

UNITED STATES PATENT OFFICE.

ROBERT C. SCHÜPPHAUS, OF BROOKLYN, NEW YORK.

PYROXYLIN COMPOUND.

SPECIFICATION forming part of Letters Patent No. 741,554, dated October 13, 1903.

Application filed June 2, 1896. Serial No. 594,006. (No specimens.)

To all whom it may concern:

Be it known that I, ROBERT C. SCHÜPPHAUS, of Brooklyn, New York, have invented certain new and useful Improvements in the Manufacture of Pyroxylin Compounds and in the Pyroxylin Compounds Resulting Therefrom, of which the following is a specification.

While the invention relates generically to pyroxylin compounds and processes of manufacturing them, it more particularly relates to new liquids for breaking down the fibrous structure of pyroxylin and converting it into a homogeneous substance of any desired plasticity ranging from a flowing solution into a stiff jelly-like mass, according to the purposes for which the pyroxylin compound is to be used.

I have discovered that there are a number of closely-related chemical substances which combine the desirable solvent or converting powers of the ordinary ether in its well-known ether-alcohol mixture with a greatly-decreased volatility and which are far less miscible with water.

It is the purpose of the present invention to utilize this discovery in the manufacture of pyroxylin compounds and to produce pyroxylin compounds thereby which contain these substances or which result from the admixture of pyroxylin and these substances. As a class these substances consist of the lower mixed alkyl oxids, or, in other words, the mixed ethers of the lower alcohols. These substances in conjunction with a suitable adjuvant—*e.g.*, ethyl alcohol—are of great value in the manufacture of pyroxylin compounds. By the “lower” alcohols I mean particularly the aliphatic alcohols of the first five series—to wit, methyl, ethyl, propyl, butyl, and amyl. All these are, with the exception of methyl or wood alcohol, produced commercially by fermentation processes. Methyl alcohol is more commonly obtained by the destructive distillation of wood, although this, too, may be made from certain sugar solutions by peculiar fermentation. The mixed alkyl oxids or mixed ethers which I particularly prefer are methylisobutyl oxid, which boils at about 60° centigrade, ethylisobutyl oxid, which boils at 78° to 80° centigrade, methylisoamyl oxid, which

boils at 92° centigrade, and ethylisoamyl oxid, which boils at 112° centigrade.

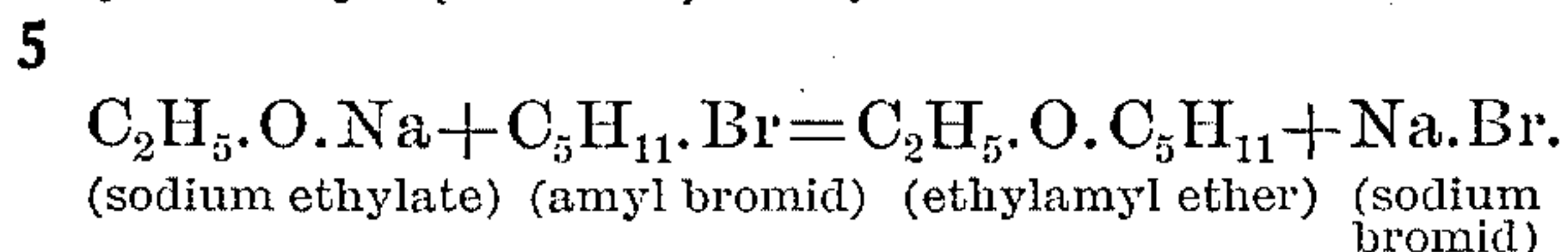
It is known that any one of the lower alcohols may, to some extent, replace ethyl alcohol in the mixture of ether and alcohol, which is very commonly employed as a solvent for pyroxylin; but while the great volatility of these combinations is of advantage in many applications in others this very volatility is a serious drawback.

One object of the present invention is to produce pyroxylin compounds by the use of substances which have far less volatility than the ordinary ether-alcohol mixture and which are far less miscible with water. The mixed alkyl oxids which I have enumerated possess when used instead of simple ether both these characteristics and at the same time exert the powerful solvent action desired. Any one of these alkyl oxids is mixed with a suitable quantity of an alcohol—as, for instance, ethyl or propyl alcohol—and the liquid solvent for the pyroxylin thereby prepared. By combining soluble pyroxylin of the varieties used for the preparation of collodion and varnishes with these liquids pyroxylin solutions are obtained which are suitable, among other purposes, for varnishes and for the preparation of films. I have also found these mixed alkyl oxids of great utility in the manufacture of solid compounds, especially in the preparation of explosive pyroxylin compounds containing a mixture of soluble and insoluble guncotton where it is desired to gelatinate the soluble variety only.

I am aware that Letters Patent of the United States have been granted to Leonard Paget, April 14, 1893, which describe the use of the substances obtained by distilling methyl and ethyl alcohol, respectively, with fusel-oil and sulfuric acid, and I am aware that these substances are spoken of in these patents as containing methylamyl ether and ethylamyl ether, and I desire to make it clearly understood that I make no claim to the mixture of substances obtained in the manner described in these two patents, which is distinct from the mixed alkyl oxids which form the subject of my present application.

I first prepared the mixed ethers which form

the subject-matter of this application according to Williamson's classical method by treating a sodium alkylate ($R-ONa$) with a halogen alkyl ($R'-Br$)—as, for instance:



- 10 This method, as will be well understood by chemists, (to whom particularly this specification is addressed,) is merely a laboratory method of preparation, and I give it in order to distinguish clearly the constitutional formula of the mixed alkyl oxids which I employ in carrying out this invention. These substances may be prepared on a large scale by other and less costly methods—such, for instance, as that described in United States Patent No. 516,766, dated March 20, 1894, to Krafft and Roos. The method of manufacture, provided it results in substances having a constitutional formula similar to that described, is immaterial to my invention.
- 25 What I claim, and desire to secure by these Letters Patent, is—

1. The improvement in the art of manufacturing pyroxylin compounds, which consists in dissolving or converting pyroxylin with the aid of the solvent action of a mixed ether, substantially as set forth.

2. As an improvement in the art of manufacturing pyroxylin compounds, the treating of pyroxylin by the aid of one or more members of the following group of substances: methylisobutyl oxid, ethylisobutyl oxid, methylisoamyl oxid, and ethylisoamyl oxid, substantially as set forth.

3. The process of gelatinating soluble pyroxylin, which consists in treating the soluble pyroxylin with a solvent that contains one or more of the mixed alkyl oxids, substantially as described.

4. The process of gelatinating soluble pyroxylin, which consists in treating the soluble pyroxylin with a solvent mixture of one or more of the mixed alkyl oxids and an adjuvant, in contradistinction to a solvent mixture in which the ether present is predominantly simple ether.

5. A pyroxylin composition containing pyroxylin and one or more mixed alkyl oxids.

6. A pyroxylin composition containing pyroxylin and one or more members of the following group of substances: methylisobutyl oxid, ethylisobutyl oxid, methylisoamyl oxid, ethylisoamyl oxid, substantially as set forth.

7. As a new article of manufacture, a liquid pyroxylin composition containing pyroxylin and one or more of the mixed alkyl oxids.

8. As an improvement in the art of manufacturing pyroxylin compounds, the treating of pyroxylin by the aid of ethylisoamyl ether and an adjuvant.

9. As an improvement in the art of manufacturing pyroxylin compounds, the treating of pyroxylin by the aid of a mixed alkyl oxid containing an alkyl radical of less than four carbon atoms and an alkyl radical of more than three carbon atoms, with a suitable adjuvant.

10. A pyroxylin composition containing pyroxylin and ethylisoamyl ether with a suitable adjuvant, substantially as set forth.

11. A pyroxylin composition containing pyroxylin and a mixed alkyl oxid containing an alkyl radical of less than four carbon atoms and an alkyl radical of more than three carbon atoms with a suitable adjuvant, substantially as set forth.

12. The process of manufacturing pyroxylin compounds which consists in mixing pyroxylin with one or more members of the group of mixed ethers consisting of ethylisobutyl oxid, ethylisoamyl oxid, ethylisopropyl oxid, propylamyl oxid, butylamyl oxid, and propylbutyl oxid.

13. A pyroxylin composition containing pyroxylin and one or more members of the group of mixed ethers consisting of ethylisobutyl oxid, ethylisoamyl oxid, ethylisopropyl oxid, propylamyl oxid, butylamyl oxid and propylbutyl oxid.

In testimony whereof I have hereunto set my hand this 1st day of June, A. D. 1896.

ROBERT C. SCHÜPPHAUS.

In presence of—

J. C. ROSS,

HAROLD BINNEY.