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**2,710,269**

# METHOD OF TREATING WOOLEN TEXTILE MATERIALS WITH DI-PENTACHLOROPHENYL-MONO-HYDROGEN PHOSPHATE FOR PROTECTING SUCH TEXTILES AGAINST BIOLOGICAL ATTACK

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**No Drawing. Application June 19, 1952,  
Serial No. 294,484**

**Claims priority, application Great Britain July 2, 1951**

**1 Claim. (Cl. 117--138.5)**

This invention relates to the treatment of woolen textile materials with di-pentachlorophenyl-mono-hydrogen phosphate for protecting them against biological attack.

It is well known that the chlorinated phenols and particularly pentachlorophenol, when applied to textile materials, possess the power to a high degree of protecting such materials from attack by moulds, bacteria and biting insects.

Unfortunately these chlorinated phenols also possess undesirable properties from the point of view of the textile technologist to varying degree, particularly solubility in water and volatility which are large for the orders of magnitude involved, although these undesirable properties are least developed in pentachlorophenol.

Various attempts have been made to find compounds of pentachlorophenol which possess the valuable properties of that substance without the undesirable properties.

Although it is recognised that prediction is not possible in regard to the properties of a chemical compound, this is much more so when its physiological properties simultaneously come into question.

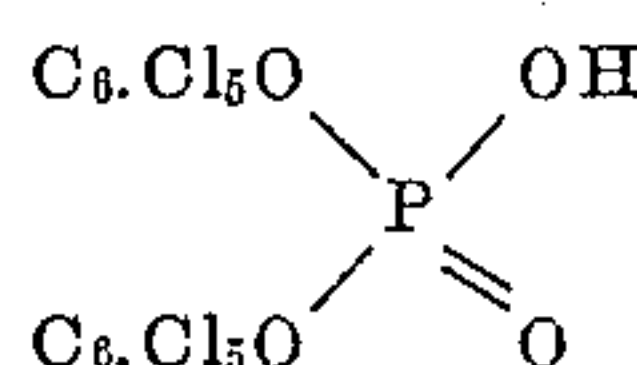
Consequently, a very large number of derivatives of pentachlorophenol have now been prepared from among which those found to possess the required properties in the highest degree have been determined by trial and experiment. Such desirable properties include the following:

1. High toxicity to the biological enemies of textile fibres with very low toxicity towards human beings per os and harmless when in contact with the human skin.

2. Substantial non-volatility at temperatures up to the temperature at which textiles are destroyed either in steam or in the dry state.

3. Substantivity and compatibility with dyestuffs and the customary dyebath additions in acid and neutral solutions and highly stable to the action of water and detergents and dry cleaners' liquids when "dyed" upon the textile.

The substances which consequently are employed in accordance with the present invention are the phosphates of pentachlorophenol, in particular the di-pentachlorophenyl mono-hydrogen phosphate which as far as can be ascertained has the composition



and which it is believed has not been previously prepared.

The customary method of preparing such compounds is to heat the phenol with phosphorous oxychloride but in the case of pentachlorophenol, the customary method of boiling the reagents together yields a very poor result—at most a few per cent of the theory. The reaction does not begin to take place at temperatures below 180° C.

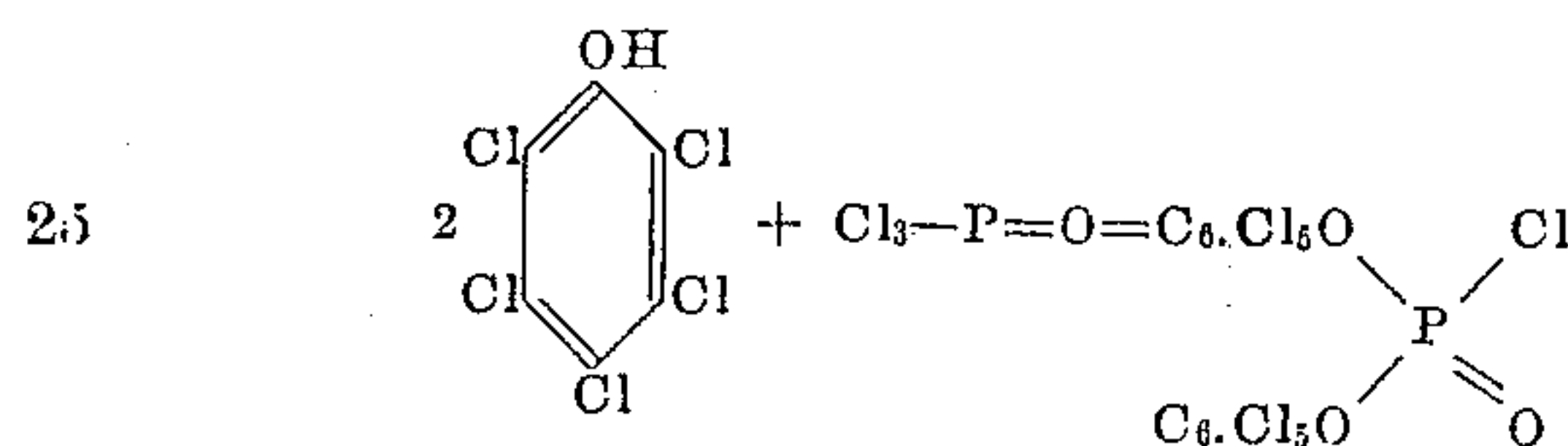
It has now been found, however, that if pentachlorophenol is caused to react with phosphorous oxychloride

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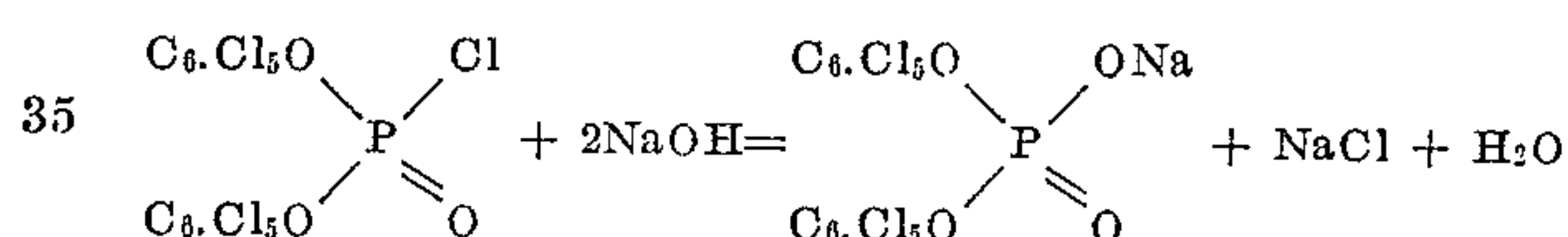
at high temperatures (between 250 and 300° C.), good yields of the desired products are obtained, the best yields being obtained at the higher temperature. This temperature, however, is greatly above the boiling point of the phosphorous oxychloride and therefore a departure from the customary method of boiling is required. Actually it is necessary to pass the phosphorous oxychloride in the vapour form through the pentachlorophenol with the latter in the molten state. Alternatively heating under pressure though possible, gives rise to great difficulties on account of the simultaneous evolution of hydrochloric acid.

### Example

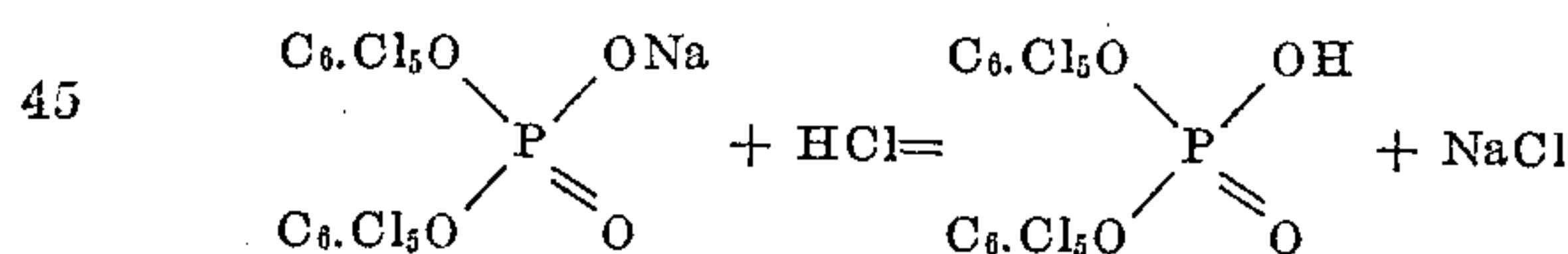
15 In the preparation of di-pentachlorophenyl monohydrogen phosphate, 533 grams of pentachlorophenol in the molten state has 160 grams of phosphorous oxychloride in the vapour form passed through it at a temperature of 290° C. for 10 hours. The primary product of  
20 the reaction is di-pentachlorophenyl chlor phosphate formed according to the equation;



30 When cold, the mass is boiled with a solution of caustic soda sufficient to give a faintly alkaline reaction and the mass breaks up into a sandy mass of the sodium compound as follows:



40 The sodium compound is filtered off and by treatment with dilute hydrochloric acid at the boil, about 430 grams of the di-pentachlorophenyl mono-hydrogen phosphate is obtained mixed with a small quantity of the neutral tripentachlorophenyl phosphate:



50 The small amount of neutral phosphate can be separated, if desired, by washing the mixture of it and the pentachlorophenyl sodium acid phosphate with toluene, from which solvent, the neutral phosphate can be recovered by evaporation.

The sodium compound forms a fine white power which  
55 disperses spontaneously in water or dilute alkali.

Woolen goods can be "dyed" with the suspension of the compound used, either per se or together with dyestuffs in the presence of the customary additions in the usual manner. Di-pentachlorophenyl-mono-hydrogen phosphate is substantive to wool and compatible with the usual dyestuffs.

Alternatively, the goods may be padded through a dispersion in water under either acid, neutral or alkaline conditions, squeezed and dried, although the "dyeing" method is preferred.

Finally, the phosphates can be applied in non-aqueous media. For example, the acid phosphate from methylated spirits solution. Textiles containing as little as 0.5 per cent of the acid phosphate are immune from the attack of even the carpet beetle, *Attagenus piceus* and retain this immunity after repeated washings with soap

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or soap and soda or after frequent commercial dry cleaning operations.

We claim:

A method for the protection of a woolen textile material against biological attack which comprises impregnating said textile with di-pentachlorophenyl-monohydrogen phosphate.

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