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CAPILLARY ACTIVE COMPOUNDS AND PROCESS OF PREPARING THEM

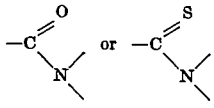
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8 Claims. (Cl. 260—67)

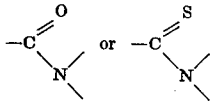
The present invention relates to capillary active compounds and to a process of preparing them as well as to a process of rendering textile materials water-repellent by means of the capillary active compounds.

We have found that capillary active compounds are obtainable by causing formaldehyde to act in the presence of a tertiary base and an organic acid anhydride upon a compound containing a hydrocarbon radical having at least 4 carbon atoms as well as one of the groupings:

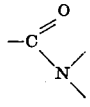


in which at least one exchangeable hydrogen atom is present at the nitrogen atom.

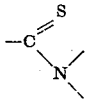
The compounds containing one of the groupings



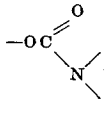
may vary greatly. They may belong to the aliphatic as well as to the cyclic series. There may be named, for instance, carboxylic acid amides with the atomic grouping



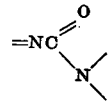
thiocarboxylic acid amides with the atomic grouping



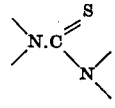
urethanes with the atomic grouping



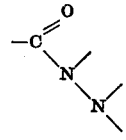
ureae with the atomic grouping



thioureae with the atomic grouping

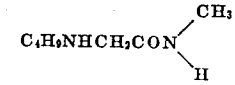


acid hydrazides with the atomic grouping

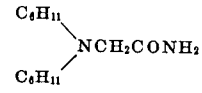


which contain in the polar group at least one hydrogen atom capable of being substituted and the molecule of which contains furthermore a hydrocarbon radical having at least 4 carbon atoms.

As examples there may be named acid amides and acid hydrazides of mono- and polybasic acid, for instance, N-butylacetamide, N-dodecylacetamide, lauric acid amide, N-dodecyl-lauric acid amide, N-methyl-lauric acid amide, stearic acid amide, isooctylphenoxyacetic acid methyl amide, methylamide of hydroxy-stearic acid, dichlorostearic acid amide, N-hydroxyethyl-undecylic acid amide, butylaminoacetic acid methylamide



dicyclohexylaminoacetic acid amide

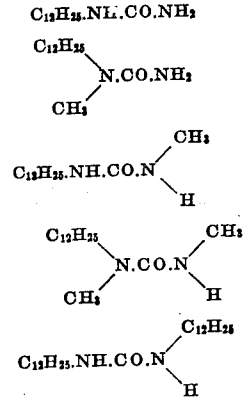


N-ethylbutoxyacetic acid amide, isooctylsalicylic acid amide, dodecylbenzamide, hexahydrophthalimide, dodecylsuccinic acid imide, lauric acid hydrazide, stearic acid hydrazide. Moreover, there may be mentioned compounds the funda-

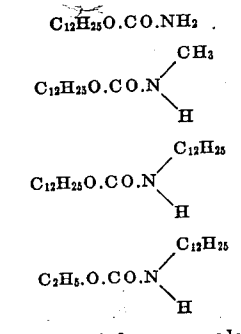
mental substance of which is urea or its derivatives wherein the



or C=N— grouping is still present, for instance, butyl urea, N-phenyl-N'-butylurea, di-octadecyl urea, octadecyl urea, oleyl urea, para-butylphenyl-dibutyl urea, the compounds of the following formulae:



diheptylmethyl thiourea, cyclohexylbutylthiourea, O-dodecyl-N-ethyl-urethane, O-octadecyl-urethane, O-cyclohexyl-N-butylurethane, O-p-octylphenyl-N-methylurethane, ethylurethane of the p-isododecylcyclohexylmonoglycol ether, cyclohexylurethane of the butylmonoglycol ether, methylurethane of the methylethanol-amide of lauric acid, the ethylurethane of perhydroabietinol, the butylurethane of a naphthene alcohol, the N-heptadecylmethylurethane, the compounds of the formulae:



The parent materials may also contain the above groups several times, as does for instance the reaction product from lauric acid chloride and meta-aminobenzoic acid methylamide.

Suitable tertiary amines are, for instance, pyridine and its homologues, quinoline, trimethylamine, methylpiperidine, N-methylmorpholine, triethylamine, tributylamine, dimethyl-aminoethyl-methyl ether, diethyl- cyclohexylamine, tetramethyl-methylenediamine, tetraethyl-ethylenediamine, tetra - methyl - hexamethylenediamine; these compounds are, in general, preferably used in excess.

Among the anhydrides of organic acids used there may, for instance, be mentioned: acetic anhydride, and dicarboxylic acids such as succinic anhydride, diglycolic anhydride, phthalic anhydride.

The reaction with formaldehyde is performed in the presence of the tertiary amine and the organic dicarboxylic acid anhydride at a moderately raised temperature the degree of which depends on the reactivity of the parent material used. The reaction may also be performed in the presence of a diluent or a solvent.

It is also possible to use a methylol compound of the parent material to react with a tertiary amine and the organic acid anhydride. One method is first to cause the methylol compound to be formed and then to cause it to react in one operation with a tertiary base and an organic acid anhydride.

The products of the present invention are, in general, wax-like or semi-solid masses; they are in part soluble in water, and in part capable of being dispersed in water. The products may be used, as such or in admixture with one another, as washing, emulsifying and wetting agents for the treatment of materials of all kinds. They may also be used together with other substances known for these purposes, such as soaps, Turkey red oils, sulfuric acid esters of alcohols of high molecular weight, the condensation products of fatty acids with amino-carboxylic and amino-sulfonic acids, glue, gums, organic solvents or the like. The products obtained according to this process and containing a hydrocarbon radical of high-molecular weight are also suitable as agents for rendering water-repellent natural or artificial textile fibers of vegetable or animal origin. For this purpose it is necessary to subject the impregnated fibrous material to a heat treatment at a temperature of not less than 80° C. The upper limit of the temperature range of this heat-treatment lies at 150° C. and in order to attain a sufficient water-repellent effect a temperature is to be chosen which, according to the condensation products used, lies between 30° C. and 150° C.

The following examples serve to illustrate the invention, but they are not intended to limit it thereto; the parts are by weight:

(1) 34 parts of acetic anhydride are run gradually, while stirring, at a temperature of 85° C. to 90° C., into a mixture of 66 parts of lauric acid amide, 20 parts of paraformaldehyde and 34 parts of tetramethylmethylenediamine. The whole is further heated for some time, until the mass has become soluble in water.

(2) A mixture of 90 parts of stearic acid amide, 50 parts of phthalic anhydride, 20 parts of para-formaldehyde and 60 parts of pyridine is heated, for 6 hours, at 100° C. while stirring. The product is a brownish, semi-solid mass the aqueous solutions of which have a good wetting, foaming and dispersing action.

(3) 40 parts of paraformaldehyde are added to a hot solution of 180 parts of stearic acid amide, 68 parts of tetramethylmethylenediamine and 98 parts of phthalic anhydride, and the whole is heated, for two hours at 80° C. to 90 C. while stirring. The product is soluble in water and has a wax-like consistency.

(4) A mixture of 72 parts of stearic acid methylamide, 15 parts of paraformaldehyde, 45 parts of pyridine and 37 parts of phthalic anhydride is heated for 3 hours, at 90° C. to 100° C. while stirring. The product is a reddish, wax-like mass which dissolves in water, to form a solution having a good capacity for foaming.

(5) 60 parts of octadecyl urea, 6 parts of paraformaldehyde, 30 parts of diglycolic anhydride and 70 parts of dimethylaminoethylmethylether are heated together, for 1 hour at 90° C. to 95° C. while stirring. The product obtained forms a colorless wax-like mass which is soluble in water.

(6) A mixture of 37 parts of N-butyl-N'-octa-

decyl urea, 23 parts of diglycolic anhydride, 3 parts of paraformaldehyde and 35 parts of dimethylaminoethylmethylether is heated for 1 hour at 90° C. to 95° C. A brownish salve-like mass is obtained, which yields with hot water well foaming solutions.

(7) A mixture of 50 parts of octadecyl-carbamic acid ester, 15 parts of paraformaldehyde, 20 parts of diglycolic anhydride and 150 parts of pyridine is heated for 2 hours at 100° C. while stirring. The excess of the base is then distilled under reduced pressure.

(8) 25 parts of iso-octylphenol-carbamic acid ester, 3 parts of paraformaldehyde, 25 parts of diglycolic anhydride and 35 parts of dimethylaminoethylmethylether are heated for half an hour at 95° C. The product obtained forms a brownish viscous oil which is soluble in water.

(9) A mixture of 30 parts of stearic acid methylol amide, 6 parts of paraformaldehyde, 15 parts of phthalic anhydride and 40 parts of pyridine is heated for 2 hours at 90° C. to 95° C. The mass obtained has become water-soluble.

(10) 32 parts of N-phenyl-N'-dodecylthiourea, 3 parts of paraformaldehyde, 15 parts of diglycolic anhydride and 35 parts of dimethylaminoethylmethylether are heated together for 1 hour, at 85° C. to 90° C. while stirring. The reaction product forms a yellowish viscous oil.

(11) A mixture of 30 parts of stearic acid hydrazide, 3 parts of paraformaldehyde, 15 parts of diglycolic anhydride and 35 parts of dimethylamino-ethylmethylether is heated, to 90° C. for 1 hour while stirring. The reaction product is a colorless wax-like mass which yields with water well foaming solutions.

(12) Artificial silk piece-goods are passed through a bath containing per liter 10 grams of a condensation product obtained in a manner analogous to that indicated in Example 4, from stearic acid amide, pyridine, paraformaldehyde and phthalic acid anhydride. After centrifuging, the material is heated, for 50 minutes, at 105° C. to 110° C. The material thus treated is, to a high extent, waterproof and easily repels water dropped upon it.

(13) A worsted yarn fabric is treated with an aqueous solution containing per liter 10 grams of a product made as described in Example 3 of stearic acid amide with paraformaldehyde, tetramethylmethylenediamine and phthalic acid anhydride. After centrifuging, the textile material is first pre-dried at 50° C. and then heated for one hour at 100° C. to 110° C.

We claim:

1. The process which comprises causing a compound selected from the group consisting of carboxylic acid amides, urethanes, ureas, containing a hydrocarbon radical of at least 4 carbon atoms and having a hydrogen atom capable of being substituted linked to nitrogen, to react with formaldehyde in the presence of a tertiary amine and an anhydride of a dicarboxylic acid, while heating to temperatures of about 70° C. to about 110° C.

2. The process which comprises causing stearic acid amide to react with formaldehyde in the presence of pyridine and phthalic anhydride while stirring and heating to about 100° C.

3. The process which comprises causing octadecyl urea to react with formaldehyde in the presence of dimethylamino-ethylmethyl ether and diglycolic acid anhydride while stirring and heating to about 95° C.

4. The process which comprises causing octadecyl-carbamic acid ester to react with formaldehyde in the presence of pyridine and diglycolic acid anhydride while stirring and heating to about 100° C.

5. The condensation products obtained according to the process described in claim 1.

6. The condensation product prepared from stearic acid amide by heating it to about 100° C. together with formaldehyde, pyridine and phthalic anhydride.

7. The condensation product prepared from octadecyl urea by heating it to about 95° C. together with formaldehyde, dimethylaminoethylmethyl ether and diglycolic acid anhydride.

8. The condensation product prepared from octadecyl carbamic acid ester by heating it to about 100° C. together with formaldehyde, pyridine and diglycolic acid anhydride.

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