

UNITED STATES PATENT OFFICE.

ALBERT HESSE, OF WILMERSDORF, GERMANY.

PROCESS FOR PURIFYING PITCH-OIL AND OTHER ETHEREAL OILS OBTAINED BY DESTRUCTIVE DISTILLATION OF RESINOUS SUBSTANCES.

No. 835,907.

Specification of Letters Patent.

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To all whom it may concern:

Be it known that I, ALBERT HESSE, a subject of the German Emperor, and a resident of Wilmersdorf, in the Kingdom of Prussia, German Empire, have invented new and useful improvements in processes for purifying pitch-oil and other ethereal oils obtained by destructive distillation of resinous substances, of which the following is a full, clear, and exact specification.

Among the turpentine-oils of commerce the American and French turpentine-oils are more valuable than turpentine-oils coming from other countries, and which are more correctly named "pitch-oils." These oils are obtained by the destructive distillation of resinous substances of different kinds, such as wood from resinous trees and the like, colophonium, &c. They are generally termed "pitch-oils," "pine-oils," "wood-turpentine," "stump-turpentine," "wood-spirits," "resinous oils," and like names—in German, "kienöle, harzöle." These oils are referred to in the well-known standard work *Die ätherischen Oele*, by E. Gildemeister and Fr. Hoffmann, Berlin, 1899, pages 329–333, in the chapter entitled "Kienöle;" see also Ino M. McCandless, *Journal of the American Chemical Society*, 26, pages 981–985. This difference is partly caused by the different methods used when preparing these oils, for these pitch-oils are obtained by destructive distillation of resinous substances, while the genuine turpentine-oils are prepared by distilling pine-resin with steam. Moreover, the parts of the plants or the exuded matter used for preparing pitch-oils often contain substances which cause ill-smelling compounds to be formed. The nature of these ill-smelling compounds has not been ascertained up to date; but their intensely bad smell, notwithstanding their small quantity, renders it impossible to use the said oils for many purposes. Many attempts have been made for eliminating these malodorous compounds in order to render these oils as valuable as the genuine turpentine-oils. These methods are based partly on a saponifying and partly on an oxidizing treatment of the oils; but the smell of the commercial pitch-oils purified by these methods shows that this treatment is not sufficient for eliminating the ill-smelling substances, and this has been confirmed by many experiments. Now I have found

that by treating these oils with alkali metals or alkaline-earth metals (both hereinafter comprised under the expression "alkaline metals") the compounds causing the disagreeable smell are transformed into non-volatile compounds. Consequently it is possible to obtain in a very simple manner and without difficulty a product from which the malodorous compounds are eliminated by distilling the oil with addition of small quantities of an alkali metal or an alkaline-earth metal. A remarkably small quantity of alkali metal or alkaline-earth metal is sufficient for eliminating the ill-smelling substances, and the process can therefore be carried out on an industrial scale with good success and cheaply notwithstanding the relatively high price of the reagents used. The distillation with addition of alkaline metals may be preceded by a purification with alkaline or alkaline-earth hydroxids, this purification being executed in the known manner.

When compared with other processes, and especially with those in which the ill-smelling compounds are to be eliminated by an oxidizing treatment, my new process has the advantage that a loss of valuable substances is avoided, as no destruction of the unsaturated compounds, which form the most valuable part of the oils, takes place, while in the oxidizing processes such destruction may occur.

In practically carrying out my process I proceed, for instance, as follows:

Example 1: Into one thousand kilos of Russian turpentine-oil (more correctly pitch-oil) three kilos of metallic sodium are stirred. The oil is then distilled under ordinary pressure. If necessary, the distillate, or a part thereof, is again treated in the same manner.

Example 2: One thousand kilos of American wood-oil are boiled for an hour with five kilos of caustic soda and thirty kilos of alcohol. The alcohol is then removed by distillation or by washing, and the saponified oil is then distilled *in vacuo* after drying with two kilos of the liquid alloy of potassium and sodium.

Example 3: One thousand kilos of Polish pitch-oil are heated with five kilos of calcium hydroxid and then distilled *in vacuo* or under ordinary pressure with addition of 1.5 kilos of metallic calcium. If necessary, the distillate is again treated in the same manner.

What I claim is—

1. The hereinbefore-described process of eliminating the ill-smelling substances from ethereal oils obtained by destructive distillation of resinous substances, said process consisting in distilling the oils with addition of alkaline metals, substantially as described.

2. The hereinbefore-described process of eliminating the ill-smelling substances from ethereal oils obtained by destructive distillation of resinous substances, said process consisting in distilling the oils *in vacuo* with addition of alkaline metals, substantially as described.

3. The hereinbefore-described process of eliminating the ill-smelling substances from

pitch-oils, said process consisting in distilling the oils with addition of alkaline metals, substantially as described.

4. The hereinbefore-described process of eliminating the ill-smelling substances from pitch-oils, said process consisting in distilling the oils *in vacuo* with addition of alkaline metals, substantially as described.

In testimony that I claim the foregoing as my invention I have signed my name in presence of two subscribing witnesses.

ALBERT HESSE.

Witnesses:

HENRY HASPER,
WOLDEMAR HAUPT.