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[Second Edition.]

## PATENT SPECIFICATION



Application Date: April 21, 1926. No. 10,485 / 26.

279,139

Complete Left: Sept. 30, 1926.

Complete Accepted: Oct. 21, 1927.

PROVISIONAL SPECIFICATION.

### Improvements in and relating to Compositions Containing Cellulose Esters and Cellulose Ethers and the like.

We, HENRY JAMES HANDS, of No. 100, rivatives. as for instance, pentachlor-

## ERRATUM.

SPECIFICATION No. 279,139. (2nd Edition.)

Page 2, line 17, for "haptachlor" read "heptachlor"

THE PATENT OFFICE,

21st February, 1928.

(R2023) Wt 25832/531 125 3/28 H & Sp 6p 112

hereby declare the nature of this invention and in what manner the same is to be performed, to be particularly described and ascertained in and by the following statement:—

This invention relates to compositions containing ethers or esters of cellulose or like carbohydrates having the empirical formula  $n(C_6H_{10}O_5)$  and particularly to compositions containing the organic acid esters of cellulose, for example cellulose acetate, together with

to provide improved compositions of the kind specified in the form, for instance, of blocks, moulded articles, sheets, pellicles or films, and particularly films or film stock for photography and cinematography, artificial threads or filaments, such as the so-called artificial silk, coating or impregnating compositions, varnishes, enamels and paints for the decoration, ornamentation or other treatment of surfaces and insulating enamels, for the production of enamelled

[Price 1. -]

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PROVISIONAL SPECIFICATION.

Improvements in and relating to Compositions Containing Cellulose Esters and Cellulose Ethers and the like.

We, HENRY JAMES HANDS, of No. 100, Duke's Avenue, Chiswick, in the County of Middlesex, a British subject, and SPICERS LIMITED, of 19, New Bridge Street, London, E.C. 4, a company registered under the laws of Great Britain, do hereby declare the nature of this invention to be as follows :—

This invention relates to compositions containing cellulose esters and cellulose ethers and the like, and particularly to compositions containing organic esters of cellulose such, for example, as cellulose acetate.

According to the invention the chlorine derivatives of propane are employed in forming compositions containing cellulose esters or ethers. Such chlorine de-

rivatives, as for instance, pentachloropropane, hexachloropropane, and heptachloropropane, are used preferably in association with cellulose ester or ether solvents of relatively low boiling points, and also, if certain surface effects are required, with the higher aliphatic alcohols. Also, either alone or in admixture with the higher aliphatic alcohols, such chlorine derivatives may be employed in conjunction with the usual plasticising agents such as mono- or tri-glyceryl benzoate, triphenyl phosphate, tricresyl phosphate, resorecnyl diacetate, ethyl oleate, and cyclohexonal.

Dated the 21st day of April, 1926.

MARKS & CLERK.

COMPLETE SPECIFICATION.

Improvements in and relating to Compositions Containing Cellulose Esters and Cellulose Ethers and the like.

We, HENRY JAMES HANDS, of 100, Duke's Avenue, Chiswick, London, W. 4, a British subject, and SPICERS LIMITED, of 19, New Bridge Street, London, E.C. 4, a company registered under the laws of Great Britain, do hereby declare the nature of this invention and in what manner the same is to be performed, to be particularly described and ascertained in and by the following statement :—

This invention relates to compositions containing ethers or esters of cellulose or like carbohydrates having the empirical formula  $n(C_6H_{10}O_5)$  and particularly to compositions containing the organic acid esters of cellulose, for example cellulose acetate, together with

chlorine derivatives of hydrocarbons the molecules of which contain more than two carbon atoms optionally in association with known solvents and plasticising agents.

The object of the present invention is to provide improved compositions of the kind specified in the form, for instance, of blocks, moulded articles, sheets, pellicles or films, and particularly films or film stock for photography and cinematography, artificial threads or filaments, such as the so-called artificial silk, coating or impregnating compositions, varnishes, enamels and paints for the decoration, ornamentation or other treatment of surfaces and insulating enamels, for the production of enamelled

- wire and for impregnating electrical windings and the like, compositions for the production of leather cloth, enamelled leather and the like and for other purposes for which compositions containing cellulose esters and cellulose ethers may be suitable.
- The invention consists broadly in compositions of the character indicated in which are contained chlorine derivatives of propane having more than three chlorine atoms in their molecules.
- As examples of suitable chlorine derivatives of propane which may be used in accordance with the invention pentachlor-propane, hexachlor-propane, and heptachlor-propane may be mentioned.
- As will be understood there are a number of isomers of the substitution products of propane and where in the Specification or claims expressions are employed which indicate a chemical individual the expression is used as including isomeric bodies or mixtures of several of such isomeric bodies.
- Normally the chlorine derivatives of propane will be used in association with other solvents or softening agents or plasticisers for the carbohydrate ethers or esters.
- As examples of plasticising agents which may be used in association with the specified chlor-propanes in accordance with the invention the mono- or tri-glyceryl benzoate, tri-phenyl phosphate, tri-cresyl phosphate, resorcinyll diacetate, ethyl oleate and cyclo-hexanol may be mentioned. It will, however, be understood that a number of plasticising agents which have hitherto been used or proposed to be used in the production of cellulose ester and cellulose ether compositions may be employed in addition to or in substitution for the plasticising agents specifically mentioned.
- Further, in compositions in accordance with the invention the higher aliphatic alcohols may be included and as occasion may require volatile solvents for the carbohydrate esters or ethers.
- In addition to the chlorine derivatives of propane containing more than three chlorine atoms in their molecules, compositions in accordance with the invention may contain also the higher chlorine derivatives of ethane; for example, hexachlor-ethane.
- The chlorine derivatives of propane employed in accordance with the invention possess very high boiling points, for instance a boiling point of 249° C., and the physical character of these bodies increases their values as plasticising materials.
- The proportion of the specified chlorine derivatives of propane present in the composition may be varied within wide limits.
- In a varnish composition adapted particularly for use in the insulation of wires for electrical purposes in accordance with the invention and containing cellulose acetate heptachlor-propane or the lower chlorine derivatives of propane containing more than three chlorine atoms, such as hexachlor-propane, pentachlor-propane and the like, may be used jointly or severally in a proportion up to one-third or more by weight of the cellulose acetate in the composition and particularly if the higher chlor-ethanes are also contained in the composition and acetone is used as the volatile solvent.
- In the production of artificial silk also the proportion of chlorine derivatives of propane containing more than three chlorine atoms in their molecules may be varied within wide limits.
- Further, in the production of transparent sheets, pellicles or films the said chlorine derivatives of propane may be used in varying proportions and even when present in a relatively small proportion they are of value not only as plasticising agents but in reducing the inflammability of the compositions; for instance, they may be used in a cellulose acetate composition in a proportion of 2 to 3 per cent. by weight of the cellulose ester and in such case the composition may contain also a proportion of known plasticising agents which may amount to between 30 to 35 per cent. by weight of the cellulose acetate.
- In carrying the invention into effect compositions containing, for instance, ethers or organic acid esters of cellulose or like carbohydrate and chlorine derivatives of propane, for instance heptachlor-propane, alone or in admixture with hexachlor- and pentachlor-propane, are worked up by the mechanical methods known in connection with the production of cellulose ester or cellulose ether compositions. Thus, for instance, the compositions may be moulded into objects under pressure and heat, converted into solutions or otherwise treated.
- In the preparation of foil or thin sheets in accordance with the invention compositions comprising, for example, hydrolysed acetyl cellulose, one or more chlorine derivatives of propane containing more than three chlorine atoms in their molecules and a higher alcohol or higher alcohols with or without the addition of plasticising agents may be dissolved in a convenient volatile solvent with the formation of compositions adapted to be worked up into foil or thin

10 sheets in known manner, as for instance, by applying the compositions to spreading tables or by the employment of spreading machines or machines furnished with an endless support to which the composition is continuously applied and from which the foil or sheet is continuously stripped. As a volatile solvent for the purpose in question acetone is suitable.

15 Cellulose ester compositions containing heptachlor-propane or other chlorine derivatives of propane containing more than three chlorine atoms in their molecules are particularly suitable for use in the preparation of foil or thin sheets by a spreading or equivalent operation.

20 Compositions in accordance with the invention are of particular value in connection with photography in the production, for instance, of photographic and cinematographic films.

25 Having regard to the very large number of materials which may be used as principal or ancillary solvents or plasticising agents in the production of compositions from cellulose or like carbohydrate esters or ethers, it is not convenient or necessary to refer to all of the known bodies which may be used in accordance with the invention in association with the chlorine derivatives of propane having more than three chlorine atoms in their molecules, but it is to be understood that the invention extends to the use of these chlor-propanes in association with such known solvents, plasticising or gelatinising agents with which they are compatible in the production of compositions containing carbohydrate ethers or esters and more particularly the production of films or coatings from such compositions.

45 Having now particularly described and ascertained the nature of our said invention and in what manner the same is to be performed, we declare that what we claim is:—

50 1. Compositions containing ethers or esters of cellulose or like carbohydrates having the empirical formula  $n(C_6H_{10}O_5)$  and particularly compositions containing the organic acid esters of cellulose, for example cellulose acetate, in which chlorine derivatives of propane having more than three chlorine atoms in their molecules are present.

55 2. Compositions as claimed in Claim

60 1, in which pentachlor-propane, hexachlor-propane or heptachlor-propane or mixtures of any two or more of such chlorine derivatives are present.

65 3. Compositions as claimed in either of the preceding claims containing also other solvents or softening agents or plasticisers for the carbohydrate ethers or esters.

70 4. Compositions containing ethers or esters of cellulose or like carbohydrates having the empirical formula  $n(C_6H_{10}O_5)$  and particularly compositions containing organic acid esters of cellulose, for example cellulose acetate, substantially as hereinbefore described.

75 5. Articles in the form of blocks, moulded articles, sheets, pellicles or films, and particularly films or film stock for photography and cinematography, artificial threads or filaments, such as the so-called artificial silk, coating or impregnating compositions, varnishes, enamels and paints for the decoration, ornamentation or other treatment of surfaces and insulating enamels, for the production of enamelled wire and for impregnating electrical windings and the like, compositions for the production of leather cloth, enamelled leather and the like and for other purposes for which compositions containing cellulose esters and cellulose ethers may be suitable, formed from compositions as claimed in any of the preceding claims.

90 6. The method of producing compositions containing ethers or esters of cellulose or like carbohydrates having the empirical formula  $n(C_6H_{10}O_5)$  and particularly compositions containing the organic acid esters of cellulose, for example cellulose acetate, which comprises incorporating with such esters or ethers chlorine derivatives of propane containing more than three chlorine atoms in their molecules.

105 7. The improved method of producing compositions containing ethers or esters of cellulose or like carbohydrates having the empirical formula  $n(C_6H_{10}O_5)$  and particularly compositions containing the organic acid esters of cellulose, for example cellulose acetate, substantially as hereinbefore described.

Dated this 30th day of September, 1926.

MARKS & CLERK.