March 25, 1952 J. ZIMMERMAN ET AL 2,590,811

SHRINKAGE RESISTANCE TREATMENT OF WOOLEN GOODS

Filed April 13, 1950 2 SHEETS—SHEET 1

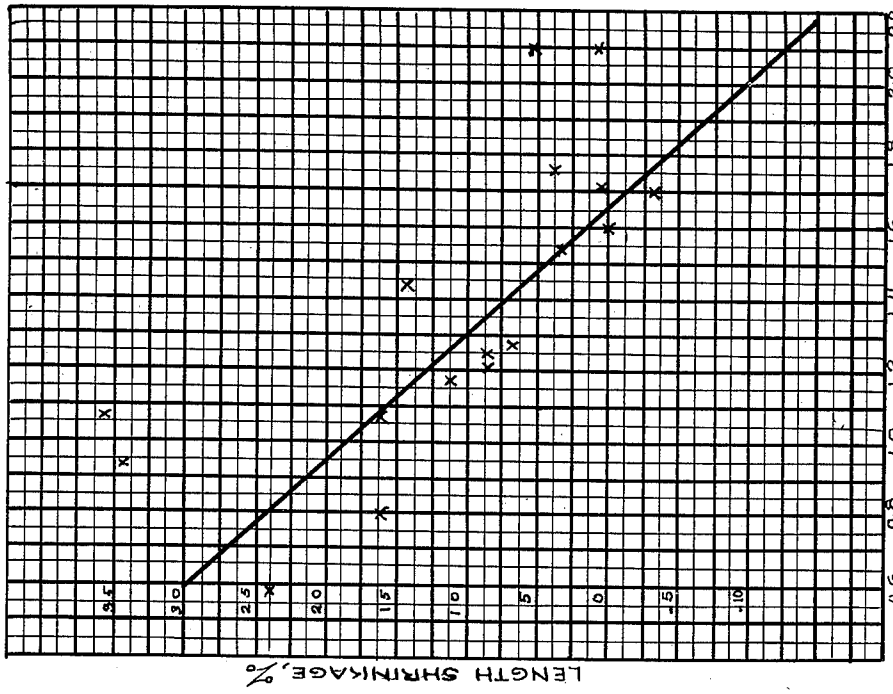


Fig. 2

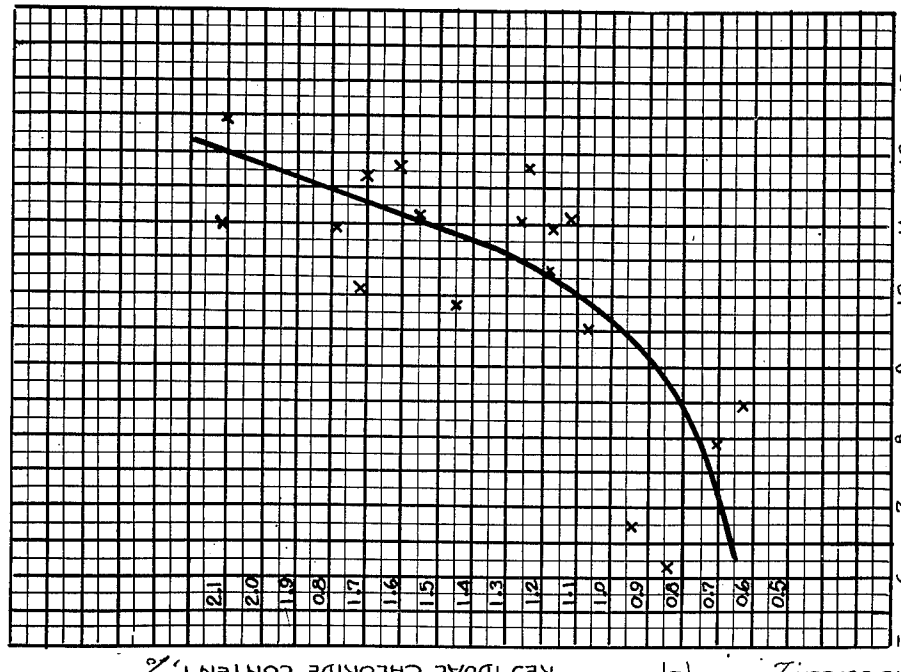


Fig. 1

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2 SHEETS—SHEET 2

Fig. 3

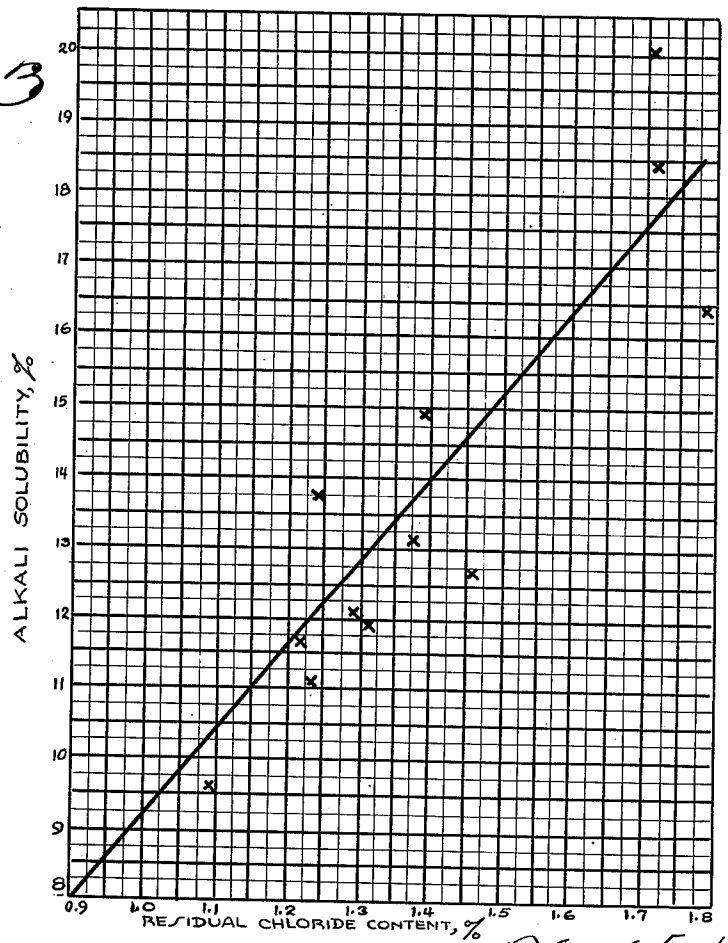


Fig. 4

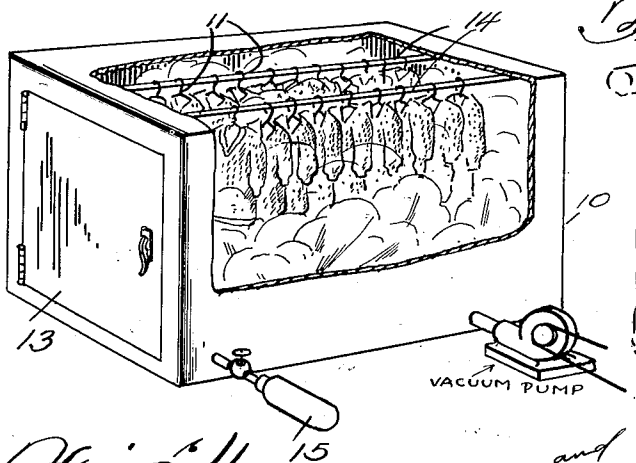
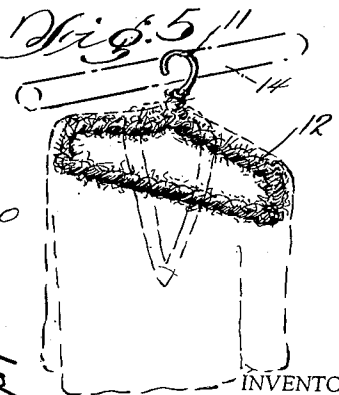


Fig. 5



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Patented Mar. 25, 1952

2,590,811

UNITED STATES PATENT OFFICE

2,590,811

SHRINKAGE RESISTANCE TREATMENT OF WOOLEN GOODS

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Application April 13, 1950, Serial No. 155,704

17 Claims. (Cl. 8—128)

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This invention relates to a treatment for imparting shrink resistance to woolen garments, and is particularly applicable to knitted woolen body garments such as sweaters and underwear.

Our shrink resistant imparting process is a chlorination process, wherein the woolen garments are contacted in a closed chamber with chlorine gas. Basically, a process of this type has been set forth in British Patents Nos. 417,719, 475,742 and 493,098 and U. S. Patent No. 2,373,974.

It was heretofore the opinion of the trade that the moisture content of woolen garments to be treated in accordance with dry gaseous chlorination methods set forth in the above British patents must not exceed 9%, because it was thought that a high moisture content of the woolen garments would result in irregular and excessive chlorination and development of deposition of hydrochloric acid on the goods ($H_2O+Cl_2 \rightarrow HOCl+HCl$), with consequent damage to the woolen fiber and to the cotton seams holding the parts of the garment together.

We have found surprisingly, that woolen garments may be made shrink resistant by the dry chlorination method at initial moisture contents of the goods of from about 10.5% to about 12%, without irregularities and hydrochloric acid damage, if further precautions, about to be described in detail, are taken to prevent irregular condensation of hydrochloric acid on the goods. On the other hand, we have found that an initial moisture content of 9% or less is insufficient to impart enough shrink resistance through the dry chlorination method to comply with Armed Forces specifications.

Thus, it is a major object of our invention to adapt the dry chlorination processes of the aforementioned patents to the treatment of woolen garments, particularly knit sweaters and underwear, for military and civilian uses, wherein a high degree of shrink resistance is obtained without a substantial sacrifice in strength.

A further object of our invention is a reduction of the amount of hydrochloric acid deposited on woolen goods treated by the dry chlorination method.

A further object of our invention is the adjustment of the residual chloride content of dry-chlorinated woolen garments to an amount which imparts the desired shrink resistance to the garments without substantially impairing their fiber strength and seam strength.

Yet another object of our invention is the provision of woolen body garments provided with cotton seams, as for instance, sleeved sweaters,

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which may be washed in hot water, without undue shrinkage.

Other objects and advantages of our invention will become apparent from the following detailed description of several examples whereby our invention may be practiced.

In the accompanying drawings:

Fig. 1 is a graph wherein initial moisture content of a sweater manufactured in accordance with Army specifications is plotted against residual chloride content;

Fig. 2 is a graph wherein the residual chloride content of the same sweater, after dry chlorination treatment, is plotted against length shrinkage;

Fig. 3 is a corresponding graph wherein residual chloride content is plotted against wool fiber damage as measured by the alkali solubility test;

Fig. 4 is a diagrammatic view of a chamber for the dry chlorination of woolen garments in accordance with our invention; and

Fig. 5 is a perspective view of a garment hanger provided with a textile surface or other surface capable of reducing condensation of hydrochloric acid on a woolen garment hung therefrom during the carrying out of the dry chlorination.

We have observed in numerous experiments on which the graphs of Figs. 1 to 3 are based, that (1) at and above a moisture content of about 10.5% of woolen garments, prior to treatment, the residual chloride content increases in substantially linear progression as the initial moisture content upon dry chlorination of the garments is increased; (2) that the length shrinkage of a dry chlorinated woolen garment decreases substantially inversely proportionally to its residual chloride content; and (3) that the damage to the woolen fibers, as measured by the alkali solubility test, increases substantially proportionally to the residual chloride content. On the other hand, we have observed that, as the initial moisture content of the chlorinated garments is adjusted to less than 10%, the residual chloride content does not drop a linear progression but in a gradually diminishing curve. The residual chloride content imparted by dry chlorination of woolen garments having an initial moisture content of less than about 10% is so low that not enough shrinkage resistance is imparted to the garments to enable them to comply with military specifications or with civilian specifications of comparable strictness.

As will be seen from Fig. 1 of the drawings, the "knee" of the curve of the graph which plots initial moisture content against residual

chloride after dry chlorination lies between the moisture content of 9%–10%, corresponding (at 2% chlorination) to a residual chloride content of about 0.9% to about 1.1%. However, the graph of Fig. 2 shows that at a residual chloride content of 1.1%, the length shrinkage of the sweater is still about 15%; this is not acceptable in goods for the Armed Forces and in civilian goods that lay claim to being shrink-resistant. Such a sweater would need either dry cleaning or washing in lukewarm water, which in civilian life is often inconvenient and commercially expensive, while under field conditions in the Armed Forces, such special treatment may be wholly unobtainable at times.

Under current military specifications, length or width shrinkage of a sweater shall not exceed 8%. This requirement can be attained by the dry chlorination method if initial moisture content of the goods is adjusted to between about 10½% and about 12%, or correspondingly, if the residual chloride content of the dry chlorinated goods is maintained at between about 1.2% and about 1.7%. At a residual chloride content of about 1.4%, length shrinkage is reduced to about 7.5%; the residual chloride content of 1.4% corresponds to an initial moisture content of the sweater of about 10¾%. At a residual chloride content of about 1.6% (corresponding to an initial moisture content of about 11¼%), length shrinkage is almost zero; at this residual chloride content, wool fiber damage, as measured in terms of alkali solubility is about 16.5%, which is within the acceptable limit for Armed Forces woolen garments. Above a residual chloride content of about 1.7% the sweater undergoes an undesired lengthening during hot water washing, while wool fiber damage, as measured by the alkali solubility test likewise exceeds tolerable limits (which are about 18–21%) as the residual chloride content is substantially increased above 1.7%. Thus, at a residual chloride content below 1.2% (corresponding to an initial moisture content of below 10¼%), chlorination is insufficient to impart acceptable shrinkage resistance to the garment; and conversely, above a residual chloride content of substantially 1.7% (as the initial moisture content exceeds about 12%) there is an undesired lengthening of the garment upon washing, as well as an unacceptable increase of wool fiber damage. On the other hand, in the residual chloride content range of from about 1.2% to about 1.7%, there is a "give and take" in the sense that shrinkage gradually approaches zero as the residual chloride content approaches the upper limit of the foregoing range, while wool fiber damage in terms of the alkali solubility test still remains within acceptable limits within that chloride content range.

We have found that shrink resistance without undue weakening of the wool fabric and cotton seams is achieved by about 1½% to about 3% chlorination; i. e. by contacting the woolen goods with an atmosphere containing chlorine gas in an amount equal to from about 1½% to about 3% on the weight of such woolen goods. With less than 1½% chlorination, the shrink resistance imparted to the goods is usually insufficient to meet Armed Forces specifications and to come up to a degree of shrink resistance acceptable for better grade civilian goods designated as "shrink resistant." On the other hand, when chlorination exceeds about 3%, we have observed that damage to the wool and to the cotton seams is frequently incurred. We prefer to use 2% chlorination and to admit the chlorine gas at the rate of about 4 pounds per hour into an autoclave having an average load, e. g. 75 sweaters weighing 1 lb. 1 oz. each or a total of about 80 pounds. The goods should preferably remain in contact with the chlorine gas for about 30 to 40 minutes. For best results the temperature in the autoclave should be maintained at a temperature above about 70° F., preferably near about 80° F., as below 70° F. undesired condensation of HCl takes place.

If chlorination is at other than the 2% rate, residual chloride content of the treated goods changes correspondingly. Thus, it was determined that while a sweater with 11% moisture content prior to chlorination received a 1.3% residual chloride content as the result of 2% chlorination (i. e. exposure to chlorine gas weighing 2% of the sweater's weight), the same initial moisture content (11%) resulted in a residual chloride content of 1.4% after 3% chlorination.

The chlorination chamber may be evacuated prior to admission of the chlorine gas (e. g. as described in British Pat. #417,719); but this is not necessary for the successful operation of our process, which can be practiced by introducing chlorine gas into the chamber containing sweaters at atmospheric pressure. We prefer to recirculate the chlorine gas, for reasons of economy, and for more even and thorough chlorination of the goods. The chlorination chamber should be pressure-tight, for obvious reasons.

Adjustment of the pH of the woolen goods (e. g. sweaters) prior to chlorination to the acid side, by pretreatment with acids down to a pH of about 3 (such as acetic, imparting a pH of about 4.5, or formic acid, imparting a pH of about 3.3), was found to have little, if any, effect on the breaking strength and seam failures of the goods after chlorination. Upward pH adjustment up to about 9, e. g. by a corresponding pretreatment with a mild alkali such as sodium bicarbonate likewise had no substantial effect in terms of breaking strength and seam failures, but above a pH of about 8 appeared to lessen the shrink resistance imparted by the dry chlorination process. The method of determining pH of a water extract of the woolen goods is accomplished in accordance with the procedure described in Federal Specification CCC–T–191*a*, section VII, par. 1*a* (3), Supplement, October 8, 1945, page 48.

We have found that damage to woolen garments, caused by uneven deposition thereon of hydrochloric acid during the dry chlorination step can be avoided by suspending each garment individually from a hanger **11** having a non-condensing, hygroscopic surface **12**, and with a low heat capacity. We prefer wool-covered hangers, but have found other textile-covered hangers, e. g. hangers covered with nylon fibers, glass fibers or mixtures of these fibers with wool fibers, to be useful for this purpose. While the base material of the hangers may be any suitable substance (wood, plastic), we prefer a conventional wire hanger for reasons of cheapness and overall sturdiness.

After the garments are placed on covered hangers as described, they should be introduced into the pressure-resistant autoclave **13** by suspending the hangers within such autoclave, e. g. from horizontal rods **14**. It is undesirable to pack the garments too tightly together, because it is essential for uniform chlorination that the chlorine gas be permitted to circulate into contact

with all parts of the goods; for this reason, the garments should be spaced from each other and from the walls of the chamber.

Even with the foregoing precaution, it is necessary to submit the goods to a neutralization treatment after completion of the chlorination step, to remove excess residual acid which, if permitted to remain on the goods, would weaken the wool fabric and particularly the cotton seams. Five per cent sodium bicarbonate (based on the weight of the load) is suitable. The treatment is conveniently accomplished in a loose stock dye machine having an approximate 25:1 or 20:1 liquor ratio. The treatment may be carried out at room temperature for a period of about 15 to 20 minutes followed by a brief rinse. The efficacy of the last rinse can be increased by adding conventional cationic finishing agents. Other weak alkalies, e. g. sodium carbonate or ammonium hydroxide may be used instead of sodium bicarbonate.

The following examples illustrate several typical treatments of woolen sweaters in accordance with our invention:

Example I

75 woolen sweaters (80 lbs.), finished with cotton seams, and with a controlled moisture content of 10.5%, were placed on individual wool-covered wire hangers and loosely placed in an autoclave. The autoclave was exhausted to a vacuum of about 26" of Hg, and 1.6 pounds of chlorine gas were admitted at a rate of 4 pounds per hour. Contact between chlorine and sweaters was maintained for about 40 minutes. The sweaters were then removed from the autoclave and immediately subjected to a neutralization treatment with 5% sodium bicarbonate (based on weight of sweaters), followed by rinse. It was found that no substantial color change had taken place. Residual chloride content of the sweaters was determined to be 1.2%. Alkali solubility of the treated sweaters was 11.7%. Length shrinkage, after hot water washing in accordance with the standard procedure described below, was 8%.

Example II

The treatment described in Example I was repeated with sweaters whose moisture content had been adjusted to 11%. The median residual chloride content of five sweaters picked at random from the treated lot was 1.5%; alkali solubility 15.0%; length shrinkage 4.3%.

Example III

The procedure of Example I was repeated with a lot of sweaters whose moisture content had been adjusted to 11¾%. The median residual chloride content of three sweaters picked at random from the treated lot was 1.6%; alkali solubility 16.2%; length shrinkage 1.4%.

In a control experiment, wherein initial moisture content in the sweater was adjusted to 8%, residual chloride content was only .7% after dry chlorination, and length shrinkage was about 25%.

The residual chloride content of the woolen goods is a measure of the degree of chlorination, and may be determined by the following test:

Take 1.5 grams chlorinated wool, place in stoppered bottle containing 75 ml. liquor made up of 73 ml. distilled water and 2 ml. normal sodium carbonate. Sample should be pulled apart and allowed to stand over night or be shaken for ½ to 1 hour. Filter off and take 50 ml. of filtrate for analysis. Acidify with 2 or 3 drops concentrated nitric acid and add exactly 10 ml. decinormal silver nitrate. Then add 5 ml. nitrobenzene to coagulate the precipitate. Add ½ to 2½ ml. ferric alum indicator and titrate with decinormal potassium thiocyanate.

Example

Chlorinated sample weight:
As received, 1. 5 grams
Corrected dry weight, 1. 36 grams
Titration based on 50 ml. taken from 75 ml. of solution
Volume of silver nitrate added: 10 ml. of N/10 $AgNO_3$
Volume of potassium thiocyanate consumed in titration: 6. 9 ml.
Volume of silver nitrate combined: 10−6. 9=3. 1 ml.

$$\text{Chloride content per milliliter}=\frac{3.55}{1000}=0.00355 \text{ gram}$$

$$\text{Residual chloride} = 3.1\times 0.00355\times\frac{75}{50}\times\frac{100}{1.36}$$

Residual chloride =1. 21 per cent

The alkali solubility test, as a measure of wool fiber damage, is described in Federal Specification CCC–T–191, sec. VII, par 3*a* (3) Supplement, October 8, 1945, page 49. It essentially consists of placing a sample of wool in 100 ml. of N/10 sodium hydroxide for 1 hour at 65° C., followed by rinsing and drying. The alkali solubility is the resulting per cent loss suffered by the sample.

$$\frac{W_0-W_t}{W_0}\times 100=\text{alkali solubilty (per cent)}$$

wherein W_0 is weight prior to treatment and W_t is weight after treatment. The quality loss expressed by this test expresses the fiber damage caused by the shrink resistance imparting treatment, chiefly believed to be due to a partial splitting of the disulfide cross linkages of the wool structures.

A standard test for shrink resistance is carried out as follows: The woolen garments are washed in water at 140° F., not over 50 p. p. m. hardness and buffered to a pH of 7. A six pound load is introduced in a standard rotary reversing type washing machine having a cylinder which measures 24 inches in diameter by 24 inches in length and agitated therein with such water for 1 hour. Thereafter, the garments are centrifugally extracted, and dried in a 36 x 24 inch tumbler drier at 130° F. for 30 minutes. The shrinkage loss is computed by dividing the difference in length before and after washing by the length before washing and multiplying this fraction by 100.

$$\frac{L_0-L_W}{L_0}\times 100=\text{length shrinkage (per cent)}$$

wherein L_0 is length prior to washing and L_W is length after washing.

Repeated tests on a considerable number of woolen sweaters, finished with cotton seams, showed conclusively that maximum resistance to shrinkage with minimum chemical damage to the wool and particularly to the cotton seams is obtainable by the dry chlorination process only if the residual chloride content of the treated goods is controlled by adjusting the moisture content of the goods prior to introduction into the chlorination chamber within the ranges disclosed in this specification. If this precaution is taken, and if the goods are subsequently neutralized as herein described, the surprising result is obtained that garments are not only not weakened, but their seam breaking strength is actually increased. Thus, in one lot of sweaters, the seam breaking strength of the untreated sweaters was measured to be 38 pounds, while the seam breaking strength of the treated sweater rose to 46

pounds. In another sweater lot, seam breakage strength of an untreated sweater was 51 pounds, and 53 pounds in the treated sweater.

Sweaters and wool garments treated in accordance with the present invention were found in field tests to remain serviceable after 20 field army mobile launderings, while untreated sweaters shrank beyond serviceability after the third laundering. The appearance and comfort of the garments are equal or superior to untreated ones.

Dry chlorination in accordance with the present invention is not only applicable to wool sweaters, but to other woolen garments as, for instance, anklets, mufflers, underwear, jerseys, and shirts. Part wool "mixed" fabric garments (e. g. part cotton, part wool) are likewise rendered shrink resistant by dry chlorination in accordance with our invention.

While the foregoing examples illustrate specific applications of the principle of our invention, they are not to be taken as a limitation of the scope of our invention to the particular methods set forth therein. Modifications and additional examples will be readily apparent to the expert as the result of our disclosure. We thus intend to claim our invention broadly and to be limited only by the appended claims.

We claim:

1. The process of imparting shrink-resistance to woolen goods without substantially impairing their strength, comprising adjusting the moisture content of said goods to from about 10.5% to about 12%, contacting said goods in a closed chamber at a temperature above 70° F. with from about 1½ to 3% chlorine gas based on the weight of said goods, and neutralizing the hydrochloric acid formed on said goods.

2. The process of imparting shrink-resistance to woolen goods without substantially impairing their strength, comprising adjusting the moisture content of said goods to from about 10.5% to about 12%, contacting said goods in a closed chamber at reduced pressure with from about 1½ to 3% chlorine gas based on the weight of said goods, and neutralizing the hydrochloric acid formed on said goods.

3. The process of imparting shrink-resistance to woolen goods without substantially impairing their strength, comprising adjusting the moisture content of said goods to from about 10.5% to about 12%, contacting said goods in the moistened state in a closed chamber at a temperature above 70° F. with from about 1½ to 3% chlorine gas based on the weight of said goods until the residual chloride content of said goods is from about 1.2% to about 1.7%, and neutralizing the hydrochloric acid formed on said goods.

4. The process of imparting shrink-resistance to woolen goods without substantially impairing their strength, comprising adjusting the moisture content of said goods to from about 10.5% to about 12%, contacting said garments in a closed chamber with from about 1½ to 3% chlorine gas based on the weight of said goods until the residual chloride content of said goods is from about 1.2% to about 1.7%, and neutralizing the hydrochloric acid formed on said goods.

5. The process of imparting shrink-resistance to woolen goods without substantially impairing their strength, comprising adjusting the surface pH value of said goods to between about 3 and about 8, adjusting the moisture content of said goods to from about 10.5 to about 12%, contacting said garments in a closed chamber at reduced pressure above 70° F. with from about 1½ to 3% chlorine gas based on the weight of said goods until the residual chloride content of said goods is from about 1.2% to about 1.7%, and neutralizing the hydrochloric acid formed on said goods.

6. The process of imparting shrink-resistance to woolen goods without substantially impairing their strength, comprising adjusting the moisture content of said goods to about 11%, contacting said goods in a closed chamber with from about 1½ to 3% chlorine gas based on the weight of said goods, and neutralizing the hydrochloric acid formed on said goods.

7. The process of imparting shrink-resistance to woolen goods without substantially impairing their strength, comprising adjusting the moisture content of said goods to about 11%, contacting said goods in the moistened state in a closed chamber with from about 1½ to 3% chlorine gas based on the weight of said goods until the residual chloride content of said goods is about 1.6%, and neutralizing the hydrochloric acid formed on said goods.

8. The process of imparting shrink-resistance to woolen goods without substantially impairing their strength, comprising adjusting the moisture content of said goods to from about 10.5% to about 12%, contacting said goods in a closed chamber with from about 1½ to 3% chlorine gas based on the weight of said goods and neutralizing the hydrochloric acid formed on said goods.

9. The process of imparting shrink-resistance to woolen goods without substantially impairing their strength, comprising adjusting the moisture content of said goods to from about 10.5% to about 12%, contacting said goods in a closed chamber with about 2% chlorine gas based on the weight of said goods, and neutralizing the hydrochloric acid formed on said goods.

10. The process of imparting shrink-resistance to a woolen body garment without substantially impairing its strength, comprising adjusting the moisture content of said garment to from about 10.5% to about 12%, placing said moistened garment on a textile-covered hanger, contacting said garment in the moist state while on said hanger in a closed chamber with from about 1½ to 3% chlorine gas based on the weight of the wool until the residual chloride content of said garment is from about 1.2% to about 1.7%, and neutralizing the hydrochloric acid formed on said garment.

11. The process of imparting shrink-resistance to a woolen body garment without substantially impairing its strength, comprising adjusting the moisture content of said garment to from about 10.5% to about 12%, placing said moistened garment on a textile-covered hanger, contacting said garment in the moist state while on said hanger in a closed chamber at a temperature above 70° F. with from about 1½ to 3% chlorine gas based on the weight of the wool, and neutralizing the hydrochloric acid formed on said garment; whereby condensation of acid at the line of contact between said garment and said hanger and local damage to the garment in the area adjacent said line of contact are avoided.

12. The process of imparting shrink-resistance to a woolen body garment without substantially impairing its strength, comprising adjusting the moisture content of said garment to from about 10.5% to about 12%, placing said moistened garment on a textile-covered hanger, contacting said garment while on said hanger with from about 1½ to 3% chlorine gas based on the weight of the

wool in a closed chamber, and neutralizing acid formed on said garment.

13. The process of imparting shrink-resistance to a woolen body garment without substantially impairing its strength, comprising adjusting the moisture content of said garment to from about 10.5% to about 12%, placing said moistened garment on a wool-covered hanger, contacting said garment in the moist state while on said hanger in a closed chamber, with from about 1½ to 3% chlorine gas based on the weight of the wool in said garment, and neutralizing acid formed on said garment; whereby condensation of acid at the line of contact between said garment and said hanger and local damage to the garment in the area adjacent said line of contact are avoided.

14. The process of imparting shrink-resistance to a woolen body garment without substantially impairing its strength, comprising adjusting the moisture content of said garment to from about 10.5% to about 12%, placing said moistened garment on a nylon-covered hanger, contacting said garment in the moist state while on said hanger in a closed chamber, with from about 1½ to 3% chlorine gas based on the weight of the wool, and neutralizing acid formed on said garment; whereby condensation of acid at the line of contact between said garment and said hanger and local damage to the garment in the area adjacent said line of contact are avoided.

15. The process of imparting shrink-resistance to a woolen body garment without substantially impairing its strength, comprising adjusting the moisture content of said garment to from about 10.5% to about 12%, placing said moistened garment on a glass-fiber-covered hanger, contacting said garment in the moist state while on said hanger in a closed chamber, with from about 1½ to 3% chlorine gas based on the weight of the wool, and neutralizing acid formed on said garment; whereby condensation of acid at the line of contact between said garment and said hanger and local damage to the garment in the area adjacent said line of contact are avoided.

16. The process of imparting shrink-resistance to a woolen body garment without substantially impairing its strength, comprising adjusting the moisture content of said garment to from about 10.5% to about 12%, placing said moistened garment on a hanger having a hygroscopic non-condensing low heat capacity surface, contacting said garment in the moist state while on said hanger in a closed chamber, with from 1½ to 3% chlorine gas based on the weight of the wool, and neutralizing acid formed on said garment; whereby condensation of acid at the line of contact between said garment and said hanger and local damage to the garment in the area adjacent said line of contact are avoided.

17. The process of imparting shrink-resistance to woolen garments without substantially impairing their strength, comprising adjusting the moisture content of said garments to from about 10.5% to about 12%, placing each of a plurality of said woolen garments on an individual textile-covered hanger, introducing said garments in the moistened state and while on hangers into a closed chamber in spaced relation from each other, circulating from about 1½ to 3% chlorine gas based on the weight of the wool through said chamber and freely around said garments, removing said garments from said chamber, and neutralizing acid formed on said garments; whereby said woolen garments are uniformly chlorinated and whereby condensation of acid at the line of contact between said garments and said hangers and local damage to the garments in the area adjacent said line of contact are avoided.

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