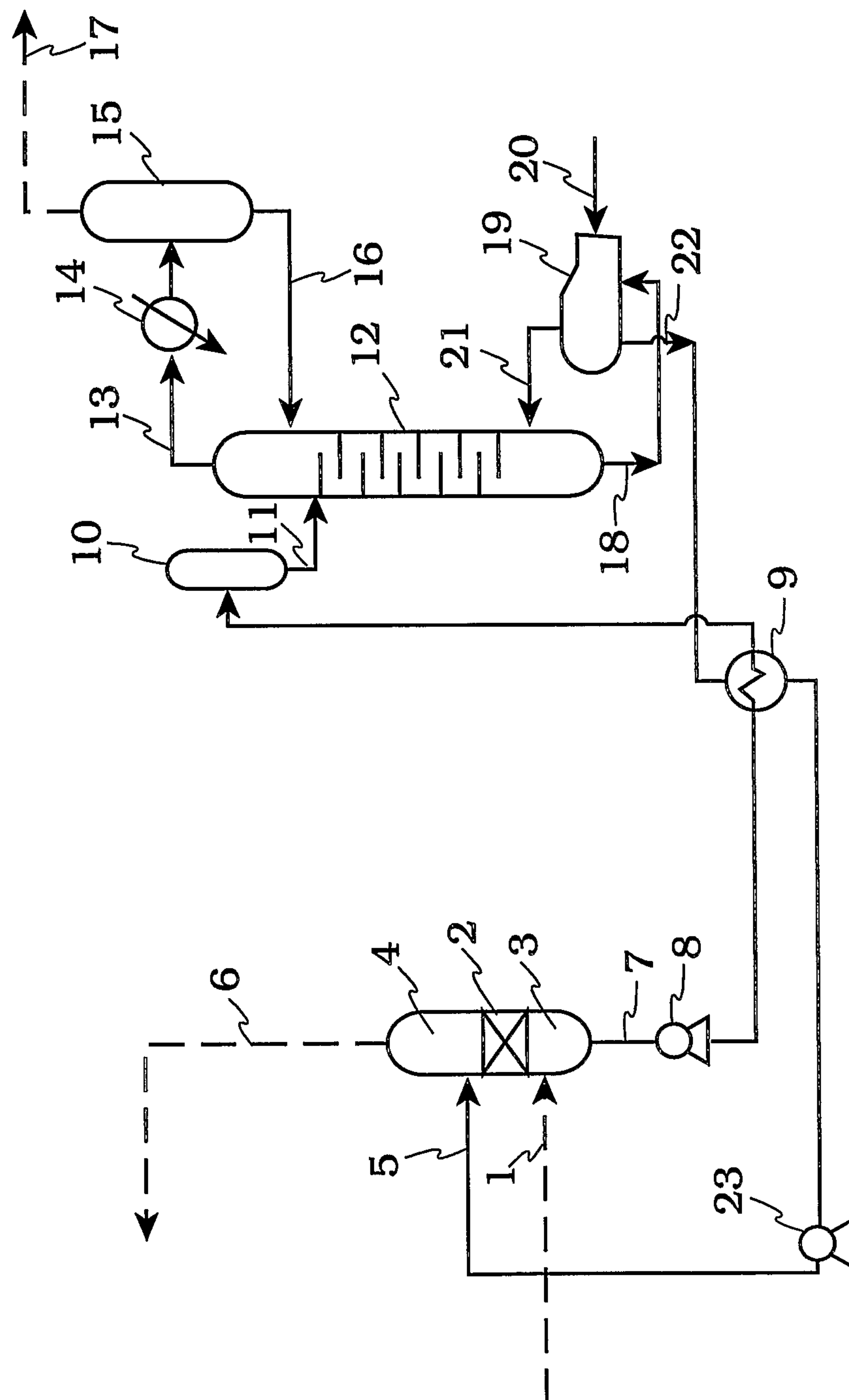
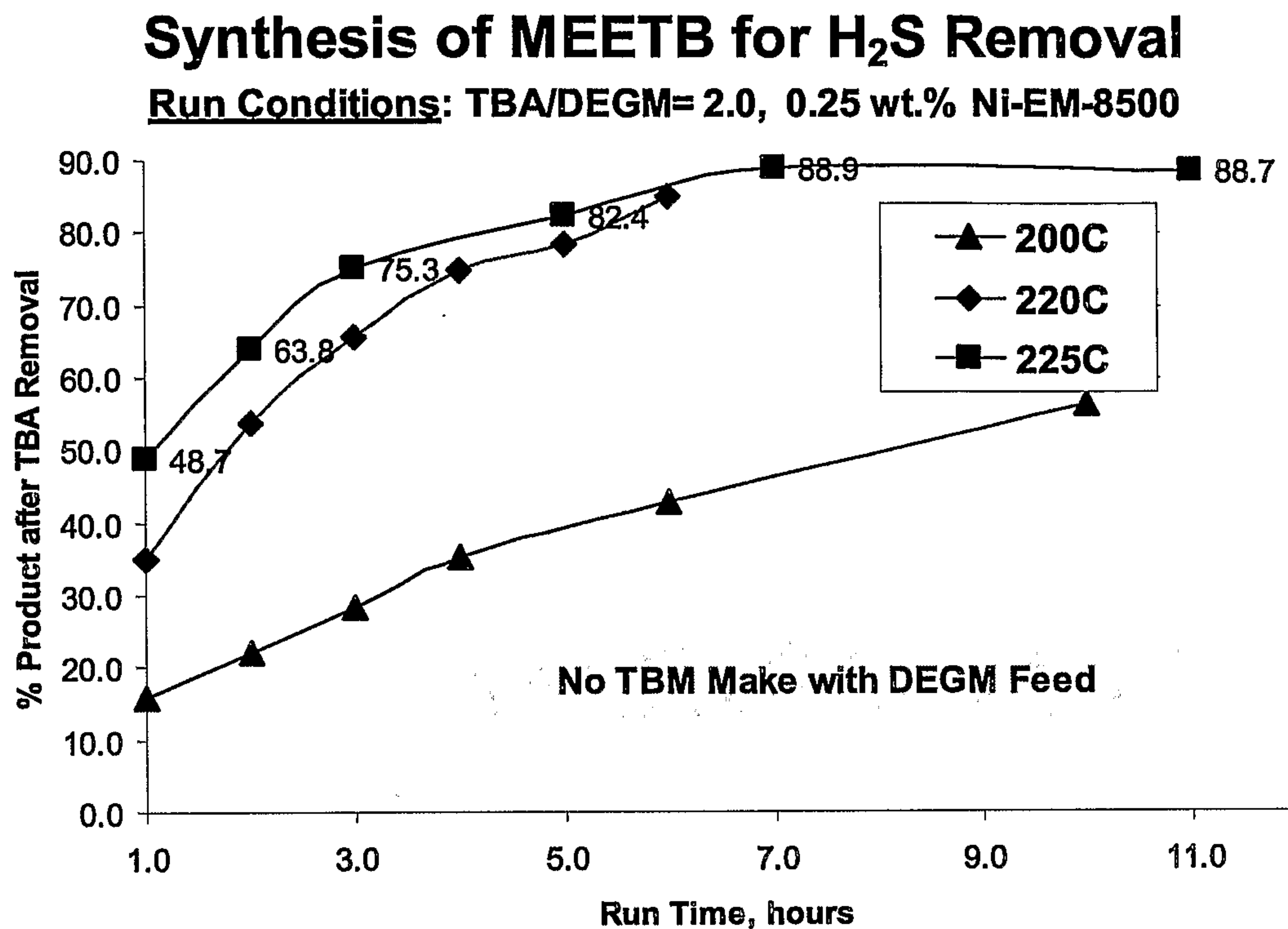


Figure 2



**High Conversion of DEGM  
Eliminates Distillation Requirement**

Figure 3

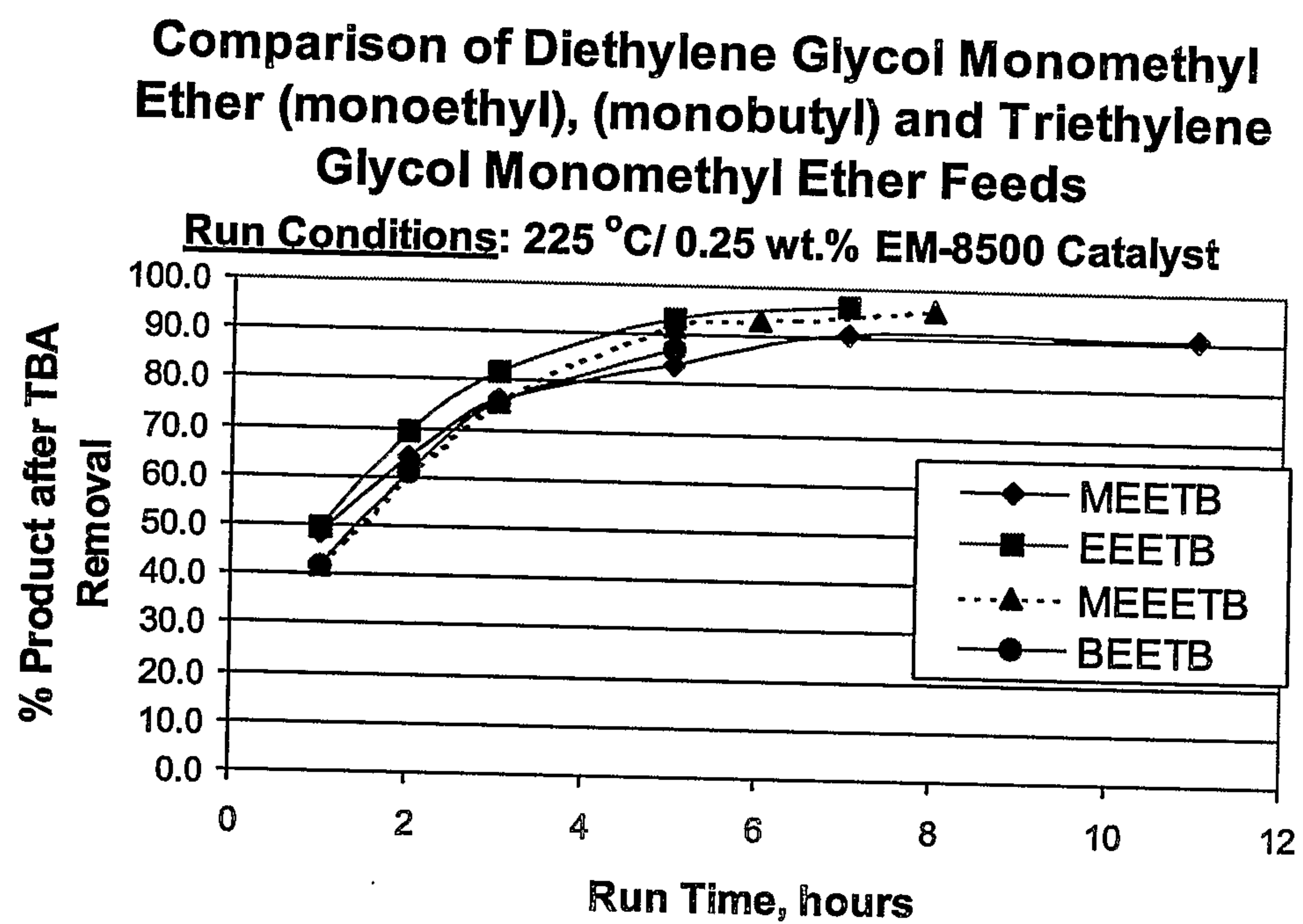


Figure 4

