

## UNITED STATES PATENT OFFICE

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## SOLVENT FOR ACETYL CELLULOSE

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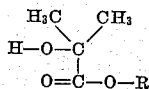
2 Claims. (Cl. 106—40)

Our present invention relates to acetyl cellulose, and more especially to solvents therefor. The main object of the invention is to provide a new solvent for acetyl cellulose resulting in important advantages both in the course of manufacture and in the resultant product.

To the above and other ends, which will hereinafter appear, the invention consists of the features hereinafter described and particularly pointed out in the claims.

The various compositions to which the present invention relates are employed in the arts as imitations of natural substances and in films used for photographic and therapeutic purposes. They consist of soluble acetyl cellulose combined with or dissolved in certain other substances or menstrua. Although the final or useful form of the different compounds of this class is that of a solid or dried material, the different processes of conversion into this final solid form involve, as is well understood, the employment of solutions or mixtures of varying condition as to plasticity, stiffness or fluidity dependent generally upon the proportion and kind of solvent used.

In the treatment of acetyl cellulose for the production of solutions for the manufacture of lacquers, varnishes, photographic films and the more massive or plastic compounds, we have discovered that ethyl oxybutyrate besides its capacity for dissolving cellulose nitrate, is also a valuable solvent for the acetate. Its empirical formula is  $(\text{CH}_3)_2\text{COHCOOC}_2\text{H}_5$ —which formula represents the iso modification. It has a boiling point of about  $145^\circ \text{C}.$ ; is water white, and the resulting products, such as films, varnishes, coatings sheets, solid objects, etc. are substantially free from odor. In addition to this we find that the ethyl oxy-butyrate is not alone in possessing this peculiar solvent power, for its homologues or other esters of oxy-butyrac acid are to a certain extent more or less efficacious as dissolvents or compatible menstrua. The most conspicuous of these homologues is methyl oxybutyrate. The other homologues will at once become apparent to every chemist. Propyl oxybutyrate and butyl oxybutyrate are, of course, included in this group of homologues. The structural formula is as follows, wherein R stands for the alkyl radical



For the purpose of this specification and the

claims, compounds conforming to the above structural formula will be termed alkyl esters of alpha-oxy-iso-butyric acid.

It will be understood by those skilled in the art that certain liquids (acetone, for example) are independent solvents of some varieties of acetyl cellulose, and likewise that other liquids (say alcohol and ether) are non-solvents; while again, certain non-solvent liquids form solvent mixtures, a good example being chloroform and alcohol. Further, some positively non-solvent menstrua act favorably when associated with active solvents. Compatibility or affinity is therefore an important operating condition for practical work and the value of our new solvent will be appreciated when we state that it has been found that it may be used in conjunction with all of the usual acetyl menstrua. In other words, it will mingle and co-operate with those menstrua which are either solvents in themselves or non-solvents which are used to restrain the volatility or modify the solvent action of the other ingredients. Such restraining or modifying influence aims to adapt the compound to particular purposes and render it subject to the varying degrees of temperature, pressure and atmospheric exposure necessary to accomplish the desired object. The properties of our new menstruum are practically efficient in connection with these aims. Other compatible agents, such as pigments or colors, can also be associated with our ethyl oxybutyrate solutions or compounds.

According to one method of carrying out our invention, there is taken one part of an acetyl cellulose more or less readily soluble in acetone, depending on the consistency or viscosity desired, this being immersed in ten parts (by weight) of ethyl oxy-butyrate. The acetyl cellulose gradually dissolves and the solution is completed by stirring, suitable mixing or warming. The solution thus prepared may be used alone or in conjunction with other liquids of solvent or non-solvent character, for example, methyl or ethyl alcohol, benzol, acetone, ethyl acetate, chloroform and ethylene dichloride; also with camphor and its substitutes, such as triphenyl phosphate, mannol, triacetin, di ethyl phthalate, or a plasticizer of the aromatic sulfonamid class, such as para ethyl toluene sulphonamid, etc.

One satisfactory use for the ethyl oxy-butyrate is in the production of cellulose acetate airplane dope where it can be substituted wholly or in part for the diacetone alcohol which has hitherto been employed as a "non-blushing" agent. In the case of incorporation with plasticizers, we associate

our solvent with other liquid agents such as acetone and alcohol and select an acetyl cellulose of solubility or viscosity adaptable to the purpose. In general, the variety of acetyl cellulose we prefer to use is that which is called "acetone soluble" to distinguish it from the modification which is resistant to acetone and only soluble in particular menstrua, as is well understood. But, as stated, this solubility in acetone is often in itself a matter of degree and subject in some sort to the influence of temperature or other associate conditions. So while we obtain superior effects by employing our acetone-soluble selection, we can apply our solvent to the more stubborn grades as a valuable constituent of the mixture.

An example of incorporation of our new solvent with plasticizers is as follows:

	Parts
Acetyl cellulose (acetone soluble) .....	100
Triphenyl phosphate .....	30
Para ethyl toluene sulphonamid .....	15
Acetone .....	85
Alcohol .....	15
Ethyl oxy-butyrate .....	20
Suitable stabilizer, such as urea .....	1

After the above stiff mass of acetyl cellulose has been suitably soaked or kneaded, it is worked up on converting rolls according to the usual celluloid practice. The ethyl oxy-butyrate functions here as a "high boiler" and retards any rapidly volatile tendency of the other solvents. Of course, by employing an excess of such diluents as acetone and alcohol, a flowable dope can be readily obtained for spreading on suitable surfaces and exposure to drying, say for the production of cinematograph film.

The proportions of plasticizers and solvents can of course vary greatly from the foregoing, according to the results desired.

On account of its superior solvent action for acetyl cellulose, ethyl oxy-butyrate can be employed alone or in conjunction with other solvents as a cementing agent for plastic sheets having a cellulose acetate base.

A good cementing formula consists of,

	Parts by weight
Ethyl oxy-butyrate .....	9
Benzyl alcohol .....	1

To give this solution body, some acetyl cellulose may be dissolved therein, but of course it

will be understood that the association of our solvent with the acetyl cellulose article forms a solution which is practically the equivalent of one made directly.

As for methyl oxy-butyrate, this also is an active solvent of the acetone soluble variety of cellulose acetate, and can be used interchangeably with the ethyl compound. It would appear that the solvent power of the ethyl oxy-butyrate is not always as energetically reflected in its homologues; for example, the amyl oxy-butyrate is not really an active solvent. The more remote oxy-butyrate relatives, say those of the non-aliphatic group are apt to show the same inclination. But, as before stated, even substances of apparently non-solvent character are really of favorable power when associated with more active menstrua, and this is because of a latent compatibility. In this sense all of the oxy-butyrate esters can be regarded as solvents. The principle is best exhibited in the non-solvent fusel oil when it is made part of a nitro-cellulose solvent mixture with which it acts as a solvent. Similarly these apparently non-solvent oxy-butyrate esters are more or less useful as compatible modifiers. In other words, we have found more or less use for the esters of oxy-butyric acid as acetyl cellulose solvents.

What we claim and desire to secure by Letters Patent is:—

1. Process for the manufacture of plastic material, which comprises forming a stiff mass of a composition comprising cellulose acetate and an alkyl ester of alpha-oxy-iso-butyric acid, the said alkyl ester of alpha-oxy-iso-butyric acid being present in an amount equal to 20% based upon the weight of the cellulose acetate whereby the rolling characteristics of the mass are improved, and then drying and hardening the mass by rolling the same with the aid of heat.

2. Process for the manufacture of plastic material, which comprises forming a stiff mass of a composition comprising cellulose acetate and ethyl alpha-oxy-iso-butyrate, the said ethyl alpha-oxy-iso-butyrate being present in an amount equal to 20% based upon the weight of the cellulose acetate whereby the rolling characteristics of the mass are improved, and then drying and hardening the mass by rolling the same with the aid of heat.

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