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ART OF REDUCING INFLAMMABILITY OF MATERIALS.

No Drawing. Application filed January 15, 1921. Serial No. 437,618.

To all whom it may concern:

Be it known that I, ARTHUR ARENT, a citizen of the United States, residing at Des Moines, county of Polk, State of Iowa, have invented certain new and useful Improvements in the Art of Reducing Inflammability of Materials; and I do hereby declare the following to be a full, clear, and exact description of the invention, such as will enable others skilled in the art to which it appertains to make and use the same.

This invention relates to art of reducing inflammability of materials; and it comprises novel compositions of matter useful in treating combustible materials to render the latter less inflammable, processes of treating materials with such compositions, and the resultant treated materials. More particularly, the invention has to do with the treatment of cellulose plastics or plastic compositions to render the same either non-inflammable or less inflammable than normally, according as one or the other of these objects may be desired in any given instance. The invention being of especially great practical importance in the production of non-inflammable photographic films, such as motion picture films, the application of the invention for this purpose will be hereinafter described in detail for the sake of a concrete example illustrating the broad principles involved.

The high standards of transparency or translucency, as well as of other light-transmitting characteristics, which it is essential to attain and to maintain in motion picture film to enable satisfactory projection of images carried thereby, render difficult the problem of so altering the composition of the film, either superficially or throughout, in such manner as to produce the desired fireproofing effect, without at the same time undesirably affecting the character of the film for projection purposes. It is also important that the non-inflammability and other desirable properties of a film treated with a fire-retardant or fireproofing agent shall be permanent, and substantially unaffected by washing or other conditions to which the film may be subjected in ordinary use. In particular therefore, it is important that no material clouding or undesirable discoloration of the film shall result from use of the fire retardant, and also that it shall resist the dissolving action of water or other form of moisture. The use of fire-retardants which are hygroscopic or deliquescent under the conditions in which they are present in or on the film is also precluded in practice. Moreover fireproofing treatment must not be such as to render the film brittle or otherwise adversely affect its flexibility, suppleness, or strength. The foregoing are among the many requirements complicating the problem of fireproofing motion picture film.

According to the present invention these and other practical requirements are successfully met in a non-inflammable film by treating the film substance with a fire-retardant metal compound of the antimony group in such manner and under such conditions that, after incorporation with the film substance either superficially or otherwise, the fire-retardant is not only insoluble in water but is also in such form or condition as not to be decomposable by water with formation of products that substantially cloud the film or undesirably alter its color. It is most desirable to employ for this purpose a treating agent consisting at least in part of a salt of antimony dissolved in a solvent which is also a solvent for the film substance or base. In practice such film substance or base consists largely of nitrocellulose (celluloid, pyroxylin), cellulose acetate, or other cellulose ester or esters, with which are usually admixed other materials suitable and desirable for production of a satisfactory film as is well understood in the art. Within the broad scope of the invention, any suitable method of associating the fire-retardant with the film substance may be employed; but in practice I find the objects in view can be accomplished effectively and expeditiously by applying the solution of the fire-retardant to the finished film strip after the photographic images to be projected have been placed thereon. In order to do this successfully without damaging the film, it is necessary to properly correlate strength of treating solution with the duration and other conditions of treatment in such manner as to ensure carrying the fire-retardant into the body of the film to the desired extent, while avoiding such dissolution of the film substance as would endanger the integrity of the film and of the pictures carried thereon. I prefer therefore to employ a co-solvent for the fire-retardant

and film substance which has a relatively mild solvent action on the film substance, and also to limit the duration of the treatment to a comparatively brief period. In the treating solution it is often desirable to incorporate, in addition to the fire retardant, one or more additional constituents capable of advantageously affecting its flexibility, toughness, or other properties, or which aids in facilitating the handling of the film during treatment by preventing so-called "rocking" or curling-up of the film while it is wet with the treating bath.

In a typical procedure for treating in accordance with my invention a motion picture film consisting of a celluloid or nitrocellulose base, a solution of an antimony salt in acetic ether is employed. Acetic ether, consisting principally of ethyl acetate accompanied by small allowable percentages of alcohol and water (U. S. Pharmacopeia—8th Edn.), is an excellent solvent for antimony salts such as antimony trichlorid, and at the same time it has a comparatively mild and controllable solvent action on cellulose nitrate. A treating solution of the character described can be variously prepared. One way is to dissolve anhydrous antimony trichlorid ($SbCl_3$) in acetic ether, in the proportion of about 10 grams of antimony trichlorid in about 30 cc. of acetic ether; then adding an excess of metallic mercury and shaking, finally separating from the solution any mercurous chlorid precipitated, together with the residual excess of mercury, by decanting the supernatant solution or by filtering. While the described method of preparing the treating solution is found to be advantageous in practice, probably because it tends to eliminate or to reduce the amount of any free hydrochloric acid that may be present as a result of using antimony trichlorid, and possibly also because the latter may be converted to a greater or less extent into an organic acid salt (the acetate in this instance), nevertheless it is not to be inferred that the solution may not be otherwise prepared, as for example by dissolving antimony acetate or other organic acid salt of antimony directly in the acetic ether or other substantially non-aqueous solvent employed.

When preparing the treating solution from antimony trichlorid in the manner above described in detail, I have found it desirable in some cases first to shake the solution of antimony trichlorid in acetic ether with an excess of sodium acetate, and then to shake with metallic mercury, the solution being allowed to stand several hours after each shaking. Employment of an acetic ether solution of antimony trichlorid without further treatment is apt to render the treated film brittle.

The use of a small portion of glacial acetic acid or other solvent having relatively slow solvent action on the film, in conjunction with the acetic ether, is sometimes advantageous especially in aiding to prevent "rocking" of the film when it is being treated with the solution. For example, I may employ a mixture of 1 part by volume of a solution of 10 grams of antimony trichlorid in 30 cc. glacial acetic acid with 5 parts of the acetic ether solution of the antimony salt already described. It is also feasible to effect the film treatment wholly with the glacial acetic acid solution, although this is ordinarily a slower procedure. Castor oil is a desirable optional addition to the treating mixture because it has a good effect in cases where it is necessary to take precautions to maintain or increase the flexibility and suppleness of the film. The solution may advantageously include a cellulose ester, such as the nitrate or acetate, where it is desirable to produce a varnishing effect on the film in addition to reducing its inflammability by penetration of the solution into the film body.

The treating solution prepared as above described is applied to the picture film in any convenient way that will enable reasonable control of the operation to ensure effectiveness and substantial uniformity of treatment. For example the film may be fed longitudinally by suitable rollers and sprockets at a controllable speed through a bath of the treating solution and then through a suitable drier for superficial removal of the volatile solvent, the complete drying of the film being thereafter accomplished at a slow rate. Provision may of course be made for recovery of the volatile solvent or solvents. The period of time that the film remains in contact with the bath may be brief, say from 10 seconds to a minute using a solution such as that above described, the penetration of the solvent carrying the fire retardant into the film body being sufficient in the example given to render the treated film, after complete drying, so resistant to fire that it does not inflame even when held vertically and a flame is applied to its lower edge. Under these conditions the film merely chars but does not burn alone. A cellit (cellulose acetate) film, though naturally less inflammable than a cellulose nitrate film, also has its inflammability greatly lessened when treated in this way.

The treated film is not adversely affected by the treatment described, the projection effects obtainable being fully equal to those obtainable with the untreated film; and in addition, there is no danger of the film flaming if exposed while stationary even for prolonged periods to the heat of the projecting arc of motion picture apparatus. Moreover the film can be washed with water

without causing clouding of the film or affecting its non-inflammability. In this connection it may be noted that a motion picture is capable of absorbing a very appreciable amount of moisture. The fact that prolonged immersion of the treated film in water produces no perceptible clouding, whereas addition of water to the treating solution produces a milky precipitate of a basic antimony salt, seems to indicate that the described method of treatment effects something in the nature of a chemical union between the antimony salt and the cellulose ester of the film. The invention is not predicated upon this theory, however, but rather upon the observed facts above mentioned.

It is evident that the details of procedure may vary considerably within the scope of the invention from those above given for purposes of explanation. For example, other co-solvents, such as amyl acetate, acetone, etc., may be employed; but if amyl acetate or acetone is used, it is best to dilute it considerably with a less active solvent for the cellulose ester of the film, or with a solvent substantially inert toward such ester. While both sides of the film may be subjected to the action of the treating solution, I find it sufficient and often advantageous in practice to treat only the smooth side, or the side opposite to that carrying the photographic emulsion. A film thus treated is also non-inflammable, and certain practical advantages are sometimes gained by confining the treatment to one side. Where the emulsion side of the film is also treated, it becomes covered with a thin protective coating of the film substance dissolved from the film to a slight extent by the treating solution. This desirable effect is still more pronounced where the treating solution includes an added cellulose ester as mentioned hereinbefore. This is advantageous in that it permits the film surface to be thoroughly cleaned from time to time, which is necessary in actual use, without so much danger of damaging the pictures thereon.

I believe it to be broadly new in the art to apply a fire-retardant or fireproofing agent to a finished motion picture film bearing photographic images for projection, irrespective of the specific fire-retardant employed.

What I claim is:

1. As a new compositon of matter, a solution comprising a salt of an antimony group metal decomposable by water dissolved in a solvent comprising ethyl acetate.

2. As a new composition of matter, a solution comprising an antimony salt dissolved in a solvent comprising ethyl acetate.

3. As a new composition of matter, a solution comprising acetate of antimony dissolved in a solvent comprising ethyl acetate.

4. As a new composition of matter, a solution comprising an organic acid salt of an antimony group metal dissolved in a solvent comprising an alkyl ester of an organic acid.

5. As a new composition of matter, a solution comprising a water-insoluble organic acid salt of antimony dissolved in a non-aqueous solvent.

6. As a new composition of matter, a solution comprising a water-insoluble organic acid salt of an antimony group metal dissolved in a non-aqueous solvent for cellulose esters.

7. As a new composition of matter, a solution comprising an anhydrous antimony trichlorid dissolved in a mixture of solvents for a cellulose ester, said solvents differing in their solvent action on such ester.

8. As a new composition of matter, a solution comprising anhydrous antimony trichlorid dissolved in a mixture of solvents for nitrocellulose, one of said solvents having a comparatively slow solvent action on nitrocellulose.

9. As a new composition of matter, a solution comprising a salt of antimony dissolved in a mixture comprising ethyl acetate and glacial acetic acid.

10. The process of preparing a non-aqueous solution of an antimony compound which comprises dissolving a mineral acid salt of antimony in a non-aqueous solvent comprising an organic acid ester, and converting said mineral acid salt of antimony at least partially into the corresponding organic acid salt.

11. The process of preparing a solution useful in fireproofing which comprises dissolving antimony trichlorid in an ester of acetic acid, and treating the solution with an excess of metallic mercury.

12. The process of preparing a solution useful in fireproofing which comprises dissolving antimony trichlorid in an ester of acetic acid, and treating the solution successively with an alkali metal acetate and metallic mercury.

13. The process of reducing the inflammability of a cellulose ester composition which comprises treating such composition with a solution of a fire-retardant compound of an antimony group metal in a solvent comprising an organic acid ester.

14. The process of reducing the inflammability of material which comprises treating such material with a solution of an antimony group metal compound in a solvent comprising ethyl acetate.

15. The process of reducing the inflammability of material which comprises treating such material with a solution of an antimony salt in a solvent comprising ethyl acetate.

16. The process of reducing the inflammability of a cellulose ester film which com-

prises treating such film with a solution of an antimony salt in a solvent having a relatively mild solvent action on said film.

17. The process of reducing the inflammability of a cellulose ester film which comprises treating such film with a solution of an antimony salt in a mixture of solvents for the film material, one of said solvents having a comparatively slow solvent action and serving to moderate the solvent action of the mixture.

18. The process of reducing the inflammability of a cellulose ester film which comprises treating such film with a solution of an antimony salt in a solvent comprising ethyl acetate.

19. The process of reducing the inflammability of a cellulose ester film which comprises treating such film with a solution of an antimony salt in a solvent comprising ethyl acetate and glacial acetic acid.

20. The process of rendering non-inflammable a motion picture film bearing photographic images which comprises subjecting such film to the action of a solution of a fire retardant.

21. The process of rendering non-inflammable a motion picture film bearing photographic images which comprises subjecting such film to the action of a solution of a fire retardant metal compound in a solvent having the power to penetrate into said film.

22. The process of rendering non-inflammable a motion picture film bearing photographic images which comprises subjecting such film to the action of a solution of a water-insoluble fire retardant dissolved in a solvent having a solvent action on the material of said film, discontinuing such treatment before extensive solution of said film has occurred, and drying the treated film.

23. The process of rendering a cellulose ester motion picture film non-inflammable which comprises treating the same with a solution comprising antimony acetate in a solvent comprising ethyl acetate.

24. The process of rendering non-inflammable a motion picture film carrying a photographic emulsion on one side, which comprises subjecting only the smooth or non-emulsion side of said film to the action of a solution of a fire retardant in a co-solvent for said fire retardant and the material of said film.

25. The process of rendering a motion picture film non-inflammable which comprises treating such film with a solution comprising a fire retardant and an oily softening agent in a solvent which is also a solvent for the material of said film.

26. As a new article of manufacture, material carrying a combined antimony group metal, at least partly as a water-insoluble organic acid salt, in sufficient quantity to render the assemblage non-inflammable.

27. As a new article of manufacture, a material carrying combined antimony, at least partly as antimony acetate, in sufficient quantity to render the assemblage non-inflammable.

28. As a new article of manufacture, a non-inflammable motion picture film comprising a base of a cellulose ester carrying a water-insoluble organic acid salt of an antimony group metal compound.

29. As a new article of manufacture, a motion picture film comprising a cellulose ester base with which is associated a sufficient proportion of an antimony salt to render the film non-inflammable, said antimony salt being present in a form in which it is substantially insoluble in and undecomposable by water.

In testimony whereof I hereunto affix my signature.

ARTHUR ARENT.