PATENT SPECIFICATION

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1367

PROVISIONAL SPECIFICATION

Improvements in the Manufacture and Production of Oxidation Products from Mixtures Containing Paraffin Waxes

I, GEORGE WILLIAM JOHNSON, a British Subject, of 47, Lincoln's Inn Fields, in the County of London, Gentleman, do hereby declare the nature of this invention (which has been communicated to me from abroad by I. G. Farbenindustrie Aktiengesellschaft, of Frankfort-on-Main, Germany, a Joint Stock Company organised under the Laws of Germany) to be as follows:—

My foreign correspondents have found that for the production of oxidation products of paraffin hydrocarbons those paraffin products are especially suitable which have been recovered by treatment with nitrobenzene or other aromatic nitro-hydrocarbons or mixtures containing the same from crude mixtures containing paraffin waxes which contain, in addition to the paraffin waxes, olefines, aliphatic compounds of low molecular weight and aromatic or naphthenic constituents. In the said manner the paraffin waxes are separated from the crude initial materials. Nitrogenous oxidising agents, in particular nitric acid or nitrous gases, are suitable for the oxidation.

Initial materials suitable for the process according to this invention are for example tar products containing paraffin waxes, such as brown coal tars or hydrogenation products of coals, tars and the like. The said initial materials are mixed for example with about an equal or greater or smaller amount of nitrobenzene, preferably while heating slightly, as for example to 50° Centigrade, stirred well, cooled to a low temperature, as for example 15° Centigrade, and allow to stand for some time. The nitrobenzene solution is then separated from the deposited paraffin waxes, the latter, which usually still contain considerable amounts of nitrobenzene, then being subjected to oxidation. In many cases it is preferable to free the paraffin waxes containing nitrobenzene from the latter before the oxidation, as for example by heating or by treatment with steam or the like.

Dinitrobenzenes, chlornitrobenzenes or solvent mixtures, as for example mixtures of nitrobenzene and chlornitrobenzene, may also be used instead of nitrobenzene.

The oxidation is effected in known manner, as for example by heating the paraffin waxes obtained with concentrated nitric acid, as for example nitric acid containing 45 per cent. of HNO_3, or by treating with nitrous gases. Generally speaking the oxidation is carried out while heating slightly, as for example to from about 50° to 80° Centigrade. After the oxidation, the resulting product is washed with water and freed from combined nitrogen, as for example by heating with alkalies. In this way oxidation products of a high degree of purity are obtained which may be used with advantage as initial materials for the preparation of soaps or assistants for the textile and related industries.

The following Example will further illustrate the nature of this invention but the invention is not restricted to this Example. The parts are by weight.

EXAMPLE.

10 parts of nitrobenzene are added at about 50° Centigrade to 10 parts of dehydrated brown coal tar and the whole stirred well. The mixture is then cooled to 15° Centigrade; after some time the nitrobenzene solution separates from the deposited paraffin wax. The crude paraffin wax thus obtained has a good crystalline form and may readily be filtered; it still has a high content of nitrobenzene. It is then treated at about 80° Centigrade with four times the amount of 45 per cent. nitric acid calculated with reference to the paraffin wax free from nitrobenzene, until a sample of the product free from acid and nitrobenzene has a saponification value between 100 and 200. The reaction mixture is then allowed to stand until a separation into two layers takes place, of which one layer consists of nitric acid and the other of a nitrobenzene solution of the oxidation product formed. After separating the layers, the nitrobenzene solution is treated with water. The nitrobenzene is then distilled off, preferably

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under reduced pressure. A paraffin wax oxidation product containing nitrogen is thus obtained which is worked up in the usual manner and from which fatty acids especially suitable for the preparation of soaps may be obtained.

If the oxidation be carried further, for example so that the product has a saponification value of more than 200, as for example 300, there may be recovered from the oxidation product not only monocarboxylic acids but also considerable amounts of dicarboxylic acids.

The crude paraffin wax used for the oxidation may also be freed from combined nitrobenzene, as for example by treatment with steam. On the other hand in many cases it is advantageous to add further amounts of nitrobenzene to the crude paraffin wax before the oxidation; in this way the course of the reaction may be rendered milder.

Dated this 7th day of October, 1936.
J. Y. & G. W. JOHNSON,
47, Lincoln's Inn Fields,
London, W.C.2,
Agents.

COMPLETE SPECIFICATION

Improvements in the Manufacture and Production of Oxidation Products from Mixtures Containing Paraffin Waxes

I, GEORGE WILLIAM JOHNSON, a British Subject, of 47, Lincoln's Inn Fields, in the County of London, Gentleman, do hereby declare the nature of this invention (which has been communicated to me from abroad by I. G. Farbenindustrie Aktiengesellschaft, of Frankfort-on-Main, Germany, a Joint Stock Company organised under the Laws of Germany) and in what manner the same is to be performed to be particularly described and ascertained in and by the following statement:—

My foreign correspondents have found that technically valuable products can be obtained by dissolving products containing considerable amounts of high-molecular solid paraffin hydrocarbons besides tar-like substances, olefines, aliphatic compounds of low molecular weight and aromatic or naphthenic constituents, in normally liquid aromatic nitro-hydrocarbons or mixtures containing the same, cooling the solutions obtained, separating the solid paraffin hydrocarbons which have been precipitated, and oxidising the latter while using nitrogenous oxidising agents.

Initial materials suitable for the process according to this invention are for example tar products containing paraffin waxes, such as brown coal tars, or hydrogenation products of coals, tars and the like. Suitable solvents are for example nitrobenzene, or mixtures containing it, as for example mixtures of nitrobenzene and chlornitrobenzenes. The said initial materials are mixed for example with about an equal or greater or smaller amount of nitrobenzene, preferably while heating slightly, as for example to 50° Centigrade, stirred well, cooled to a low temperature, as for example 15° Centigrade, and allowed to stand for some time. The nitrobenzene solution is then separated from the deposited paraffin waxes, the latter, which usually still contain considerable amounts of nitrobenzene, then being subjected to oxidation. In many cases it is preferable to free the paraffin waxes containing nitrobenzene from the latter before the oxidation with nitrogenous oxidising agents, as for example by heating or by treatment with steam or the like. It may also be advantageous to subject the solid paraffin hydrocarbons separated off in the manner described above which still contain part of the solvent employed for a second and if desired a third time to the same treatment while employing the same or another solvent or mixture of solvents of the type hereinbefore defined. In this manner high-molecular paraffin hydrocarbons of a higher degree of purity are obtained than by a single treatment.

The oxidation is effected in known manner, by heating the paraffin hydrocarbons with nitrogenous oxidising agents, in particular nitric acid or nitrous gases. It is preferable to work while using concentrated nitric acid, as for example nitric acid containing 45 per cent. of HNO_3. Generally speaking the oxidation is carried out while heating slightly, as for example to from about 50° to 100° Centigrade. After the oxidation the resulting product is separated from the nitric acid and then freed from adhering nitric acid by washing with water or warming. If desired the oxidation product may also be freed from organic nitrogen compounds for example by treating with alkaline reacting substances. In this way oxidation products of a high degree of purity are obtained which may be used with advantage as initial materials for the preparation of soaps or

assistants for the textile and related industries.

The following Examples will further illustrate how the said invention may be carried out in practice but the invention is not restricted to these Examples. The parts are by weight.

EXAMPLE 1.

10 parts of nitrobenzene are added at about 50° Centigrade to 10 parts of dehydrated brown coal tar and the whole stirred well. The mixture is then cooled to 15° Centigrade; after some time the nitrobenzene solution separates from the deposited paraffin wax. The crude paraffin wax thus obtained has a good crystalline form and may readily be filtered; it still has a high content of nitrobenzene. It is then treated at about 30° Centigrade with four times the amount of 45 per cent. nitric acid calculated with reference to the paraffin wax free from nitrobenzene, until a sample of the product freed from acid and nitrobenzene has a saponification value between 100 and 200. The reaction mixture is then allowed to stand until a separation into two layers takes place, of which one layer consists of nitric acid and the other of a nitrobenzene solution of the oxidation product formed. After separating the layers, the nitrobenzene solution is treated with water. The nitrobenzene is then distilled off, preferably under reduced pressure. A paraffin wax oxidation product containing nitrogen is thus obtained which is worked up in the usual manner and from which fatty acids especially suitable for the preparation of soaps may be obtained.

If the oxidation be carried further, for example so that the product has a saponification value of more than 200, as for example 300, there may be recovered from the oxidation product not only monocarboxylic acids but also considerable amounts of dicarboxylic acids.

The crude paraffin wax used for the oxidation may also be freed from admixed nitrobenzene, as for example by treatment with steam. On the other hand in many cases it is advantageous to add further amounts of nitrobenzene to the crude paraffin wax before the oxidation; in this way the course of the reaction may be rendered milder.

EXAMPLE 2.

100 parts of nitrobenzene are added to 100 parts of brown coal tar at 50° Centigrade. The solution obtained is cooled down to 15° Centigrade while stirring. After some time the paraffin hydrocarbons precipitated are separated from the nitrobenzene solution by filtration. Thus 33 parts of solid paraffin hydrocarbons containing nitro-benzene are obtained which are then subjected to a second purification treatment with 66 parts of nitrobenzene at 50° Centigrade. The solution is then cooled down to 20° Centigrade while stirring and the paraffin hydrocarbons precipitated are again separated by filtration. After distilling off the adhering nitrobenzene from the paraffin hydrocarbons under reduced pressure, the paraffin hydrocarbons are subjected to the action of nitric acid of 60 per cent. strength at 90° Centigrade until a sample of the oxidation product has a saponification value greater than 200. After standing for some time the mixture separates into two layers, one of which consists of the oxidation product, the other of nitric acid. The layers are separated. The oxidation product is then freed from adhering nitric acid by heating up to 120° Centigrade. It can be worked up in usual manner for recovering fatty acids which are particularly suitable for the preparation of soaps.

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

1. The process for the manufacture and production of oxidation products of paraffin hydrocarbons, which comprises dissolving products containing considerable amounts of high molecular solid paraffin hydrocarbons besides tar-like substances, olefines, aliphatic compounds of low molecular weight and aromatic or naphthenic constituents in normally liquid aromatic nitro-hydrocarbons or mixtures containing the same, cooling the solutions obtained, separating the solid paraffin hydrocarbons which have been precipitated and oxidising the latter while using nitrogenous oxidising agents.

2. In the process claimed in claim 1, subjecting the high molecular solid paraffin hydrocarbons obtained by treating the crude product with aromatic nitro-hydrocarbons or mixtures containing the same for a second or third time to a similar treatment prior to the oxidation.

3. The process for the manufacture and production of oxidation products substantially as described in each of the foregoing Examples.

4. Oxidation products when obtained according to the process particularly described and ascertained or its obvious chemical equivalents.

Dated this 10th day of September, 1937.

J. Y. & G. W. JOHNSON,
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