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PROCESS FOR DISINTEGRATING OR DECOMPOSING COAL TAR OR ITS DISTILLATES INTO PHENOLS AND NEUTRAL OILS.

No Drawing.

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To all whom it may concern:

Be it known that I, ANTON WEINDEL, a citizen of the German Republic, residing at Essen-Ruhr, Germany, have invented
5 new and useful Improvements in Processes for Disintegrating or Decomposing Coal Tar or Its Distillates Into Phenols and Neutral Oils, of which the following is a specification.

10 For separating the acid constituents (phenols) of tar or tar oils the property of these substances to form with alkali, salts soluble in water (phenates) is generally utilized. This process is very expensive,
15 as in addition to the expensive caustic soda, equivalent quantities of carbonic acid or mineral acid must also be used. Attempts to replace the inconvenient and expensive phenol extraction by some cheaper
20 process have hitherto only been successful in the case of tar from lignite. According to the process of the Riebeckschen Montanwerke, the constituents of the phenol type which are present in comparatively small
25 quantities in lignite tar and distillate, are dissolved in high per cent alcohol in which the paraffin hydrocarbons are almost insoluble. This process is a failure in the case of
30 ordinary coal tar because here about 90% of the neutral oils are of an unsaturated nature and have a very different solubility in alcohol. Further in the case of crude coal tar the conditions are substantially different. Whilst in the case of distilled lignite tar
35 (braunkohlenschwelteer) the phenols are of no use for chemical or pharmaceutical purposes as they are too impure and mostly represent high boiling point products which under the name of "Fresol" can merely be
40 used for crude disinfecting purposes, in crude coal tar the phenols are present to a far larger extent (up to about 50% of the tar) and further these phenols exist in a form adapted for use as intermediate products
45 of the chemical industry and as valuable technically approved disinfectants. From an economic point of view therefore, it is of the greatest importance to separate the phenols of the crude tar from the neutral oils in a simple and inexpensive manner.

According to this invention the method of operating consists in that crude coal tar

or better still its distillate is agitated under heat with a sufficient quantity of aqueous
55 alcohol containing not more than 60% by weight of alcohol and then after a brief settling the dilute alcohol layer of phenol can be separated from the layer of neutral
60 oil. It is not advisable to employ stronger alcohol, as, contrary to expectation the separation becomes always more incomplete with an increase in the alcoholic content until it entirely ceases with about 90% alcohol. The differences of solubility are first shown
65 with highly diluted alcohol. The quantity of alcohol to be employed should be large enough to obtain the main separation as far as possible in one operation. In contrast to
70 what is the case with lignite tar, the counter-current principle is here not found suitable as with too small quantities of alcohol it is almost entirely divided into an alcoholic crude tar distillate layer and a water layer
75 almost entirely free of alcohol. It is even possible to concentrate in this way highly diluted alcohol by means of crude tar distillate.

The separation of the phenols from neutral oils derived from coal or wood tar oils with
80 aqueous alcohol has already been proposed. However the use of this method of operating was not immediately apparent in the case of crude coal tar oils and shows quite other results. In the process already known
85 it is a question of obtaining phenols from a given fraction of coke oven tar whilst by the process according to this invention the whole crude tar distillate is divided into its main constituents, phenols and neutral
90 hydrocarbons. Coke oven tar and crude tar are quite different from one another. In coke oven tar phenols and cresols are especially present whilst in crude tar phenol is hardly visible and cresols are only present
95 in very small quantities.

The methods of carrying out the two processes in question are also different. The prior inventors work with alcohol containing
100 25 to 35% of water and at the most the proportion of water is 40%. In the present invention 10% of water is the smallest permissible proportion, that is to say this process only commences to yield profitable results where the older process ends. Alcohol containing 49% of water gives the

best results. While the older inventors obtained, according to their own statements, impure phenols, by this improved process pure products are obtained. It is almost impossible to obtain pure substances from the impure phenols by fractional distillation as the neutral impurities present have a boiling point approximately the same as that of the phenols.

10 This invention operates in similar manner with crude tar and crude tar distillate.

Preferably the crude tar fraction is selected in which the most numerous and valuable phenols are accumulated, that is to say, 15 the fraction from about 150°-250° C. boiling point.

The alcohol is almost entirely recovered by re-distillation from the two layers.

If it be desired to obtain as pure phenols as possible, it is preferable to cool the phenol solution which is at about 70° C. down to ordinary temperature whereby the last remains of any dissolved hydrocarbons are separated off in a middle fraction. In 20 this way phenols having a degree of purity of over 99% are obtained. For most purposes it is not necessary to carry the separation so far. The layer of alcoholic hydrocarbon (the middle fraction above mentioned) is further purified (up to 98% degree of purity, that is to say, until it contains only about 2% of phenols) by repeated washing with small quantities of alcohol of the same dilution, but, as experiments have 25 shown, there is nothing to prevent the use of hydrocarbons which are not entirely free from phenol as substances for operating motors as the easily soluble phenols which act on metal have already been removed.

40 It is, of course, immaterial whether the washing is commenced with dilute alcohol of the above mentioned concentration or whether concentrated alcohol is first added and then brought to the desired dilution.

45 The term, "crude coal tar" or "crude tar" in

the present specification means coal tar obtained by low temperature distillation.

Having now particularly described and ascertained the nature of the said invention and in what manner the same is to be performed, I declare that what I claim is:— 50

1. A process for separating the phenols and neutral oils in crude or distilled coal tar, consisting in extracting the phenols by means of aqueous alcohol of a strength not greater than 60% by weight. 55

2. A process for separating the phenols and neutral oils in crude or distilled coal tar, consisting in extracting the phenols by means of aqueous alcohol of a strength not greater than 60% by weight, the extraction being effected with the aid of heating. 60

3. A process for separating the phenols and neutral oils in crude or distilled coal tar, consisting in extracting the phenols by means of aqueous alcohol of a strength not greater than 60% by weight, the extraction of the whole of the phenols being effected by a single charge of alcohol. 65

4. A process for separating the phenols and neutral oils in crude or distilled coal tar, consisting in extracting the phenols by means of aqueous alcohol of a strength not greater than 60% by weight, the alcoholic phenol solution being cooled before the alcohol is distilled off to yield a purer phenol. 70 75

5. A process for separating the phenols and neutral oils in crude or distilled coal tar, consisting in extracting the phenols by means of aqueous alcohol of a strength not greater than 60% by weight, the alcoholic phenol solution being washed with small quantities of dilute alcohol and then with water, preferably at a raised temperature, in order to free the alcoholic phenol solution from any neutral oils which may have dissolved. 80 85

In testimony whereof I have signed my name to this specification.

ANTON WEINDEL.