This invention relates to a process of refining tar acids. More specifically it relates to a process for the removal of the objectionable odor possessed by commercial tar acids, such as phenols, cresols, xylenols, and the like.

Tar acids as normally produced, as from coal tar or from petroleum, possess a disagreeable and for some purposes an objectionable odor. The exact nature of the impurities causing the odor is not known. There is no doubt, however, that some of the odor-producing impurities are sulfur-containing compounds, such as thiophenol, thiochresols, mercaptans, and organic sulfides.

It is the object of this invention to produce tar acids—phenols, cresols, xylenols, and the like—having no objectionable or disagreeable odors.

We have found that tar acids free from objectionable and disagreeable odors can be produced by refluxing the crude material with a small proportion of alkali metals, alkali metal oxides, or alkali metal hydroxides, at a higher pressure, of the order of atmospheric pressure, and then distilling the refluxed acids under reduced pressure, by which we mean at less than atmospheric pressure.

Refluxing of the crude tar acids with the alkali metals or alkali metal oxides or hydroxides at atmospheric pressure followed by distillation at atmospheric pressure does not give satisfactory results. Distilling the crude tar acids under a high vacuum without previously refluxing them at a higher pressure with alkali metals, oxides, or hydroxides does not give satisfactory results. It is only by first refluxing the crude acids with the alkali metals, oxides, or hydroxides at a higher pressure, or heating the crude acids with the alkali-metals, oxides, or hydroxides to a temperature at which refluxing occurs at atmospheric pressure but avoiding actual refluxing by using a pressure sufficiently high to prevent boiling, and then distilling them under reduced pressure, that tar acids of good odor can be obtained.

The examples given below illustrate the manner in which our invention may be practiced.

**Example 1.**—Two hundred grams of crude tar phenol is heated to about 100° C., and 0.75-1.5 grams of metallic sodium is added. The temperature of the phenol is then gradually raised. When the sodium has completely dissolved in the phenol the solution is refluxed for approximately four to six hours, at or near atmospheric pressure. After this reflux period the phenol is distilled under reduced pressure (10 mm. to 70 mm. of mercury).

The resulting phenol has a good, clean, sweet, non-sulfur odor.

**Example 2.**—Two hundred fifty grams of crude cresylic acid is heated to about 100° C., and 1.5 to 2.5 grams of metallic sodium is added. After the sodium has dissolved in the cresylic acid the solution is refluxed for five to seven hours, at a pressure of 700 to 800 mm. of mercury. At the conclusion of the reflux period the cresol is distilled under vacuum (desirably below 70 mm. mercury pressure).

The resulting cresylic acid has a good, clean, sweet, non-sulfur odor.

**Example 3.**—To 275 grams of dry, crude, undistilled coal tar acid, (the whole acid, a mixture of many components,) is added from 0.50 gram to 1.22 grams of metallic sodium. The acid is warmed to about 100° C. either before, during, or after the addition of the sodium. When the metallic sodium has dissolved, the solution is refluxed for five to eight hours. At the end of the reflux period the acid is distilled under a reduced pressure (10 mm. to 70 mm. mercury). The first 80% of acid recovered by the vacuum distillation has a clean, sweet, non-sulfur odor. The last part of the distillate (15% or so) possesses a very slight sulfur odor.

Instead of sodium in the procedure of the foregoing examples we can use potassium or lithium; or we can use solid sodium or potassium hydroxide or oxide.

The above examples are merely illustrative of the way in which our invention may be practiced. They are not to be construed to limit our invention in any way.

In the examples given above the preferred pressure for the vacuum distillation is indicated as from 10 to 70 mm. of mercury. But we are not restricted to operating between these pressure limits. We have found that good results can be obtained when the vacuum distillation is carried out at somewhat higher pressures, such as 100 mm. of mercury. It has been our experience, however, that the lower the pressure under which the distillation is carried out the better the results. We, therefore, prefer to perform the vacuum distillation at as low a pressure as is feasible.

Similarly, in these examples we have indicated that the refluxing with alkali is done at or near atmospheric pressure. The refluxing pressure may be somewhat higher or lower than that; but should not be high enough to cause
material decomposition of the tar acids, as indicated by the formation of tarry deposits during the vacuum-distilling operation, nor low enough to cause failure of the reaction with the alkali. The desirable range of refluxing pressure, and so of refluxing temperature, varies with the tar acid being treated, but for best results the refluxing pressure is fairly close to atmospheric pressure.

Instead of refluxing as described in the examples, we may heat the tar acids with the alkali metal or oxide or hydroxide to about the temperature at which refluxing occurs at atmospheric pressure but avoid actual refluxing by using a pressure sufficiently high to prevent boiling.

We claim as our invention:

1. The method of purifying tar acids, which comprises refluxing the tar acids at or near atmospheric pressure with an alkaline substance of the class consisting of alkali metals, alkali-metal oxides, and alkali-metal hydroxides, with retention of substantially all the tar acids, and then distilling off the tar acids under less than atmospheric pressure.

2. The method of purifying tar acids, which comprises heating the tar acids with an alkaline substance of the class consisting of alkali metals, alkali-metal oxides, and alkali-metal hydroxides, to about the temperature at which refluxing of the tar acids occurs at atmospheric pressure, with retention of substantially all the tar acids, and then distilling off the tar acids under less than atmospheric pressure.

3. The method of purifying tar acids, which comprises refluxing the tar acids at or near atmospheric pressure with an alkaline substance of the class consisting of alkali metals, alkali-metal oxides, and alkali-metal hydroxides, with retention of substantially all the tar acids, and then distilling off the tar acids under a pressure of not more than 70 mm. of mercury.

4. The method of purifying tar acids, which comprises heating the tar acids with an alkaline substance of the class consisting of alkali metals, alkali-metal oxides, and alkali-metal hydroxides, to about the temperature at which refluxing of the tar acids occurs at atmospheric pressure, with retention of substantially all the tar acids, and then distilling off the tar acids under a pressure of not more than 70 mm. of mercury.

5. The process of purifying tar acids as set forth in claim 1, in which the tar acid is phenol.

6. The process of purifying tar acids as set forth in claim 2, in which the tar acid is phenol.

7. The process of purifying tar acids as set forth in claim 3, in which the tar acid is phenol.

8. The process of purifying tar acids as set forth in claim 4, in which the tar acid is phenol.

9. The process of purifying tar acids as set forth in claim 1, in which the tar acid is cresylic acid.

10. The process of purifying tar acids as set forth in claim 2, in which the tar acid is cresylic acid.

11. The process of purifying tar acids as set forth in claim 3, in which the tar acid is cresylic acid.

12. The process of purifying tar acids as set forth in claim 4, in which the tar acid is cresylic acid.

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