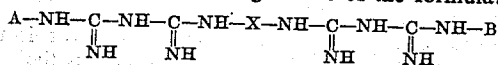


PROCESS OF MAKING FUNGICIDAL, BACTERI- CIDAL, AND DETERGENT COMPOSITIONS BY REACTING AN AROMATIC AMINE AND A DICYANDIAMIDE IN THE PRESENCE OF A NONIONIC SOLVENT

**No Drawing. Application March 16, 1954
Serial No. 416,715**

3 Claims. (Cl. 167—30)

In co-pending U. S. application Serial No. 295,122, now abandoned, there are described and claimed new fungicidal, bactericidal and detergent compositions which comprise one or more bis-biguanides of the formula:



We have now found that useful fungicidal, bactericidal and detergent compositions can conveniently and advantageously be made by conducting a process for the manufacture of a bisbiguanide in a non-ionic wetting agent or detergent as solvent. The so-manufactured compositions contain the bis-biguanides used in making the compositions of copending U. S. application S. N. 295,122.

$$\text{NH}_2\text{---X---NH}_2$$
$$\text{A}-\text{NH}-\text{C}(\text{:NH})-\text{NH}-\text{CN}$$
$$\text{CN}-\text{NH}-\underset{\text{NH}}{\underset{\parallel}{\text{C}}}-\text{NH}-\text{X}-\text{NH}-\underset{\text{NH}}{\underset{\parallel}{\text{C}}}-\text{NH}-\text{CN}$$

ANH,

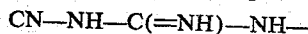
Thus for example hexamethylenediamine hydrochloride may be caused to react with p-chlorophenyldicyan diamide in the presence of, as reaction solvent, a condensation product of ethylene oxide and octylcresol. The reaction product possesses fungicidal, bactericidal and

As diamines of the formula NH_2XNH_2 there may also be used, besides hexamethylenediamine, for example the following: trimethylenediamine, tetramethylenediamine, pentamethylenediamine, heptamethylenediamine, decamethylenediamine, 1:4-bis- γ -aminopropoxybenzene, 4:4'-diaminodiphenylmethane and p-phenylenediamine.

$$\text{A}-\text{NH}-\text{C}(=\text{NH})-\text{NH}-\text{CN}$$

there may also be used, besides p-chlorophenyldicyandiamide, for example the following, phenyldicyandiamide, p-tolyldicyandiamide, 2,4-dimethylphenyldicyandiamide, 15 p-anisyldicyandiamide, o-chlorophenyldicyandiamide, m-chlorophenyldicyandiamide, 2:5-dichlorophenyldicyandiamide, 3:4-dichlorophenyldicyandiamide, m-nitrophenyldicyandiamide, α -naphthyldicyandiamide and β -naphthyldicyandiamide.

20 Correspondingly, compounds of the formula



25 —[CH₂]₃—, —[CH₂]₄—, —[CH₂]₅—, —[CH₂]₆—,
—[CH₂]₇—, —[CH₂]₁₀—, —[CH₂]₃OC₆H₄O[CH₂]₃—,
—C₆H₄—NH—C₆H₄— and —C₆H₄—, and amines of
the formula ANH₂ may for example be aniline, p-tolui-
dine, 2,4-dimethylaniline, p-anisidine, o-chloroaniline, m-
30 chloroaniline, p-chloroaniline, 2,5-dichloroaniline, 3,4-
dichloroaniline, m-nitroaniline, α-naphthylamine and β-
naphthylamine.

As said, in the reaction the amine is used in the form of a salt thereof. Suitable salts include for example the 35 hydrochloride, the hydrobromide and the sulphate.

As non-ionic substances possessing wetting and/or detergent properties there may be mentioned for example condensation products of ethylene oxide with for example, alkylphenols, long-chain fatty alcohols, and long-chain fatty acids or their amides.

The products of the process of the invention are useful in cleansing and disinfecting operations, particularly in farm, especially dairy, hygiene.

The invention is illustrated but not limited by the following examples in which the parts are by weight:

Example 1

50 77.8 parts of p-chlorophenyldicyandiamide, 37.8 parts of hexamethylenediamine dihydrochloride and 300 parts of the condensation product of octyl cresol with 9.5 molecular proportions of ethylene oxide are mixed and the mixture is stirred at 140–150° C. for 18 hours. The reaction product is then boiled with 860 parts of water and there is obtained a homogeneous liquid composition
55 which possesses valuable fungicidal, bactericidal and detergent properties.

Example 2

In a manner similar to that described in Example 1, 2.59 parts of p-chlorophenyldicyandiamide are caused to react with 1.26 parts of hexamethylenediamine dihydrochloride in 10 parts of a condensation product of cetyl alcohol with 17 molecular proportions of ethylene oxide. The reaction mixture is then dissolved in 28.4 parts of water at 80° C. and a detergent solution is obtained which possesses valuable fungicidal, bactericidal and detergent properties.

Example 3

70 2.59 parts of p-chlorophenyldicyandiamide, 126 parts of hexamethylenediamine dihydrochloride, and 10 parts of the condensation product of stearic acid with 16.3 molecular proportions of ethylene oxide, are heated to-

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gether with agitation for 18 hours at 140–150° C. The reaction product is treated with 67 parts of water at 80° C., and the mixture is cooled and filtered. The filtrate is a clear liquid which possesses valuable fungicidal, bactericidal and detergent properties.

Example 4

By working in a manner similar to that described in Example 3, 2.71 parts of 4:4'-diaminodiphenylmethane dihydrochloride, 3.2 parts of phenyldicyandiamide, 15.3 parts of the condensation product of octyl cresol with 9.5 molecular proportions of ethylene oxide, and 43.5 parts of water are caused to react to give a composition which possesses valuable fungicidal, bactericidal and detergent properties.

Example 5

By working in a manner similar to that described in Example 3, 6.4 parts of phenyldicyandiamide, 3.78 parts of hexamethylenediamine dihydrochloride and 21.2 parts of a condensation product of octyl cresol with 9.5 molecular proportions of ethylene oxide are heated together at 140–150° C. and there is obtained a liquid composition which possesses valuable fungicidal, bactericidal and detergent properties.

Example 6

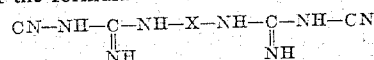
3.3 parts of p-chloraniline hydrochloride, 3.5 parts of hexamethylene-bis-dicyandiamide and 17.5 parts of the condensation product of octylcresol with 9.5 molecular proportions of ethylene oxide are stirred together at 140–150° C. for 18 hours. The mixture is then cooled to 80° C. and 50 parts of water are added. It is then filtered and the filtrate is a homogenous liquid composition which possesses valuable fungicidal, bactericidal and detergent properties.

What we claim is:

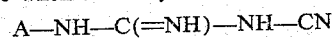
1. Process for the manufacture of fungicidal, bactericidal and detergent compositions which comprises interaction of a hexamethylenediamine salt with p-chlorophenyldicyandiamide in the presence of a non-ionic detergent as a solvent for said reactants, said detergent being a condensation product of ethylene oxide and octylcresol.

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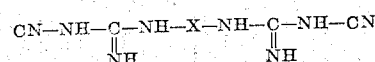
2. In a process for the manufacture of fungicidal, bactericidal and detergent compositions wherein the salt of an amine selected from the group consisting of diamines of the formula $\text{NH}_2\text{—X—NH}_2$ and monoamines of the formula ANH_2 is reacted with a dicyandiamide selected from the group consisting of dicyandiamides of the formula A—NH—C(=NH)—NH—CN and dicyandiamides of the formula



wherein X is selected from the group consisting of hydrocarbon and dialkoxybenzene radicals of not more than 15 carbon atoms; A is selected from the group consisting of aromatic hydrocarbon and halo, nitro, alkyl and alkoxy substituted aromatic hydrocarbons, said amine is a diamine when said dicyandiamide is



and said amine is a monoamine when said dicyandiamide is



the improvement wherein said reaction is carried out in the presence of a non-ionic substance as a solvent for said reactants, said substance being selected from the group consisting of non-ionic detergents and non-ionic wetting agents formed from higher polyoxyethylene ether condensation products with long-chain alkyl phenols, fatty alcohols and fatty acids.

3. The process of claim 2 wherein said salt is the hydrochloride and said condensation product is the condensation product of octylcresol with 9.5 molecular proportions of ethylene oxide.

References Cited in the file of this patent

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