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3,464,847  
**TREATMENT OF FABRICS WITH ANTHRANILIC ACID AND SILVER NITRATE SOLUTIONS**

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7 Claims

**ABSTRACT OF THE DISCLOSURE**

A method for imparting antivesicant properties to air permeable fabrics, comprising treating the fabrics with a solution of anthranilic acid and silver nitrate.

The present invention relates to an improved method for imparting antivesicant properties to cellulosic fabrics and more particularly to imparting antivesicant properties to cellulosic fabrics by impregnating or modifying them with chelates derived from anthranilic acid and silver nitrate.

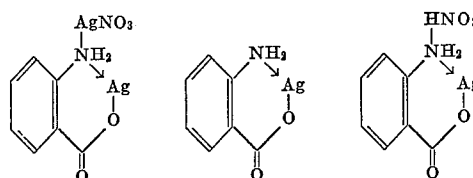
Vesicant agents, such as mustard gas, attack the skin as well as the respiratory system of exposed personnel. Therefore, a body shield, in addition to a protective mask, is essential to prevent casualties. A fabric impermeable to all fluids would afford complete protection, but would be impractical, especially in warm climates, for extended periods of wear. More preferable is protection built into occupational clothing; thereby permitting air circulation and preventing moisture accumulation.

Mustard gas is the term applied to the well-known vesicant, bis(2-chloroethyl) sulfide. Standard mustard gas exists as a vapor or as airborne droplets. Various processes and compounds have been suggested and employed to impart finishes to the fabric used for military uniforms which would deactivate mustard gas and would prevent its passage through the cloth. But the efficiency of prior methods leaves much to be desired. For example, among the most commonly used are the N-chloroamides. Once on the fabric, these compounds lose activation by hydrolysis, atmospheric moisturization, perspiration, thermal decomposition, and the like.

The present invention is premised on the discovery that certain silver chelates or complexes, particularly those of anthranilic acid, are effective agents for applying to fabrics and for imparting superior antivesicant properties thereto. Accordingly, it is among the objects of the present invention to provide a treatment for air-permeable cellulosic fabric materials that will prevent passage of vesicant agent vapors through the garment. It is another object to provide a noninjurious finish for such fabric materials which requires no further treatment by the wearer for antivesicant protection. It is a final object to provide a vesicant-resistant treatment for textile fabrics that is relatively stable and that does not adversely affect the tensile strength of the fabric. These and other objects and advantages of the instant invention will be fully apparent from the following description and examples.

The silver compounds employed in the method of the instant invention provide surprisingly effective results. Their use produces effective fabric barriers to mustard gas which are stable to moisture and to temperature up to about 150° C. The mustard gas is inactivated and its passage prevented through the cellulosic cloth. The high effectiveness of the silver chelates or complexes of anthranilic acid is unexpected in view of the slight effectiveness of anthranilic acid itself and of other metal chelates of anthranilic acid such as copper and iron.

In its simplest form, the invention is practiced by padding a solution of anthranilic acid onto the fabric, air drying, treating the fabric with silver nitrate solution, and drying once more. The padding process involves submerging the fabric in the solution, permitting it to soak for a few minutes, and gently squeezing it between rollers. The degree of antivesicant effect is adjusted by the amount of silver anthranilic complex applied. Although the exact nature of the silver complex which affords the vesicant protection has not been completely identified, it is probably at least one of the following: the silver nitrate complex of the silver salt, the silver salt of anthranilic acid, and the nitric acid salt of the silver complex. Each of these are formularized below:



A possible alternative to the method set forth above is to first form the complex by reaction of anthranilic acid with silver nitrate and then apply the reaction product by padding a solution or dispersion thereof on the fabric.

The efficiency of the mustard gas barrier is determined by the "Dawson Test"—a measurement of the percent of mustard gas, initially present in an air stream, retained on a fabric after air passage through the fabric. Table I following Example I depicts the relative effectiveness of the silver derivative of anthranilic acid as compared with anthranilic acid itself and with the copper and iron derivatives; all applied as prescribed in Example I.

**EXAMPLE I**

A piece of cloth is soaked for about one hour in a 20% solution of anthranilic acid in dimethyl formamide, then dried in air at room temperature (20-25° C.) for approximately twenty hours.

The dry fabric is then soaked without agitation for about one hour in a large excess of a 10% aqueous solution of silver nitrate (AgNO<sub>3</sub>), is squeezed gently through a padder, and is air dried at room temperature. Analysis of the fabric showed it to contain 0.14% nitrogen and 8.4% silver by weight.

TABLE I

Compound applied	Mustard retention, percent		
	0-2 hrs.	2-4 hrs.	4-6 hrs.
Anthranilic acid.....	26		
Cupric anthranilate.....	26		
Silver anthranilate.....	98	98	97
Ferric Anthranilate.....	20		

**EXAMPLE II**

To a solution of 27.4 parts (0.20 m.) of anthranilic acid of 99.7% purity in 200 parts by volume of anhydrous 2B alcohol, is added a solution of 34.0 parts of silver nitrate (0.20 m.) in 25 parts by volume of water. The addition is made slowly at 50-55° C. with good stirring. A granular precipitate forms. After stirring for an additional 15 minutes at 50-55° C., the reaction mixture is filtered rapidly while hot and the solid dried overnight at 50-55° C. This crude product consists of 11.2 parts of sparkling light brown crystals which decompose sharply at 160° C. with the immediate formation of a silver mirror. The crude is extracted with 100 parts by volume of boiling anhydrous-2B alcohol and dried at 50-55° C.; 10.6 parts of sparkling gray violet crystals result. These crystals also decompose sharply at 160° C. with the concomitant formation of a

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silver mirror. Analysis disclosed a product having an empirical formula  $C_7H_6N_2O_5Ag_2$ .

The product so formed is applied by padding a dispersion of it on the fabric. In a standard test procedure, 60 micrograms of mustard gas treated with 4000 micrograms of the product was completely inactivated.

### EXAMPLE III

The alcohol filtrate from Example II, when permitted to stand overnight at room temperature deposits 27.8 parts of large light violet crystals. These are purified by dissolving in 250 parts by volume of boiling anhydrous 2B alcohol clarifying with adsorbent charcoal, concentrating the filtrate to a small volume, filtering at room temperature, washing the crystals with alcohol, and drying overnight at 50–55° C. The recrystallized product consists of 14.0 parts of light violet crystals which melt at 155–158° C., evolve gas, but don't form a silver mirror. Analysis shows the empirical formula to be  $C_7H_7N_2O_5Ag$ . The product is applied to the cloth as a dispersion by padding as in Example II. It gives standard test results identical with the product of Example II—total inactivation of mustard gas.

While only preferred forms of the invention are shown and described, other forms of the invention are contemplated and numerous changes and modifications may be made therein without departing from the spirit of the invention as set forth in the appended claims.

What is claimed is:

1. The method of imparting antivesicant properties to an air permeable fabric by treating said fabric with a solution of the reaction product of silver nitrate and anthranilic acid, and drying said fabric.

2. The method set forth in claim 1, wherein said treatment consists of submerging said fabric in said solution, and gently squeezing it between rollers prior to drying.

3. The method set forth in claim 1, wherein said anthranilic acid consists of a 20% solution of anthranilic

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acid in dimethyl formamide and said silver nitrate consists of a 10% aqueous solution.

4. The method of imparting antivesicant properties to an air permeable fabric by treating said fabric with a solution of anthranilic acid, drying and then treating the fabric with a solution of silver nitrate.

5. The method set forth in claim 4 wherein said anthranilic acid consists of a 20% solution of anthranilic acid in dimethyl formamide and said silver nitrate consists of a 10% aqueous solution.

6. The method set forth in claim 4, wherein said treatment consists of soaking said fabric in said acid solution for about one hour, drying said soaked fabric in air at room temperature for about twenty hours, soaking said dried fabric without agitation for about one hour in an excess of said silver nitrate solution and squeezing said fabric gently through rollers.

7. An air permeable fabric vested with antivesicant properties comprising a fabric treated with the reaction products of silver nitrate and anthranilic acid.

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