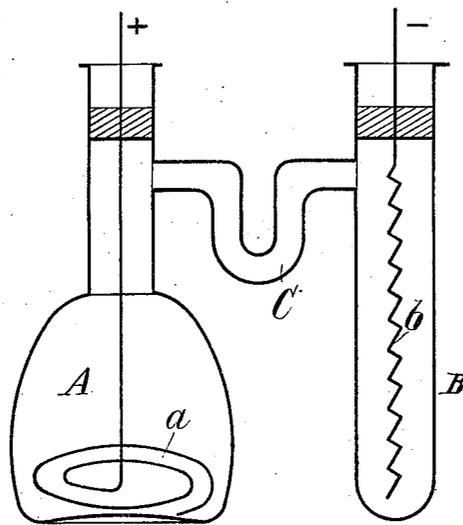


(No Model.)

M. OTTO & A. VERLEY.  
MANUFACTURE OF VANILLIN.

No. 553,593.

Patented Jan. 28, 1896.



Witnesses.

*David Lewis*  
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*by Joseph Mauro,*  
*their attorneys,*

# UNITED STATES PATENT OFFICE.

MARIUS OTTO AND ALBERT VERLEY, OF COURBEVOIE, FRANCE.

## MANUFACTURE OF VANILLIN.

SPECIFICATION forming part of Letters Patent No. 553,593, dated January 28, 1896.

Application filed July 8, 1895. Serial No. 555,333. (No specimens.)

*To all whom it may concern:*

Be it known that we, MARIUS OTTO and ALBERT VERLEY, chemists, citizens of the French Republic, residing at 7 Quai de Seine, Courbevoie, (Seine,) in the Republic of France, have invented certain Improvements in the Manufacture of Vanillin, of which the following is a specification.

The object of our invention is to obtain vanillin in a cheap and expeditious manner by synthesis. To effect this object we oxidize a solution of isoeugenate of soda (or other convenient base) and treat the resulting product with an acid and separate the vanillin so produced. This oxidation may be effected by electrolyzing an alkaline solution of isoeugenate of soda, either cold or warm, preferably at the temperature of an ordinary water-bath. This electrolysis may be conveniently effected by placing the solution of isoeugenate of soda in a vessel standing in a water-bath and connected with a vessel containing a solution of soda by a U-shaped tube, which passes through the sides of the respective vessels at a convenient height above the bottoms of the vessels. Electrodes, preferably of platinum, are placed in the vessels and connected to the respective poles of a source of electricity, the electrode in the vessel containing the solution of isoeugenate of soda being at the bottom and connected by an insulated wire, so that the current may pass through the whole mass of the fluid and oxidize the isoeugenate of soda. By this arrangement the oxidation is effected without any substantial mixing of the two liquids. The reaction which takes place is the oxidation of the isoeugenate of soda, as before explained, and thereby its change into vanillate of soda. The subsequent treatment with an acid (which may, for instance, be oxalic acid or sulphuric acid diluted with its own volume of water) forms a sodium salt in accordance with the acid used and sets free the vanillin.

To ascertain if the oxidation of the isoeugenate of soda is complete the quantity of hydrogen which is set free at the negative pole may be measured and from it be deduced the quantity of oxygen set free at the posi-

tive pole; or the test may be simply made by reference to the quantity of oxygen at the positive pole; but the invention is not limited, of course, in any way to any particular test.

The accompanying drawing illustrates diagrammatically an apparatus which may conveniently be used in carrying out our process.

The solution of isoeugenate of soda is placed in the vessel A. In the vessel B we place a solution of soda serving merely as a conductor between the positive electrode *a* and the negative electrode *b*. These vessels are connected by a U-shaped tube C at a convenient distance above the bottoms of the vessels, so that while the liquid forms a continuous path closing the internal circuit of the electrolytic bath there will be no mingling of the products formed around the respective electrodes. This arrangement is adopted for economy and to increase the yield from the operation. The products of oxidation formed around the positive electrode are those utilized in our process of making vanillin, and it is preferred that they should not be contaminated by the products formed at the negative pole. The electric circuit is completed by connecting the terminals of electrodes *a* and *b* respectively with the positive and negative poles of an electric generator, preferably one furnishing a continuous current of low tension.

When the oxidation is judged to be complete the solution is treated with an acid which sets free the vanillin. It can be purified with bisulphide of soda and by repeated crystallization, as may be found requisite.

We claim—

The manufacture of vanillin by subjecting a solution of iso-eugenate of soda or other base to electrolysis and then treating by means of an acid.

In testimony whereof we have signed our names to this specification in the presence of two subscribing witnesses.

MARIUS OTTO.  
A. VERLEY.

Witnesses:

J. CINELLERMET,  
G. DÉCHAUS.