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ANTI-VESICANT COMPOSITION

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The invention described herein may be manufactured and used by or for the Government for governmental purposes without the payment to me of royalty thereon.

This invention relates generally to protective clothing adapted to afford personnel protection against known chemical warfare vesicant agents such as mustard gas and lewisite, and the invention relates in particular to compositions to be used in providing such protective clothing.

It has been found that known chemical warfare vesicant agents, such as mustard gas and lewisite, may be rendered non-vesicant and relatively harmless by some type of halogenation, and particularly by chlorination, so as to convert the vesicants into innocuous products. Conceivably, any compound having an available halogen atom may be considered as having possible use as an anti-vesicant agent for halogenating or chlorinating vesicants. However, due to practical considerations, the chloramides have been found to be most suitable, as a class, to be used as chlorinating agents. Of the class of chloramides, bis dichlorophenyl chlorurea and bis 2,4,6-trichlorophenyl chlorurea have been found to be very satisfactory anti-vesicant agents.

Considerable difficulty has been encountered in the past in developing compositions and techniques whereby fabric and clothing may be treated with the anti-vesicant agents so as to leave sufficient deposits thereof in the fibers of the fabric or clothing to give effective protection against vesicants and which will adhere firmly thereto so as to resist a reasonable amount of wear and laundering. Generally, the anti-vesicant agents and suitable inert binders have been dissolved in various volatile organic solvents so as to provide a so-called "impregnating composition." The fabric and clothing have then been soaked or dipped into such an impregnating composition so as to become thoroughly wetted therewith. The fabric or clothing is then allowed to dry so that the volatile solvent will evaporate leaving the anti-vesicant agent held on the fibers of the fabric or clothing by the inert binder.

Due to the relatively low solubilities of the anti-vesicant agents in the organic solvents, it has not been possible to obtain at ordinary temperatures as high concentrations of the anti-vesicant agents as desired. This difficulty may be overcome to a limited degree by heating the solutions to provide increased solubility of the anti-vesicant agents. However, such heating is objectionable since the anti-vesicant agents are easily decomposed at rather low temperatures. Furthermore, the resort to such heating is very objectionable in connection with field impregnation since heating in the field is inconvenient, difficult to control, and often impossible to accomplish for various reasons.

The time, space, and labor requirements in connection with the drying operation to remove the volatile solvent is also a very important factor. Heretofore, due to the relatively low concentrations of anti-vesicant agents in

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the impregnating compositions, a large amount of solvent evaporation was required for the amount of anti-vesicant agent deposited in the fabric or clothing.

In view of the foregoing difficulties of the prior art, one of the principal objects of the present invention is the provision of impregnating compositions for treating fabric or clothing to render the same resistant to chemical warfare vesicants, such as mustard gas and lewisite, which compositions contain in uniform distribution, and at ordinary temperatures, amounts of anti-vesicant or neutralizing agents in excess of the amounts normally soluble therein. Such improved and relatively concentrated impregnating compositions permit very satisfactory impregnation of fabric and clothing at ordinary temperatures, and eliminate the most objectionable difficulties of the prior art in regard to the heating of the impregnating compositions and the extended drying periods required to remove the volatile solvents.

A further object of this invention is the provision of impregnating compositions containing dyeing or coloring material whereby fabric and clothing may, in one operation, be simultaneously dyed and rendered resistant to chemical warfare vesicants such as mustard gas and lewisite.

Still another object of the invention is the provision of an improved technique of providing protective clothing wherein clothing or fabric is given a conditioning treatment which involves using bleaching agents to eliminate impurities in the clothing or fabric prior to the impregnation of the same with an anti-vesicant agent.

Other objects of the invention will, in part, be obvious and will in part appear hereinafter.

Briefly stated, a principal feature of this invention resides in the inclusion in an impregnating composition of an ingredient which serves to maintain a substantially uniform distribution throughout the composition of an amount of an anti-vesicant agent in excess of that amount thereof which would be normally soluble and remain uniformly distributed. When the impregnating composition has only one liquid phase, the added ingredient serves as a suspending agent, while in certain instances where an impregnating composition is in the form of an emulsion having two liquid phases, the added ingredient appears to function as an emulsifying agent.

For a more complete understanding of the nature and scope of this invention, reference may be had to the following detailed description mentioning certain specific compositions and techniques as illustrative.

One type of impregnating compositions embodying the invention has a single liquid phase and comprises, in general, an anti-vesicant agent having available chlorine for destroying mustard gas, lewisite and like vesicants, and a binder, the anti-vesicant agent and binder being dissolved in an organic solvent in which a suspending agent has been dissolved. The anti-vesicant agents may be such compounds as bis dichlorophenyl chlorurea or bis 2,4,6-trichlorophenyl chlorurea, both compounds being selected from the general class of chloramides. Several binders may be used which are inert to the anti-vesicant agents, such as chlorinated naphthalene (Halowax) and chlorinated paraffin. Various organic solvents may be used such as monochlorobenzene, beta trichlorethane, and perchlorethylene. These solvents may be classed as chlororganic solvents. As an example of a suspending agent, ethyl cellulose has been found to be very satisfactory for the purposes intended.

Impregnating compositions may include the foregoing ingredients or components in various percentage ranges. By way of example, very satisfactory impregnating com-

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positions may be made according to the following formula:

	Percent by weight
Ethyl cellulose.....	0.3-0.5
Monochlorbenzene.....	50.0-60.0
Beta trichlorethane.....	30.0-34.0
Chlorinated naphthalene.....	1.5-2.5
Chlorinated paraffin.....	3.0-4.0
Bis 2,4,6-trichlorophenyl chlorurea.....	5.0-8.0

An impregnating composition having the above general formula may be prepared according to the following steps:

(1) The ethyl cellulose is dissolved in the monochlorbenzene and beta trichlorethane to give a clear, colorless solution.

(2) The chlorinated naphthalene and chlorinated paraffin are added to the solution.

(3) Finally, and preferably just prior to the use of the impregnating composition, the bis 2,4,6-trichlorophenyl chlorurea is added.

(4) On shaking or other mild agitation, a stable suspension of low viscosity results. All steps are carried out at ordinary temperatures.

The technique of impregnating clothing with the foregoing type of impregnating compositions is simple and may be referred to as the "suspension process." Clothing, without previous drying, is immersed in the impregnating composition and soaked for a few minutes so as to thoroughly wet the same. The clothing is then removed and wrung out so as to remove the impregnating composition in excess of that required for adequate impregnation. It will be noted that the impregnation is carried out at ordinary temperatures without treating, and is adapted to either large, or small scale field impregnation.

According to one embodiment of the invention, impregnating compositions may be prepared which may be used to simultaneously dye and impregnate fabric and clothing in one stage at ordinary temperatures. Several classes of dyestuffs may be used, such as: solvent soluble anthraquinone dyestuffs; water soluble anthraquinone dyestuffs; pigment powders; pigment pastes; and, flushed pigments. The flushed pigments have been found to be very suitable for the purpose intended. These pigments are produced by first grinding the pigment in water and then transferring or "flushing" the pigment particles to a medium such as one consisting of ethyl cellulose and monochlorbenzene. The resulting flushed pigment composition may then be readily incorporated in the base solution used in the suspension process for clothing impregnation.

While the present invention was particularly concerned with imparting a khaki shade to white cotton drill, it will be understood that any desired color may be imparted to clothing or fabric by the proper selection of the pigments in the flushed pigment mixture. A typical formula for an impregnating composition adapted to provide for the simultaneous dyeing and impregnation of white cotton drill to give a khaki shade to the clothing is as follows:

	Percent by weight
Ethyl cellulose.....	0.28 or greater.
Monochlorbenzene.....	47.0-56.4.
Beta trichlorethane.....	28.2-32.0.
Chlorinated naphthalene.....	1.4-2.4.
Chlorinated paraffin.....	2.8-3.8.
Flushed pigment (khaki).....	About 6%.
Bis 2,4,6-trichlorophenyl chlorurea.....	4.7-7.5.

An impregnated and dyeing composition following the above general formula, may be prepared according to the following steps:

(1) The ethyl cellulose is dissolved in the monochlorbenzene and beta trichloroethane, giving a clear, colorless solution.

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(2) The chlorinated naphthalene and chlorinated paraffin are added to the solution of step 1.

(3) The flushed pigment is thoroughly mixed into the solution from step 2.

(4) Finally, and preferably just prior to the use of the composition, the bis 2,4,6-trichlorophenyl chlorurea is added with shaking or other mild agitation.

The technique of treating fabric or clothing with a combined impregnating and dyeing composition is the same as that described hereinbefore in connection with the compositions used only for impregnation.

According to a further embodiment of the invention, emulsion-type impregnating compositions have been prepared which can be used for impregnating clothing or fabric with anti-vesicant material. These emulsions require strong mechanical agitation such as obtained by a colloid mill or homogenizer. Several types of emulsifying agents may be used to impart reasonable stability to the emulsions. The following is a list of some of the emulsifying agents that may be used:

Sulfated fatty alcohols
Sodium octyl sulfate
Sodium lauryl sulfate
Abietene sodium sulfonate
Alkyl naphthalene sodium sulfonate
Water soluble petroleum sulfonates
Sulfonated coconut oil
Sodium dioctyl sulfosuccinate
Diglycol laurate
Sulfonated castor oil

A typical impregnating composition may have the following composition:

	Percent by weight
Monochlorbenzene.....	15
Perchloroethylene.....	10
Chlorinated paraffin.....	5
Emulsifying agent.....	3
Bis 2,4,6-trichlorophenyl chlorurea.....	7
Water.....	60

The emulsion is prepared by first making a mixture of the monochlorbenzene, perchloroethylene, chlorinated paraffin, emulsifying agent, and bis 2,4,6-trichlorophenyl chlorurea. The proper amount of water is then added to the above mixture, and the complete mixture is then passed thru a colloid mill to give a homogeneous, stable emulsion.

Clothing and fabric may be impregnated in the emulsions at ordinary temperatures in the usual manner.

Since certain changes and modifications may be made in the foregoing compositions and techniques of application without departing from the scope of this invention, it is intended that all matter contained in the above description shall be interpreted as illustrative and not in a limited sense.

What is claimed as new is:

1. A fabric impregnating composition for rendering fabric and clothing resistant to mustard gas, lewisite, and like vesicants susceptible of being rendered non-vesicant by chlorination, which comprises, bis 2,4,6-trichlorophenyl chlorurea for rendering mustard gas, lewisite, and like vesicants relatively harmless, a binder comprising a mixture of chlorinated paraffin and chlorinated naphthalene, a solvent comprising a mixture of monochlorbenzene and beta-trichlorethane, and a small amount of ethyl cellulose as a suspending agent for maintaining a relatively stable suspension of bis 2,4,6-trichlorophenyl chlorurea in the solvent.

2. A fabric impregnating composition for rendering fabric and clothing resistant to mustard gas, lewisite, and like vesicants susceptible of being rendered non-vesicant by chlorination, which comprises, from about 5.0% to 8.0% by weight of bis 2,4,6-trichlorophenyl chlorurea for rendering mustard gas, lewisite, and like vesicants relatively harmless, a binder comprising a mixture of from

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about 1.5% to 2.5% by weight of chlorinated naphthalene and from about 3.0% to 4.0% by weight of chlorinated paraffin, a solvent comprising a mixture of from about 50.0% to 60.0% by weight of monochlorobenzene and from about 30.0% to 34.0% by weight of beta-trichlorethane, and about 0.3% by weight or greater of ethyl cellulose as a suspending agent for maintaining a relatively stable suspension of bis 2,4,6-trichlorophenyl chlorurea in the solvent.

3. A fabric impregnating composition for simultaneously coloring and rendering fabric and clothing resistant to mustard gas, lewisite, and like vesicants susceptible of being rendered non-vesicant by chlorination, which comprises, bis 2,4,6-trichlorophenyl chlorurea for rendering mustard gas, lewisite, and like vesicants relatively harmless, a binder comprising a mixture of chlorinated paraffin and chlorinated naphthalene, flushed pigment for coloring said fabric or clothing, a solvent comprising a mixture of monochlorobenzene and beta-trichlorethane, and a small amount of ethyl cellulose as a suspending agent for maintaining a relatively stable suspension of bis 2,4,6-trichlorophenyl chlorurea in the solvent.

4. A fabric impregnating composition for simultaneously coloring and rendering fabric and clothing resistant to mustard gas, lewisite, and like vesicants susceptible of being rendered non-vesicant by chlorination, which comprises, from about 4.7% to 7.5% by weight of bis 2,4,6-trichlorophenyl chlorurea for rendering mustard gas, lewisite, and like vesicants relatively harmless, a binder comprising a mixture of from about 1.4% to 2.4% by weight of chlorinated naphthalene and from about 2.8% to 3.8% by weight of chlorinated paraffin, about 6% by weight of a flushed pigment for coloring said fabric or clothing, a solvent comprising a mixture of from about 47.0% to 56.4% of monochlorobenzene and from about 28.2% to 32.0% by weight of beta-trichlorethane, and about 0.28% by weight or greater of ethyl cellulose as a suspending agent for maintaining a relatively stable suspension of bis 2,4,6-trichlorophenyl chlorurea in the said solvent.

5. A fabric impregnating composition for rendering fabric and clothing resistant to mustard gas, lewisite, and like vesicants susceptible of being rendered non-vesicant by chlorination, and which is in the form of an oil-in-water emulsion having an oil phase comprising bis 2,4,6-

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trichlorophenyl chlorurea for rendering mustard gas, lewisite, and like vesicants relatively harmless, a binder comprising a mixture of chlorinated paraffin and chlorinated naphthalene, and a solvent comprising a mixture of monochlorobenzene and beta-trichlorethane, and having an emulsifying agent to stabilize the emulsion.

6. A fabric impregnating composition for rendering fabric and clothing resistant to mustard gas, lewisite, and like vesicants susceptible of being rendered non-vesicant by chlorination, and which is in the form of an oil-in-water emulsion having an oil phase comprising about 7% by weight of the composition of bis 2,4,6-trichlorophenyl chlorurea for rendering mustard gas, lewisite, and like vesicants relatively harmless, a binder comprising about 5% by weight of the composition of chlorinated paraffin, and a solvent comprising a mixture of about 15% by weight of the composition of monochlorobenzene and about 10% by weight of the composition of perchlorethylene, having a water phase comprising about 60% by weight of the composition, and having an emulsifying agent to stabilize the emulsion comprising about 3% by weight of the composition.

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