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N(3-METHYL-5-PYRAZOLONE)-HALO-CARBOXANILIDES

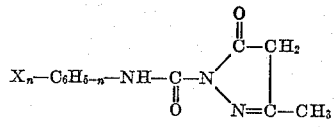
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This invention relates to new and useful carboxanilides and to processes for making same.

The new compounds of this invention are N-(3-methyl-5-pyrazolone) halo-carboxanilides of the structure



wherein $X_n—C_6H_{5-n}$ is a halogen substituted phenyl radical free of substituents ortho to the nitrogen atom, where X is bromine or chlorine, and where n is a whole number from 2 to 3. As illustrative of the new compounds of this invention is the following:

N-(3-methyl-5-pyrazolone) 3,4-dichloro-carboxanilide
N-(3-methyl-5-pyrazolone) 3,4-dibromo-carboxanilide
N-(3-methyl-5-pyrazolone) 3,5-dichloro-carboxanilide
N-(3-methyl-5-pyrazolone) 3,4,5-trichloro-carboxanilide
N-(3-methyl-5-pyrazolone) 3,4,5-tribromo-carboxanilide

These compounds are prepared by reacting 3-methyl-5-pyrazolone with a halo-phenylisocyanate of the formula

$$X_n—C_6H_{5-n}—N=C=O$$

in the presence of an inert organic solvent. As illustrative of the preparation of the compounds of this invention is the following:

Example I

To a suitable reaction vessel equipped with a thermometer, reflux condenser and an agitator is charged 4.9 parts by weight of 3-methyl-5-pyrazolone and 356 parts by weight of acetone. The mass is heated to boiling and thereto is slowly added 9.4 parts by weight of 3,4-dichlorophenylisocyanate dissolved in 40 parts by weight of acetone. Upon completion of the isocyanate addition the mass is agitated for a short period, cooled and filtered. The filter cake is then dissolved in hot dimethyl formamide. Upon cooling the so formed solution, filtering and drying the filter cake, there is obtained as a white powder N-(3-methyl-5-pyrazolone) 3,4-dichloro-carboxanilide (M. P. 228–229° C.).

Example II

Employing the procedure of Example I but replacing 3,4-dichlorophenylisocyanate with an equal weight of 3,5-dichlorophenylisocyanate, there is obtained N-(3-methyl-5-pyrazolone) 3,5-dichloro-carboxanilide.

Example III

Employing the procedure of Example I but replacing 3,4-dichlorophenylisocyanate with a chemically equivalent weight of 3,4,5-trichlorophenylisocyanate, there is obtained N-(3-methyl-5-pyrazolone) 3,4,5-trichloro-carboxanilide.

Example IV

Employing the procedure of Example I but replacing

3,4-dichlorophenylisocyanate with a chemically equivalent weight of 3,4-dibromophenylisocyanate, there is obtained N-(3-methyl-5-pyrazolone) 3,4-dibromo-carboxanilide.

The compounds of this invention are particularly useful in controlling bacterial growth, particularly *Micrococcus pyogenes* var. *aureus*. In this regard the carboxanilides when compounded with a detergent soap (i. e. an alkali metal salt of a higher fatty acid of animal or vegetable origin, such as stearic acid, lauric acid, palmitic acid, oleic acid, linoleic acid, ricinoleic acid, and the like, or mixtures thereof obtained from tallow, lard, cocoanut oil, palm oil, castor oil, olive oil, cottonseed oil, and the like) provide highly useful antiseptic detergent soap compositions.

In order to illustrate the activity of the carboxanilides of this invention several were incorporated in an alkali metal fatty acid soap—specifically a mixture of alkali metal salts of fatty acids whose fatty acid content analyzes:

	Percent
Oleic and linoleic acid	About 46
Stearic acid	About 14
Palmitic acid	About 30
Lower fatty acids (myristic, lauric, etc.)	About 10

in a weight ratio of one part to 50 parts soap. Aliquots of each were added to a Sabourard's dextrose agar medium so as to give concentrations in parts per million as set forth below. The agar in each case was then poured into a petri dish, allowed to harden and then inoculated with a standard culture of *Micrococcus pyogenes* var. *aureus* of standard resistance. The incubation in each instance was made at 37° C. for 48 hours. The extent of growth is noted below:

Compound/Concentration, p. p. m.	1,000	100	10
N-(3-methyl-5-pyrazolone) 3,4-dichloro-carboxanilide	none	none	none

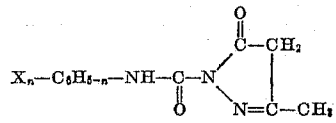
The same control of *Micrococcus pyogenes* var. *aureus* is obtained by replacing N-(3-methyl-5-pyrazolone) 3,4-dichloro-carboxanilide in the foregoing detergent soap composition with an equal weight of N-(3-methyl-5-pyrazolone) 3,5-dichloro-carboxanilide, N-(3-methyl-5-pyrazolone) 3,4,5-trichloro-carboxanilide, and N-(3-methyl-5-pyrazolone) 3,4-dichloro-carboxanilide.

Relatively small amounts of these carboxanilides in a detergent soap composition have been found to yield effective antiseptic detergent soap compositions. Amounts as low as 0.5 to 1% by weight based on the weight of the detergent soap have proved satisfactory. However, it is preferred to employ these carboxanilides in amounts in the order of 1 to 5% by weight based on the detergent soap. While larger amounts, as for example up to 10% by weight, may be employed the upper limit will be determined by practical considerations. Various colors, antioxidants, perfumes, water softeners, emollients, and the like, may be included where desirable in detergent compositions containing these new carboxanilides. The term "soap" or "detergent soap" as used herein is employed in its popular or ordinary meaning, i. e. those cleansing compositions prepared from an alkali metal compound such as potassium or sodium hydroxide and a fat or fatty acid, both saturated and unsaturated.

While this invention has been described with respect to certain embodiments, it is not so limited and it is to be understood that variations and modifications thereof obvious to those skilled in the art may be made without departing from the spirit or scope of this invention.

What is claimed is:

1. Compounds of the structure



wherein $X_n—C_6H_{5-n}$ is a halogen substituted phenyl radical free of substituents ortho to the nitrogen atom, wherein X is a halogen atom selected from the group consisting of bromine and chlorine, and where n is a whole number from 2 to 3.

2. Compound of claim 1 wherein X is chlorine and where n is two.

3. N-(3-methyl-5-pyrazolone) 3,4-dichloro-carboxanilide.

4. N-(3-methyl-5-pyrazolone) 3,5-dichloro-carboxanilide.

5. The process of making the compounds of claim 1, which comprises reacting in substantially equimolecular proportions 3-methyl-5-pyrazolone and an isocyanate of the formula

$$X_n—C_6H_{5-n}—N=C=O$$

wherein $X_n—C_6H_{5-n}—$, X and n have the same significance as in claim 1, in the presence of an inert organic solvent.

No references cited.