

## UNITED STATES PATENT OFFICE

2,416,447

## WEATHER RESISTANT FLAMEPROOF PAPER

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No Drawing. Application July 27, 1943,  
Serial No. 496,344

1 Claim. (Cl. 92-3)

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This invention relates to flameproof paper and more specifically to weather resistant flameproof paper made flameproof by the addition of antimony oxides and chlorine-containing organic compounds.

For the purpose of this specification and the subjoined claims, a flameproof paper is defined as one which, if tested in a draft-free room by putting in contact with an initiating flame, will not continue to flame of its own accord for more than 5 seconds after removal of said initiating flame, and will not sustain combustion by afterglow beyond the charred area upon removal of said initiating flame. The term "paper" is to be taken in a generic sense, to include all articles fashioned from cellulosic pulp, such as light-weight sheets, the heavier-weight paper boards, specifically processed papers such as parchment, and articles molded from paper pulp.

This invention has as an object the production of a flameproof paper, the flameproof qualities of which are weather resistant in the sense that they are not materially impaired by prolonged exposure to weather.

It is a further object of this invention to achieve flameproof qualities in paper by means which do not appreciably alter the important physical characteristics of tensile and bursting strength, flexibility, thickness, porosity, bulk, or even absorbency.

A still further object of this invention is to provide processes by which such paper may be manufactured in a relatively simple manner on a large scale by the use of conventional paper mill equipment. Other and further important objects of this invention will appear as the description proceeds.

These objects are accomplished by a novel use of antimony trioxide and chlorine-containing organic compounds in the paper, as more fully set forth hereinbelow.

To accomplish flameproofing by means of metallic oxides is not new. It is old to coat paper and textiles with heavy metal oxides such as oxides of tin, lead, antimony, arsenic, bismuth, titanium and the like, and to decrease their combustibility thereby. It is also old to impregnate, coat, saturate, or otherwise surface-treat textile fabrics with heavy metal oxides and water-insoluble, chlorine-containing organic compounds having flameproofing qualities. The latter are generally applied from an organic solvent, which sometimes also acts as a vehicle for the metallic oxide. Under these conditions the chlorine-con-  
taining organic compound is deposited in a con-

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tinuous film on the surface of the fabric, and since said chlorine-containing organic compounds generally possess also waterproofing qualities, the fabric is thereby rendered impervious and water-  
proof. It is also frequently necessary to include  
auxiliary compounds which play no part in the  
actual flameproofing process but which are em-  
ployed as binders to facilitate the adherence of  
the insoluble oxide and thereby prevent its re-  
moval thru abrasion; other compounds are often  
included as plasticizers in an effort to overcome  
the impairment of flexibility.

As a consequence of these surface applications, which, for effective flameproofing involve the use of substantial quantities of opaque and insoluble materials, the physical character of the fabric is altered. This would be particularly true in the case of paper, which as a consequence of the coating or impregnation process would be increased in bulk or thickness, altered in color, flexibility and porosity, and so changed in general character as to become frequently unserviceable for many uses.

Furthermore, whereas the application of chlorinated organic compounds to fabric may be economically achieved and is in fact generally achieved by the aid of organic solvents, the use of organic solvents in the case of paper is ruled out by the sheer factor of economy, considering both the low price of a paper sheet compared to fabric and its bulk or surface area compared to a fabric of equal weight.

In other instances, in the case of textile fabric, the metallic oxide is deposited within the fiber by a mordanting process which involves first impregnating the fabric with a solution of a metallic salt, and then treating the same with a solution of another compound which will cause precipitation of a metallic oxide in the fiber. Such a double-treatment process with aqueous solutions could not universally be applied to paper, for if the latter is not absorbent it will not be uniformly impregnated by the aqueous solutions, and if it is absorbent it will generally be disintegrated by the dual impregnation.

Now, according to our present invention, the primary goal of producing a weather resistant, flameproof paper is achieved by methods which obviate many of the serious disadvantages of surface application processes, particularly those which result in alteration of the physical properties of the flameproofed article. We have found that if the antimony trioxide be mixed with the paper fibers before they are formed into a sheet, then quantities of it as high as 20% by weight of

the finished sheet may be incorporated into the paper without seriously affecting its essential physical qualities. The product differs only inappreciably from paper not containing antimony oxide as regards its general physical properties of flexibility, absorbency, thickness, porosity, etc., except for a minor decrease in strength and a slight lightening of color. Furthermore, according to our invention, we further incorporate into the paper a chlorine-containing organic compound which co-acts in some way with the antimony trioxide to produce a flameproof effect of a degree not otherwise obtainable by using either agent alone even if used in a quantity corresponding to the combined weight of the two agents. This chlorine-containing organic compound may likewise be added to the pulp in the form of powder or an aqueous suspension, or the treatment with the chlorine-containing organic compound may be postponed until after the sheet is formed in which event the sheet is impregnated with an aqueous emulsion or suspension of the chosen organic compound.

The physical properties of the paper are not seriously affected in either event, inasmuch as by the said joint action of the chlorine-containing agent and antimony trioxide, the quantity of either one may be reduced below the point where it might adversely change the quality and appearance of the resultant paper. Moreover, wet strength materials, color, and all other ingredients or paper-making auxiliaries customary in the fabrication of paper, paper specialties and the like may be included in the furnish and the paper may be formed on conventional equipment.

The flameproof effect on paper achieved by the processes of this invention is particularly resistant to weathering in that prolonged leaching in running water or prolonged outdoor exposure does not materially decrease the flameproof quality. However, this ability to withstand weathering is a characteristic of the flame proof quality, and is not dependent upon any waterproofing qualities which the chlorine-containing organic compound may bestow upon the paper itself. In fact a highly bibulous flameproof sheet may be fabricated by the processes of this invention which will undergo prolonged leaching without loss of flame resistance. Although the chlorine-containing compound may be water-insoluble and even possess waterproof properties when applied as a continuous film, the discontinuous nature of its distribution in the paper when applied as a powder or from aqueous emulsion as described above, does not generally interfere with the natural absorptive qualities of the cellulose fiber. This discontinuous but uniform distribution of the chlorine-containing organic compound differs markedly from the type of continuous film resulting from a solvent application, with its attendant changes in physical properties of the paper.

Paper prepared according to this invention may be subjected to the customary after-treatments to further modify its physical properties, as for instance by parchmentizing, printing, embossing, or creping.

Without limiting our invention to any particular procedure, the following examples are given to illustrate our preferred mode of operation. Parts mentioned are by weight.

In these examples, "basis-weight" shall be understood as referring to the weight of a ream consisting of 480 sheets of 24" x 36" each,

#### Example 1.—Application in the beater

In this method of application the ingredients for flameproofing may be mixed with the paper pulp any time prior to sheet formation. The color and antimony trioxide, for example, are added to the beater with or without size followed by alum. The chlorine-containing organic compound may be added to the beater or continuously to the stock at any convenient place ahead of the paper machine, such as at the head box. If it is desired that the paper shall have exceptional wet strength, either of two methods well known to the paper trade may be used for this purpose: (a) parchmentizing or (b) the use of a wet strength resin such as the condensation product of melamine and formaldehyde.

In the following example typical of sized, unparchmentized paper, olive drab in color, the melamine-formaldehyde resin was added at the head box:

|                                                                   | Pounds |
|-------------------------------------------------------------------|--------|
| Unbleached kraft—400 freeness (Green) pulp                        | 1,000  |
| 91.5% pure antimony trioxide                                      | 200    |
| Chrome Yellow (C. I. No. 1270)                                    | 86.5   |
| Metallized (copper) p-nitrophenyl-azobeta-naphthol                | 95     |
| Dispersed copper phthalocyanine                                   | 28.7   |
| Soluble Prussian Blue (C. I. No. 1288)                            | 1      |
| (Solid basis) polyvinylidene chloride, (25% emulsion) at fan pump | 150    |
| Size                                                              | 20     |
| Alum                                                              | 20     |
| Melamine-formaldehyde resin                                       | 50     |

When formed into a sheet approximately 60 lb. basis-weight this formula produces a flameproof sheet which retains its flameproof quality to a high degree even after continuous leaching in running water for several days.

Not all of the antimony trioxide and polyvinylidene chloride, however, are retained in the sheet. Part of these agents are lost in the white water until equilibrium is established. Analysis showed that approximately 9 to 11% of the total weight of the sheet was antimony trioxide and about 12 to 14% polyvinylidene chloride.

In the above process, it is not necessary that the chlorine-containing resin be in emulsion form. The finely powdered product may be added directly to the beater as in the following example using polyvinyl chloride-diethyl fumarate interpolymers.

#### Example 2.—Adding chlorinated resin in powdered form

This example deals with the production of unsized, absorptive paper.

|                                                                       | Pounds |
|-----------------------------------------------------------------------|--------|
| Unbleached kraft—400 freeness pulp                                    | 1,000  |
| Antimony trioxide                                                     | 200    |
| Chrome Yellow (C. I. No. 1270)                                        | 103    |
| Metallized (copper) p-nitrophenyl-azobeta-naphthol                    | 47     |
| Dispersed copper phthalocyanine                                       | 29     |
| Powdered (200 mesh) polyvinyl chloride-diethyl fumarate interpolymers | 250    |
| Alum                                                                  | 25     |

The sheet is formed in the same manner as above, and possesses weather resistant flameproof qualities. It possesses the essential feel, absorptiveness and other physical qualities of ordinary

waterleaf, and may be used for preparing parchment paper as in Example 5 hereinbelow.

**Example 3.—Beater application of antimony trioxide and subsequent impregnation with chlorinated resin**

In this process paper containing beater-applied antimony trioxide and color is impregnated subsequent to sheet formation with an aqueous emulsion of the chlorine-containing resin. This process is applicable to both sized sheets and waterleaf for parchmentizing. The following is a typical formula for sized, dark-green, high-wet-strength paper.

|                                                    | Pounds |
|----------------------------------------------------|--------|
| Unbleached kraft—400 freeness (Green) pulp         | 1,000  |
| 91.5% pure antimony trioxide                       | 200    |
| Chrome Yellow (C. I. No. 1270)                     | 112    |
| Metallized (copper) p-nitrophenyl-azobeta-naphthol | 30     |
| Dispersed copper phthalocyanine                    | 24     |
| Rosin size                                         | 20     |
| Alum                                               | 25     |
| Melamine-formaldehyde resin                        | 15     |

After forming into a sheet this paper was impregnated at the size press, while still approximately 30% wet, with an aqueous emulsion of polyvinylidene chloride so that it retained, upon drying, 8 to 12% polyvinylidene chloride (dry weight).

In this process if a heavy weight sheet is formed, the chlorinated compound may be applied at the calender stacks, for example, as follows:

**Example 4.—Heavy sheet**

A sheet of 90 lb. basis-weight, formed from the following typical formula:

|                                                    | Pounds |
|----------------------------------------------------|--------|
| Unbleached kraft                                   | 1,000  |
| 91.5% pure antimony trioxide                       | 200    |
| Chrome yellow (C. I. No. 1270)                     | 112    |
| Metallized (copper) p-nitrophenyl-azobeta-naphthol | 30     |
| Dispersed copper phthalocyanine                    | 24     |
| Size                                               | 25     |
| Alum                                               | 25     |

was surface treated at the calender stacks to pick up 5 to 8% polyvinylidene chloride applied from an aqueous emulsion.

This invention is also adaptable to the production of parchmentized paper. The following is a formula for a water-leaf sheet to be parchmentized:

**Example 5.—Parchment paper**

|                                                    | Pounds |
|----------------------------------------------------|--------|
| Unbleached kraft—400 freeness                      | 1,000  |
| 91.5% pure antimony trioxide                       | 200    |
| Chrome yellow (C. I. No. 1270)                     | 48.5   |
| Metallized (copper) p-nitrophenyl-azobeta-naphthol | 14.3   |
| Dispersed copper phthalocyanine                    | 15.5   |
| Alum                                               | 40     |

This formula is formed into a 30 lb. basis-weight bibulous sheet and treated at the size press with an aqueous emulsion of the interpolymers of vinylidene chloride and up to 15% vinyl chloride so that approximately 10% is retained by the paper. The sheet may now be parchmentized by treatment with sulfuric acid in the usual manner. The resulting paper is characterized by retaining a high degree of flameproofness

even after exposure to weather for several months.

**Example 6.—Twisting paper**

A light-weight sheet flameproofed in this manner may be used for twisting as in the following example:

|                                                    | Pounds |
|----------------------------------------------------|--------|
| Unbleached kraft—400 freeness                      | 1,000  |
| Antimony trioxide                                  | 200    |
| Chrome yellow (C. I. No. 1270)                     | 86.5   |
| Metallized (copper) p-nitrophenyl-azobeta-naphthol | 95     |
| Dispersed copper phthalocyanine                    | 28.75  |
| Prussian blue (C. I. No. 1288)                     | 1      |
| Alum                                               | 25     |
| Melamine-formaldehyde                              | 50     |

A 30 lb. basis-weight bibulous sheet is formed from this formula and treated at the size press with an emulsion of polyvinyl chloride-diethyl fumarate interpolymers (95:5) so that about 20% is retained by the paper. The paper is then twisted.

The coloring of paper is not an essential part of this invention, and an uncolored sheet may be made, for instance, as follows:

**Example 7.—Uncolored sheet**

|                                                                  | Pounds |
|------------------------------------------------------------------|--------|
| Unbleached sulfite pulp                                          | 1,000  |
| 91.5% pure antimony trioxide                                     | 250    |
| Chlorinated paraffin (containing 54% chlorine) as a 30% emulsion | 300    |
| Size                                                             | 20     |
| Alum                                                             | 30     |

The sheet is formed as in Example 1.

**Example 8.—0.016 natural Fourdrinier kraft board for conversion into corrugated boxes**

|                                                                             | Pounds |
|-----------------------------------------------------------------------------|--------|
| Unbleached kraft                                                            | 1,000  |
| Antimony oxide                                                              | 220    |
| Polyvinyl chloride-diethyl fumarate interpolymers from 19% aqueous emulsion | 150    |
| Rosin size                                                                  | 10     |
| Alum                                                                        | 40     |

The sheet is then formed on a Fourdrinier machine in the usual manner. This sheet may then be converted by customary procedure into corrugated board, and fashioned into boxes which exhibit the property of being flameproof even after exposure to weather.

**Example 9.—Yellow kraft lined newsboard 0.009**

|                                  | Pounds |
|----------------------------------|--------|
| Waste kraft clippings            | 1,000  |
| Antimony oxide                   | 220    |
| 6 oz. metanil yellow (C. I. 138) | 1      |
| Rosin size                       | 7      |
| Alum                             | 35     |

The sheet is formed on a cylinder machine and surface treated at the calender stacks with polyvinylidene chloride 32% emulsion to pick up 7%.

**Example 10.—Red 45 lb. basis-weight high-wet-strength absorbent paper**

|                                                     | Pounds |
|-----------------------------------------------------|--------|
| Semi-bleached kraft                                 | 1,000  |
| Antimony oxide                                      | 200    |
| Fast scarlet 4BS (C. I. 327)                        | 12     |
| Powdered polyvinyl chloride                         | 220    |
| Cellulose regenerated from viscose for wet strength | 100    |
| Alum                                                | 80     |

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The sheet is then formed on a Fourdrinier machine in usual manner.

**Example 11.—Blue unbleached sulfite light weight wrapping paper**

|                                                    | Pounds |
|----------------------------------------------------|--------|
| Unbleached sulfite.....                            | 1,000  |
| Antimony oxide.....                                | 250    |
| Methyl violet (C. I. 680).....                     | 1      |
| Victoria green (C. I. 657).....                    | 8      |
| Chlorinated polymyrcene from aqueous emulsion..... | 175    |
| Size.....                                          | 20     |
| Alum.....                                          | 35     |

**Example 12.—Article of moulded pulp**

|                                                               | Pounds |
|---------------------------------------------------------------|--------|
| Groundwood.....                                               | 1,000  |
| Antimony oxide.....                                           | 200    |
| Powdered polyvinyl acetate-trichlorethylene interpolymer..... | 250    |
| Copper phthalocyanine blue.....                               | 25     |
| Wax-methacrylate resin size.....                              | 90     |
| Alum.....                                                     | 65     |

The above furnish was dewatered in pulp molding equipment and dried. The molded article possessed flameproof qualities durable to weather.

**Example 13.—Kraft specialty sheet**

|                                            | Pounds |
|--------------------------------------------|--------|
| Unbleached kraft.....                      | 1,000  |
| Antimony oxide.....                        | 225    |
| Iron complex of nitroso beta naphthol..... | 45     |
| Rosin size.....                            | 15     |
| Alum.....                                  | 25     |

The sheet, after formation on a Fourdrinier machine, is saturated at the size press with an aqueous emulsion of an interpolymer of polyvinylidene chloride and polyvinyl chloride calculated to deposit 10% on the weight of the dry paper. After drying, the sheet is over-printed with a design to simulate foliage and is suitable for use in outside decorations, in view of its weather resistant flameproof qualities.

Although the omission of the organic solvent is one of the major economic advantages in our improved process, such solvents may nevertheless be used in the second stage of the process typified by Examples 3 and 4 above, i. e., after incorporation of the metallic oxide through the pulp stage. For instance, one could apply in the second stage a solution of chlorinated polymyrcene in carbon tetrachloride, a solution of chlorinated stearic acid in a 1:1 alcohol-water solution, a solution of a chlorine-containing vinyl-resin in toluene, and so on.

As will be seen from the foregoing examples, the nature of the auxiliary chlorine-containing organic compound employed and its amount may vary within wide limits. The quantity to be employed depends first of all on the amount of HCl liberated by the particular compound when heated to about the kindling temperature of paper, and secondly on the quantity of antimony oxide it is intended to replace. In other words, it varies somewhat inversely with the quantity of antimony oxide employed per unit weight of paper. It is also dependent to some extent on the thickness of the sheet to be produced, and is best determined experimentally for any particular set of conditions. The process of Example 3 above lends itself particularly to such evaluation, as illustrated by the following example applied to a waterleaf sheet:

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**Example 14**

|                                                         | Pounds |
|---------------------------------------------------------|--------|
| Unbleached kraft—400 freeness (Green) pulp.....         | 1,000  |
| Antimony trioxide—91.5% pure.....                       | 200    |
| Chrome Yellow (C. I. No. 1270).....                     | 48.5   |
| Metallized (copper) p-nitrophenyl-azobeta-naphthol..... | 14.3   |
| Dispersed copper phthalocyanine.....                    | 15.5   |
| Alum.....                                               | 40     |

A sheet of 45 lb. basis-weight was formed. Analyses showed the sheet to contain 10.2% antimony trioxide.

Small strips of this paper were treated in the laboratory with emulsion or organic solvent solutions of the chlorine-containing organic compound to determine the least amount necessary for flameproofing. Any resins which were not available in emulsion form were impregnated from a solution in a volatile solvent for purposes of evaluation. After drying, the gain in weight was noted. These strips were then tested for flameproofness by igniting a piece ¼ inch wide in the low blue flame of a Bunsen burner. Increasing quantities of the chlorine-containing agent to be tested were used on successive strips until a point was reached where the flame was immediately extinguished upon removing the strip from the burner. The following is a list of the compounds found feasible for flameproofing paper and the minimum percent (based on the weight of the treated paper) necessary to attain the special degree of flameproofness assumed above for this comparative study:

| Compound                                              | Total chlorine content, per cent | Per cent weight of HCl liberated at designated temperature |         |         | Minimum quantity needed in above example, by per cent weight of treated sheet |
|-------------------------------------------------------|----------------------------------|------------------------------------------------------------|---------|---------|-------------------------------------------------------------------------------|
|                                                       |                                  | 200° C.                                                    | 300° C. | 400° C. |                                                                               |
| Polyvinylidene chloride.                              | 72                               | 5.2                                                        | 45.7    | 52.9    | 12.3                                                                          |
| Polyvinyl chloride-diethyl fumarate interpolymer..... | 54                               | 4.0                                                        | 29.1    | 44.1    | 18.0                                                                          |
| Chlorinated polyvinylchloride.....                    | 65                               | 5.7                                                        | 33.3    | 53.3    | 17.0                                                                          |
| Polyvinyl chloride.....                               | 57                               | 2.7                                                        | 25.5    | 50.4    | 24.0                                                                          |
| Chlorinated polymyrcene.....                          | 64                               | 1.8                                                        | 30.8    | 46.1    | 17.0                                                                          |
| 52% chlorinated stearic acid.....                     | 52                               | 2.3                                                        | 32.6    | 37.8    | 31.0                                                                          |
| 54% chlorinated paraffin.....                         | 54                               | 0.2                                                        | 23.6    | 43.5    | 35.0                                                                          |
| 40% chlorinated paraffin.....                         | 40                               | 0.3                                                        | 19.0    | 33.7    | 43.0                                                                          |
| Chlorinated myrcene.....                              | 67.5                             | ---                                                        | 31.3    | ---     | 30.0                                                                          |
| 65% chlorinated rubber.....                           | 65                               | ---                                                        | ---     | ---     | 19.0                                                                          |

It will be noted that the minimum quantity of chlorine-containing organic compound per unit sheet weight to achieve a specified degree of flameproofness varies inversely with the percentage HCl liberated by this compound at the kindling temperature, and that the product of the two is approximately constant for certain groups of organic compounds. Thus, for polymerized resins, the product of the datum in the fifth column by that in the third column is between 5 and 6 (%). The same product for the unpolymerized compounds is about 8 to 9.5 (%). This product will be designated as the hydrogen-chloride equivalent of the given organic compound. If the Sb<sub>2</sub>O<sub>3</sub> content is at its highest (20%), the requisite hydrogen-chloride equivalent is 2% for polymeric resins and 3% for unpolymerized organic compounds.

To summarize all the above, the chlorinated organic compounds found most suitable in our invention are those which possess at least 40% of

combined chlorine and which are adapted to liberate at least 50% of this combined chlorine in the form of hydrogen chloride upon being heated alone for five minutes at a temperature between 300 and 400° C., which corresponds to the average kindling point of untreated paper. Organic compounds of lower chlorine content, say 25 to 30%, may also be used, provided larger quantities of the compound are employed. The quantity of any given agent is in general selected so as to give a hydrogen-chloride equivalent not less than a predetermined minimum for any particular type of paper.

Chemically, the more suitable chlorinated organic compounds may be classified as chlorinated higher aliphatic hydrocarbons (i. e., having more than 8 carbon atoms), chlorinated higher fatty acids, chlorine-containing polyvinyl-type resins (polymers, copolymers, and interpolymers), chlorinated rubber, etc.

In lieu of antimony trioxide, the pentoxide, tetroxide or any other oxide of antimony may be employed. The quantity thereof may vary from 5 to 20% by weight based on the weight of the final sheet.

The advantages and utility of this invention will now be readily apparent.

This invention may be used to provide flameproof paper for a large number of uses such as: camouflage, decorations, stage settings, draperies, packaging, insulation for electric equipment, seat covers, upholstery, rugs and many others. It may be used in molded pulp articles such as ash trays.

This invention is advantageous in that the flameproofing processes by which it may be achieved need not detract from the physical properties of the resulting paper to an extent inconsistent with good practice. The paper retains most of its strength, may be dyed, printed, parchmentized, or twisted in the same manner as untreated paper.

Furthermore the application of the flameproofing agents may be easily adapted to most types of paper or mill conditions. Thus (1) the antimony trioxide and chlorine-containing compound, either emulsion or powder, may be added to the paper pulp prior to sheet formation, or (2) the antimony trioxide may be added to the pulp prior to sheet formation and the chlorine-containing resin, emulsion or solution in solvent, may be applied to the sheet at any convenient point after sheet formation, at the size press or calender stacks for example.

Finally this invention is particularly advantageous in that the flameproof properties of this paper are not destroyed by weathering or exposure to water. In fact, in some cases, as for instance in the case of bibulous paper, the flameproof quality may be more durable to weather than the paper itself.

For the purpose of this application and the claims below, a flameproof effect on paper shall be considered as "weather resistant" or as "not materially impaired by prolonged exposure to weather" if a sample of the paper after being submitted to a continuous leaching test in running water at room temperature (70° F.) for 24 hours followed by drying, still exhibits flameproofness to an extent as defined in the introductory paragraphs of this specification.

We claim:

A flameproof parchment paper containing, uniformly distributed throughout its mass, from 5 to 20% of antimony oxide, based on the weight of the paper, and from 5 to 25% of a chlorine-containing, water-insoluble, organic compound of the group adapted to liberate hydrogen chloride at the kindling temperature of the paper, the said parchment paper being characterized by a flameproof quality which is weather-resistant.

EDWIN ROMIG LAUGHLIN.  
JOHN LECROY AYRES.  
PAUL JONES MITCHELL, JR.

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American Dyestuff Reporter, July 5, 1943, pages 297 to 301.

## Certificate of Correction

Patent No. 2,416,447.

February 25, 1947.

EDWIN ROMIG LAUGHLIN ET AL.

It is hereby certified that errors appear in the printed specification of the above numbered patent requiring correction as follows: Column 7, line 6, Example 11, beginning with the word "Pounds" strike out all to and including "Alum----- 35" in line 14, and insert instead the following—

|                                                    |             |       |
|----------------------------------------------------|-------------|-------|
| Unbleached sulfite-----                            | Pounds----- | 1,000 |
| Antimony oxide-----                                | do-----     | 250   |
| Methyl violet (C. I. 680)-----                     | do-----     | 1     |
| Victoria green (C. I. 657)-----                    | Ounces----- | 8     |
| Chlorinated polymyrcene from aqueous emulsion----- | Pounds----- | 175   |
| Size-----                                          | do-----     | 20    |
| Alum-----                                          | do-----     | 35    |

column 10, lines 1 and 2, for "advantages" read *advantageous*; and that the said Letters Patent should be read with these corrections therein that the same may conform to the record of the case in the Patent Office.

Signed and sealed this 27th day of May, A. D. 1947.

[SEAL]

LESLIE FRAZER,

*First Assistant Commissioner of Patents.*