UNITED STATES PATENT OFFICE.

JOHN H. STEVENS, OF NEWARK, NEW JERSEY, ASSIGNOR TO THE CELLULOID COMPANY, A CORPORATION OF NEW JERSEY, OF SAME PLACE.

PYROXYLIN COMPOUND.

SPECIFICATION forming part of Letters Patent No. 553,270, dated January 21, 1896.

Application filed July 26, 1895. Serial No. 557,235. (No specimens.)

To all whom it may concern:

Be it known that I, JOHN H. STEVENS, of the city of Newark, in the county of Essex and State of New Jersey, have invented certain new and useful Improvements in Pyroxylin Compounds, of which improvements the following is a specification.

The pyroxyline compounds to which the present invention relates, and the state of the art, are fully described in my United States Patent No. 517,987.

The present invention has for its object the formation of new compositions of matter rendered possible by my discovery that dinitroxylol, $C_6H_2(NO_2)_2(CH_3)_2$, when melted by means of heat, is a solvent of pyroxyline and forms extremely useful compound solvents with camphor and acetanilid. While the dinitroxylol possesses solvent powers in itself which give plasticity to the compounds containing it as the only solid solvent when they are heated under pressure, the most important feature of my invention is the fact that the use of the dinitroxylol in connection with either camphor or acetanilid, or both, improves the action of the camphor and acetanilid, so that the combinations are more plastic or easily manipulated, and the sheets or films deposited from liquid solutions are better than the same products made with either of the solid components of the solvent when it is used as the only solid solvent.

I find that the dinitroxylol does not harmonize with either ethylic or methylic alcohol, and that acetone is the proper liquid menstruum to be used in connection with it. With excessive proportions of either camphor or acetanilid, however, used in connection with the dinitroxylol the operator can employ ethylic or methylic alcohol, basing his operations on the information given in my United States Patent No. 517,987, before referred to.

When used alone as a solid solvent, I find that it is best to employ no more than fifteen parts or twenty parts of the dinitroxylol to each one hundred parts of soluble pyroxyline, and I would not recommend this solvent for that process of conversion which involves simply the use of solid solvents without the presence of liquids, except when the dinitroxylol is associated with the camphor or acetanilid, in which case it is capable of being used in all the processes, liquid and solid.

When used with camphor or acetanilid, I obtain the best plasticity in the final compound by the use of forty parts of a mixture of equal parts camphor and dinitroxylol to each one hundred parts of pyroxyline, or forty parts of a mixture of three parts dinitroxylol and one part acetanilid to each one hundred parts of pyroxyline.

The above proportions are used regardless of the amount of liquid solvent employed in the compound. A mixture of equal parts dinitroxylol and either camphor or acetanilid, or a mixture of both, however, forms an excellent solvent which can be used in any of the proportions employed in this art.

In addition to the usefulness dinitroxylol exhibits in conjunction with acetanilid and camphor, I have also found that it improves the action of other solid solvents.

I do not confine myself to any particular proportions, nor to any special method of mixing the ingredients, nor to the introduction of the dinitroxylol into the mixture at any particular stage of the mixing operation, for its effects on the compound are exhibited without regard to these conditions, though, of course, I prefer to use the best-known methods for mixing and converting the compounds.

The dinitroxylol exists in different modifications dependent on molecular structure. Practically I use the ordinary product containing a mixture of the different kinds of dinitroxylol, for they are all equivalents in this application.

In making the dinitroxylol which I prefer to use ordinary commercial xylol is acted on by a well-cooled mixture of strong nitric and sulphuric acids, the methods and proportions being well known to chemists.

The product consists almost entirely of the two modifications of the meta—*i. e.*, adjacent metadinitroxylol, $(C_6H_2)CH_3:NO_2:CH_3:NO_2: = 1:2:3:4:$, and symmetrical metadinitroxylol, $(C_6H_2)CH_3:CH_3:NO_2:NO_2: = 1:3:4:6:$. As a rule the adjacent modification preponderates, though it is understood that the proportion of the symmetrical modification is increased if the temperature is permitted to rise during the reaction. The rise of temperature is to be

avoided, as it permits the formation of the undesirable metatrinitroxylol. The ortho and para varieties of dinitroxylol, if present in the product, are in small proportion. Practically, therefore, it is a mixture of the two modifications of the metadinitroxylol.

What I claim, and desire to secure by Letters Patent, is—

1. The process of manufacturing pyroxyline compounds which consists in mixing dinitroxylol with pyroxyline, and afterward subjecting the compound to heat and pressure sufficient to render the compound plastic, substantially as described.

2. A method for the production of compounds of pyroxyline, which consists in mixing pyroxyline with dinitroxylol and one or more of the known solvents of pyroxyline and afterward subjecting the mixture to heat and pressure sufficient to render the compound plastic, substantially as described.

3. The process of manufacturing pyroxyline compounds which consists in mixing pyroxyline, dinitroxylol, one or more solid solvents of pyroxyline, and a liquid menstruum or liquid menstrua sufficient in amount to transform the mass into a pyroxyline solution or compound, substantially as set forth.

4. A composition of matter containing pyroxyline and dinitroxylol, substantially as described.

5. A composition of matter containing pyroxyline, dinitroxylol, and one or more of the known solvents of pyroxyline, substantially as set forth.

In witness whereof I have hereunto signed my name this 24th day of July, 1895.

JOHN H. STEVENS.

In presence of—
ABRAHAM MANNERS,
GEO. E. STOUT.