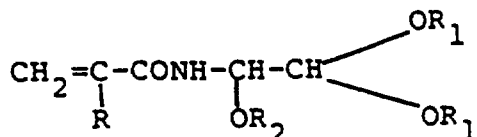


The invention relates to new substituted derivatives of N-ethyl(meth)acrylamide and a process for their preparation.

The invention relates to products having the general Formula (I)



(I)

where R denotes a hydrogen atom or a methyl radical, R_1 denotes a C_1 - C_8 alkyl group and R_2 denotes a hydrogen atom or a C_1 - C_8 alkyl group. The invention relates more particularly to the products as defined hereinbefore, characterized in that in Formula (I) R denotes a hydrogen atom, R_1 denotes a C_1 - C_4 alkyl group and R_2 denotes a hydrogen atom or a C_1 - C_4 alkyl group. Amongst these products, the following may be more particularly mentioned:

N-(1,2,2-trimethoxy ethyl) 2-propene amide.

N-(1,2,2-triethoxy ethyl) 2-propene amide.

N-(2,2-dimethoxy-1-hydroxy ethyl) 2-propene amide.

N-(2,2-diethoxy-1-hydroxy ethyl) 2-propene amide.

Derivatives of (meth)acrylamide substituted on the nitrogen atom have either an acetal function (European Patent Application No. 0 002 767 and United States Patent Specification No.

4 508 594), or a N-(meth)acrylyl hemiacetal function (United States Patent Specification No. 4 521 563), but the monomers according to the invention and having the general Formula (I) possess simultaneously two adjacent carbon atoms one of which performs an acetal function and the other a N-acyl hemiacetal function and which are not disclosed in the Literature, as far as

the Applicants are aware.

According to the invention the above products having the Formula (I) can be prepared by a process characterized in that (meth)acrylamide is reacted with a 2,2-dialkoxy ethanal having the Formula (II):



where R_1 has the meaning given in claim 1, to obtain a corresponding product of Formula (I), where R_2 denotes a hydrogen atom and which, if required, is etherified to form a corresponding product of Formula (I), where R_1 and R_2 , which are identical or different, denote a $\text{C}_1\text{-C}_8$ alkyl group.

In preferential conditions for the performance of the invention, the condensation of the (meth)acrylamide with a dialkoxy ethanal having the Formula (II) is performed in an inert atmosphere and:

at a temperature between 50 and 100°C, preferably $70 \pm 5^\circ\text{C}$;

with a slight excess of one or other of the reagents;

in the presence of a small quantity of an antioxidant, such as paramethoxy phenol.

According to the invention the etherification of the above products having the Formula (I), where R_2 denotes a hydrogen atom, into abovementioned products having the Formula (I), where R_2 denotes a $\text{C}_1\text{-C}_8$ alkyl group, is performed by a process characterized in that the corresponding alkanol having the formula (III) R_3OH , where R_3 denotes a $\text{C}_1\text{-C}_8$ alkyl group, with a product having the formula (I), where R_2 denotes a hydrogen atom, in the presence of catalytic quantities of an acid.

In preferential conditions for the performance of this last-mentioned process, etherification is performed at a temperature between 50°C and the reflux temperature of the reaction medium in the presence of an excess of alkanol having the Formula (III) and catalytic quantities of an acid, such as hydrogen chloride, paratoluene sulphonic acid, sulphuric acid.

The products of Formula (I) have advantageous properties in macromolecular chemistry. They can more particularly be used as monomers in the preparation of various polymers.

The following Examples illustrate the invention in a non-limitative manner.

The spectra of nuclear magnetic resonance were established in deuterated chloroform. The chemical shifts are expressed in ppm (parts per million) in relation to the tetramethyl silane used as an internal reference. The H^1 NMR were determined on a VARIAN apparatus at 60 MHz and the C^{13} NMR spectra on a VARIAN XL 100 apparatus at 25.2 MHz with Fourier transform. The mass spectra were determined on a VARIAN Mat CH5 apparatus at 70 eV.

Example 1

The following were heated for 2 hours at 70°C in an inert atmosphere:

23.8 mmoles of 2,2-diethoxy ethanal,
24 mmoles of acrylamide,
0.1 mmole of paramethoxyphenol.

In this way 23.3 mmoles of crude N-(2,2-diethoxy-1-hydroxy ethyl) 2-propene amide were obtained, whose C^{13} NMR spectrum was as follows: 15.26 (2q. $2CH_3$) 63.53 (t. CH_2); 63.96 (t. CH_2); 73.6 (d. N-CHO); 102 (d. O-CHO); 127.3 (m. $=CH_2$); 130.8 (m. $=CH$); 166 (s. N-CO).

The crude product was dissolved in 1.38 g (30 mmoles) of ethanol containing 0.49 g of concentrated sulphuric acid per litre, whereafter the solution was heated in an inert atmosphere for 4 hours at 70°C. The cooled reaction medium was then redissolved in water and the desired product was extracted with dichloromethane. The combined organic phases were then washed with water, dried, filtered and evaporated in vacuo.

5.1 g (21.2 mmoles) of N-(1,2,2-triethoxy ethyl) 2-propene amide were obtained having a melting point of $33 \pm 2^\circ\text{C}$ - i.e., a yield of 89% of the calculated theoretical value in relation to the 2,2-ethoxy ethanal used.

Physical analyses

^1H NMR: 1.2 (m. 9H); 3.6 (m. 6H); 4.5 (d. 1H); 5.4 (d. 1H); 5.8 (d.d., 1H); 6.2 (m. 2H)

^{13}C NMR: 15.08 (q. CH_3); 15.18 (q. CH_3); 15.25 (q. CH_3); 63.36 (t. CH_2); 64.01 (2t. CH_2); 79.04 (d. O-CH-N); 101.71 (d. O-CHO); 127.2 (m. CH); 130.7 (m. $=\text{CH}_2$); 165.9 (s. N-C=O).

Mass spectrum: M 231 (0%); 186 (2%); 161 (2%); 141 (5%); 139 (5%); 128 (6%); 103 (100%); 75 (53%); 59 (25%); 55 (20%).

Example 2

The following were heated for two hours at 70°C in an inert atmosphere:

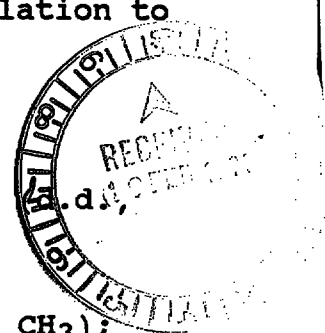
67 mmoles of acrylamide,

69 mmoles of 2,2-dimethoxy ethanal,

0.14 mmole of paramethoxyphenol.

67 mmoles of crude N-(2,2-dimethoxy-1-hydroxy ethyl) 2-propene amide were obtained in this way.

The following were heated for four hours at 70°C in an inert atmosphere:



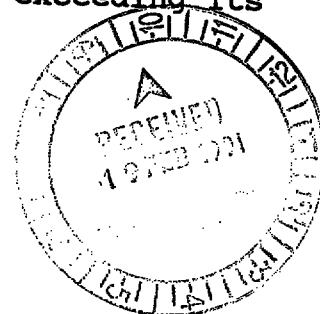
67 mmoles of N-(2,2-dimethoxy-1-hydroxy ethyl) 2-propene amide previously prepared in:

84 mmoles of methanol containing 5 mmoles of concentrated sulphuric acid per litre.

The reaction medium was then redissolved in water and the required product was extracted with dichloromethane.

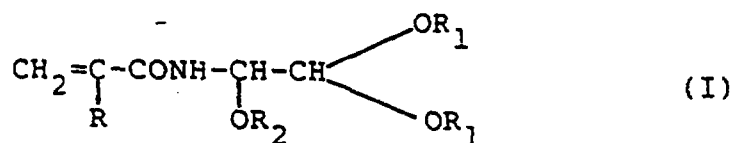
The combined organic phases were washed with water, dried, filtered and concentrated in vacuo. In this way 40 mmoles of N-(1,2,2-trimethoxy ethyl) 2-propene amide were obtained.

Clearly, the invention has been described only by way of non-limitative example and any modification, more particularly as regards equivalents, can be made thereto without exceeding its scope.



CLAIMS

Products having the general Formula (I):



where R denotes a hydrogen atom or a methyl radical, R₁ denotes a C₁-C₈ alkyl group and R₂ denotes a hydrogen atom or a C₁-C₈ alkyl group.

2. Products as defined in claim 1. characterized in that in the formula (I) R denotes a hydrogen atom, R₁ denotes a C₁-C₄ alkyl group and R₂ denotes a hydrogen atom or a C₁-C₄ alkyl group.

3. N-(1,2,2-trimethoxy ethyl) 2-propene amide.

4. N-(1,2,2-triethoxy ethyl) 2-propene amide.

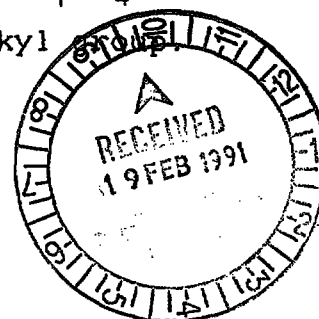
5. N-(2,2-dimethoxy-1-hydroxy ethyl) 2-propene amide.

6. N-(2,2-diethoxy-1-hydroxy ethyl) 2-propene amide.

7. A process for the preparation of the products as defined by Formula (I) in claim 1, characterized in that (meth)acrylamide is reacted with a 2,2-dialkoxy ethanal having the Formula (II):



where R₁ has the meaning given in claim 1. to obtain a corresponding product of Formula (I), where R₂ denotes a hydrogen



atom and which, if required, is etherified with a C_1-C_8 alkanol to form a corresponding product of Formula (I), where R_1 and R_2 , which are identical or different, denote a C_1-C_8 alkyl group.

8. A process according to claim 7, characterized in that the etherification of the products of Formula (I), where R_2 denotes a hydrogen atom, is performed in the presence of catalytic quantities of an acid with a C_1-C_8 alkanol.

