UNITED STATES PATENT OFFICE.

ALBERT BAUR, OF GERNRODE, ASSIGNOR TO THE FABRIQUES DES PRODUITS CHIMIQUES DE THANN ET MULHOUSE, OF THANN, GERMANY.

ARTIFICIAL MUSK.

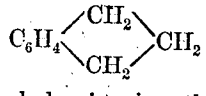
SPECIFICATION forming part of Letters Patent No. 536,324, dated March 26, 1895.

Application filed November 30, 1894. Serial No. 530,382. (Specimens.) Patented in France June 30, 1894, No. 195,360, and in England July 4, 1894, No. 12,980.

To all whom it may concern:

Be it known that I, ALBERT BAUR, a resident of Gernrode, in the dukedom of Anhalt, Germany, have invented certain new and useful Improvements in the Manufacture of Artificial Musk, which are fully described in the following specification, and for which I have obtained Patents in France June 30, 1894, (certificate of addition to Patent No. 195,360,) and in England July 4, 1894, No. 12,980.

In previous patents, Nos. 416,710, dated December 10, 1889, 451,847, dated May 5, 1891, and 481,685, dated August 30, 1892, I have described and claimed the preparation of artificial musk by nitration of aromatic hydrocarbons containing two or three lateral chains, one of which must be a butylic, amylic or propylic one, while the others are methyle or ethyle. Hydrocarbons of this family are butyl-toluene, butyl-xylene and many others. I have found that not only the hydrocarbons of the said series are able to yield musk by nitration, but ethol aromatic hydrocarbons with lateral chains for instance hydrindene



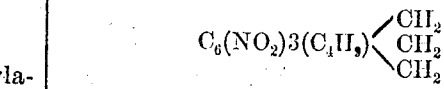
show the same behavior, *i. e.*, that by butylation (propylation or amylation) and nitration they are transformed into nitro-derivatives possessing a strong odor of musk.

To carry out my present invention I introduce the butylic (propylic or amylic) radicle in the hydrindene by one of the well known methods, for instance by the method of Friedel and Crafts. For instance, I heat thirty parts of hydrindene, seven parts of chlorid of aluminium, ten parts of chlorid of tertiary butyl, until no more evolution of hydrochloric acid takes place. I pour in water, distill the hydrocarbon with steam and purify the butyl-hydrindene by fractional distillation. This butyl-hydrindene is itself a new body. The boiling point of the pure hydrocarbon is 237° to 240° centigrade.

In order to transform the butyl-hydrindene into its musk-smelling trinitro derivative, it is more convenient to prepare first the dinitro compound in a state of purity and to submit it afterward to a further nitration. For this purpose I introduce ten parts of butyl-hydrindene into a well cooled mixture of forty parts of fuming nitric acid (specific gravity 1.48 about) and eighty parts of fuming sulfuric acid, containing about fifteen per cent. of SO_3, and allow the solution to stand a few hours. I then pour in water, filter the nitro compound, wash it with soda and afterward with water and crystallize it out of alcohol. I thus obtain white crystals melting at 121° centigrade and possessing no musky odor.

In order to transform the dinitro into trinitro derivative I introduce ten parts of the dinitro compound into a strong nitration mixture containing forty parts of fuming nitric acid and eighty parts of fuming sulfuric acid containing from thirty to forty per cent. of SO_3, and heat at a temperature not exceeding 50° or 55°. After a few hours I pour in water, filter and crystallize the new compound from alcohol.

The trinitrobutylhydrindene



comes out in the form of white needles, sparingly soluble in cold alcohol, melting at 139° to 140° centigrade and possessing a very strong musk odor.

I may also effect the butylation of hydrindene by introducing the same mixed with butylic alcohol in concentrated sulfuric acid. I obtain in this way a mixture of the hydrocarbon and of its sulfuric acid. The latter by nitration yields also a musk but the formerly described process is much better.

By using chlorid of propyle or amyle instead of chlorid of butyle, I obtain derivatives analogous to the butylic but presenting no advantage. It will be understood therefore that where mention is made of the butylic chlorid, the propylic and amylic chlorids are included as equivalents. It will also be understood that the amylic and propylic deriva-

tives of hydrindene, and bodies of that class, are analogous to the butylic derivative, and are embraced in the invention.

Having now particularly described my invention and the manner in which the same is to be carried out, what I claim is—

1. The described process of obtaining an artificial musk, by heating an ethol-aromatic hydrocarbon, such as hydrindene, with a chlorid such as butylic chlorid in presence of a metallic chlorid, and nitrating the product of this reaction, thereby obtaining a trinitro derivative of the hydrocarbon treated, as set forth.

2. In the manufacture of artificial musk, the improvement consisting in heating hydrindene with butylic chlorid and a metallic chlorid, thereby producing butyl hydrindene, as set forth.

3. The artificial musk herein described, the same being the trinitroderivative of butylhydrindene in the form of white needles, sparingly soluble in alcohol, and characterized by a strong odor of musk, as set forth.

In witness whereof I have hereunto signed my name in the presence of two subscribing witnesses.

ALBERT BAUR.

Witnesses:
EDUARD MÜAZLL,
EMIL NORT.