

UNITED STATES PATENT OFFICE.

CARL METTLER, OF MUNICH, GERMANY, ASSIGNOR TO BADISCHE ANILIN & SODA FABRIK, OF LUDWIGSHAFEN-ON-THE-RHINE, GERMANY, A CORPORATION.

PRODUCTION OF AROMATIC ALCOHOLS.

No. 815,193.

Specification of Letters Patent.

Patented March 13, 1906.

Application filed October 11, 1905. Serial No. 282,321.

To all whom it may concern:

Be it known that I, CARL METTLER, doctor of philosophy and chemist, a citizen of the Swiss Republic, residing at Munich, in the Kingdom of Bavaria, German Empire, have invented new and useful Improvements in the Production of Aromatic Alcohols, of which the following is a specification.

In the specification of my application for Letters Patent, Serial No. 231,149, filed November 2, 1904, I have described the manufacture of aromatic alcohols and of the corresponding ethers by electrolytically reducing aromatic esters, making use of a cathode of high cathodic tension. I have now discovered that the free carboxylic acids themselves can also be reduced in an analogous manner, giving rise solely to the alcohols. It is necessary to employ for the cathode a metal of high cathodic tension, and for this purpose lead is particularly useful, although of course other metals—such, for instance, as mercury—which fulfil the above condition may be employed. The course of the reaction can be conveniently illustrated by the following equation:



The following examples will serve to further illustrate the nature of my invention and the manner of carrying it into practical effect; but my invention is not confined to these examples. The parts are by weight.

Example 1: Introduce into the cathode-compartment of an electrolytic apparatus a solution of two hundred (200) parts of benzoic acid in four hundred (400) parts of concentrated sulfuric acid and fourteen hundred (1,400) parts of alcohol. The cathode should consist of sheet-lead prepared according to the receipt given by Tafel in the *Berichte*, 33, 2,215. The anode should consist of pure lead, and the anode-compartment should be filled with dilute sulfuric acid. Send through the liquid an electric current of from six (6) to twelve (12) amperes per one hundred (100) square centimeters of cathode-surface and cool by means of water, so that the temperature remains between twenty (20) and forty (40) degrees centigrade. As soon as no more hydrogen is absorbed neutralize the cathode liquid with caustic soda and extract the alcohol which is formed by means of ether. The yield of benzyl alcohol boiling at

a temperature of two hundred and one (201) degrees centigrade is almost quantitative.

Example 2: Introduce into the cathode-compartment of an electrolytic apparatus a solution of twenty (20) parts of para-hydroxy-benzoic acid, fifty-five (55) parts of concentrated sulfuric acid, and fifty-six (56) parts of alcohol. The cathode and the anode should consist of pure lead, and the anode-compartment should be filled with thirty (30) per cent. sulfuric acid. Pass through the cell an electric current of ten (10) amperes per one hundred (100) square centimeters of the cathode-surface. As soon as from two (2) to three (3) times the theoretically-necessary quantity of electricity has been passed through the cell work up the cathode liquid by adding sodium-carbonate solution until it is alkaline and drive off the alcohol employed by distilling with steam. Then extract the reaction product from the residue by means of ether.

The reduction of meta-hydroxy-benzoic acid can be carried out in a manner similar to that above described.

Example 3: Introduce into the cathode-compartment of an electrolytic apparatus a solution of one (1) part of anthranilic acid in fifteen (15) parts of sulfuric acid and carry out the electrolysis in the manner described in the foregoing example 2, taking care that the temperature of the cathode liquid does not exceed thirty (30) degrees centigrade. Then add solid ammonium carbonate to the cathode liquid until it is alkaline. Filter, saturate the filtrate with ammonium sulfate, and extract with ether. On distilling off the ether ortho-amido-benzyl alcohol remains behind in the form of an oil which solidifies and can be purified by crystallization from light petroleum.

Example 4: Dissolve one hundred (100) grams of meta-nitro-benzoic acid in one (1) liter of thirty (30) per cent. alcoholic sulfuric acid and reduce this in the manner described in the foregoing examples, maintaining the temperature at about forty (40) degrees centigrade. After passing about three hundred (300) ampere hours through the cell make the reaction liquid alkaline by means of ammonium carbonate, saturate with ammonium sulfate, and extract several times with ether. Upon distillation the ether leaves behind an oil which rapidly solidifies,

and by crystallization meta-amido-benzyl alcohol can be obtained in the form of white crystalline needles.

Now what I claim is—

5 1. The manufacture of aromatic alcohols by electrolytically reducing aromatic carboxylic acids making use of a cathode of high cathodic tension.

10 2. The manufacture of aromatic alcohols by electrolytically reducing aromatic carboxylic acids, making use of a lead cathode.

3. The manufacture of para-hydroxy-benzyl alcohol by electrolytically reducing para-hydroxy-benzoic acid, making use of a lead cathode.

In testimony whereof I have hereunto set my hand in the presence of two subscribing witnesses.

CARL METTLER.

Witnesses:

MATHILDE K. HELD,
GEORG KÖRNER.