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PATENT SPECIFICATION

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Int. Cl.:—C 07 d // A01n.

COMPLETE SPECIFICATION

NO DRAWINGS

3,5-Dichloro-2,6-Dimethyl-4-Pyridinol

We, THE DOW CHEMICAL COMPANY, a Corporation organised and existing under the Laws of the State of Delaware, United States of America, of Midland, County of Midland, State of Michigan, United States of America, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement :—

The present invention is directed to 3,5-dichloro-2,6-dimethyl-4-pyridinol, which is normally an equilibrium mixture of enol and keto forms. This compound is a crystalline solid material which is somewhat soluble in many common organic solvents and of very low solubility in water.

The new compound can be prepared by the chlorination of 2,6-dimethyl-4-pyridinol. The chlorination is preferably carried out in an oqueous solution of hydrochloric acid. Good results are usually obtained when the chlorination is carried out with any substantial amount of chlorine. However, it is generally preferred to convert the entire 2,6-dimethyl-4-pyridinol reactant. In accordance with a preferred procedure, one molecular proportion of 2,6-dimethyl-4-pyridinol is employed with at least two molecular proportions of chlorine. Larger or smaller amounts of chlorine can be employed but are undesirable from the standpoint of economy of operation. The reaction is carried out at a temperature at which hydrogen chloride is formed as a product of reaction, advantageously at a temperature within the range of from 15° to 80°C. The chlorination normally takes place smoothly under the foregoing conditions with the production of the desired 3,5-dichloro-2,6-dimethyl-4-pyridinol as a crystalline solid. Upon completion of the chlorination, the desired product can be separated from the reaction mixture by conventional procedures, such as filtration, and further purified, for example, by such procedures as washing with water and recrystallization from various organic solvents.

The 2,6-dimethyl-4-pyridinol (enol and keto forms) employed as a starting material in accordance with the foregoing teachings can be prepared in known procedures from 2-acetyl-3-oxo-4-hexenoic acid, Δ-lactone. In such method, the 2-acetyl-3-oxo-4-hexenoic acid, Δ-lactone is rested with hydrochloric acid at about 100°C. to obtain the 2,6-dimethyl-4H-pyran-4-one. Ammonolysis or treatment of the 2,6-dimethyl-4H-pyran-4-one with aqueous ammonia gives the 2,6-dimethyl-4-pyridinol, usually in excellent yield.

The compound of the present invention is useful as apestide for the control of a number of species of pests. For such use, this product is dispersed on a finely divided solid and employed in the form of a dust. Also, such mixtures may be dispersed in water with or without the aid of a surface active dispersing agent and the resulting aqueous suspensions employed as sprays. In other procedures, the product is employed as a constituent in solvent solutions, water-in-oil or oil-in-water emulsions or aqueous dispersions. In representative operations aqueous compositions containing 2,000 parts per million by weight of 3,5-dichloro-2,6-dimethyl-4-pyridinol give substantially complete kills of southern army worms (*Prodenia eridania*).

The following Example further illustrates the present invention.

EXAMPLE

Chlorine (1430 grams; 20.05 moles) was introduced portionwise into a mixture of 1127 grams (9.15 moles) of 2,6-dimethyl-4-pyridinol, 915 milliliters of concentrated

[Price 4s. 6d.]

hydrochloric acid, and 9150 milliliters of water. The addition of chlorine was carried out over a period of 12.5 hours at a temperature of 25°C. Following the chlorination, the reaction mixture was filtered to separate the 3,5-dichloro-2,6-dimethyl-4-pyridinol in a yield of 82 per cent. This product was a white crystalline solid which failed to melt at temperatures up to 350°C. The product was recrystallized from dimethyl-formamide and had a nitrogen content of 7.59 per cent and a chlorine content of 36.74 per cent as compared to a theoretical content of 7.29 per cent for nitrogen and 36.74 per cent for chlorine.

WHAT WE CLAIM IS :—

1. 3,5-Dichloro-2,6-dimethyl-4-pyridinol.

2. Process for the production of 3,5 dichloro-2,6-dimethyl-4-pyridinol characterized in that 2,6-dimethyl-4-pyridinol is reacted with chlorine.

3. Process in accordance with Claim 2, characterized in that the reaction is carried out in an aqueous solution of hydrochloric acid.

4. Process in accordance with Claim 2 or 3, characterized in that at least two molecular proportions of chlorine are employed for each molecular proportion of the 2,6-dimethyl-4-pyridinol.

5. Process in accordance with any of Claims 2 to 4, characterized in that the reaction is carried out at a temperature within the range of from 15 to 80°C.

6. Process for the production of 3,5-dichloro-2,6-dimethyl-4-pyridinol substantially as herein described with reference to the specific Example.

7. 3,5-Dichloro-2,6-dimethyl-4-pyridinol whenever produced by the process of any one of Claims 2 to 6.

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