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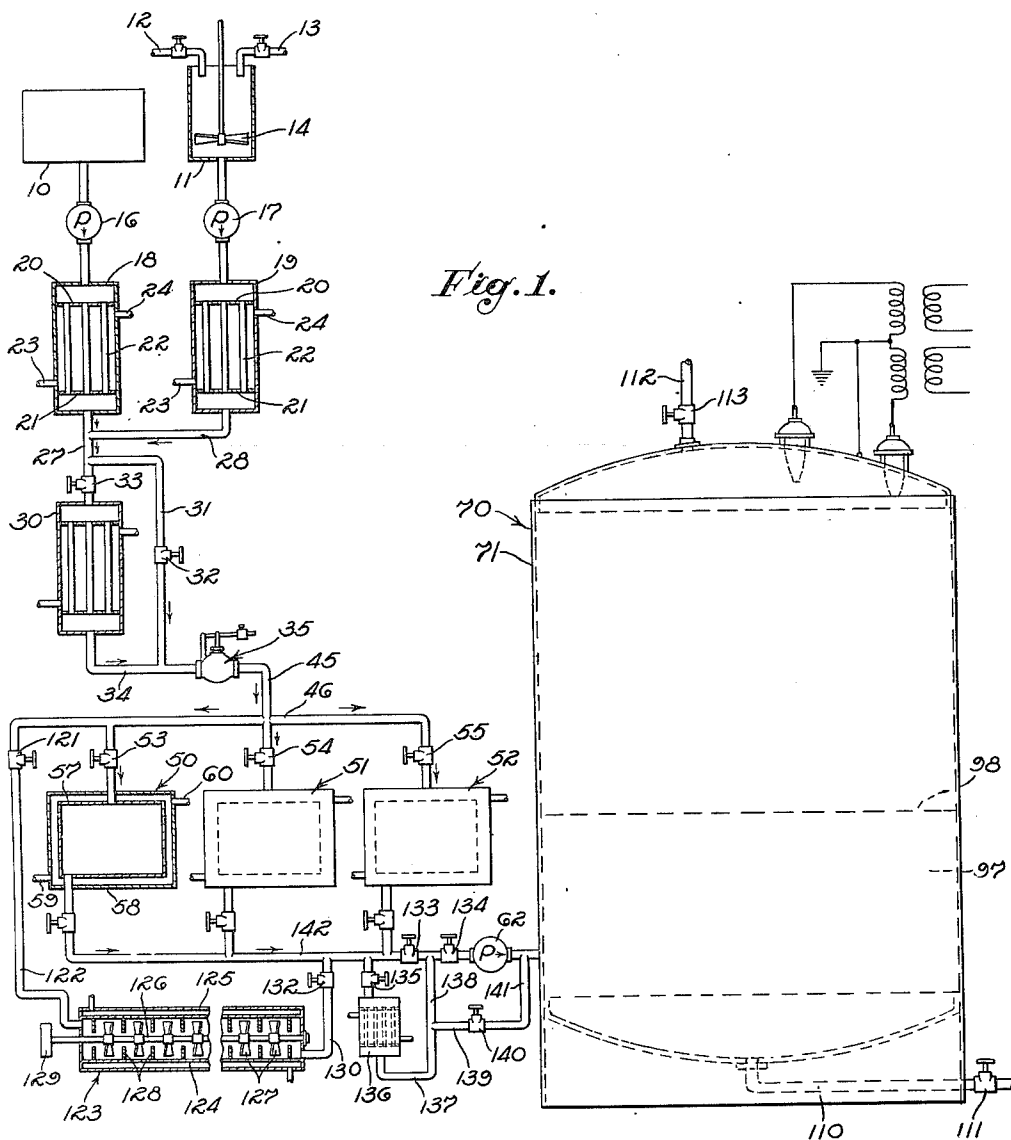
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2,447,529

METHOD FOR REMOVING IMPURITIES FROM HYDROCARBONS

Filed April 5, 1938

2 Sheets-Sheet 1



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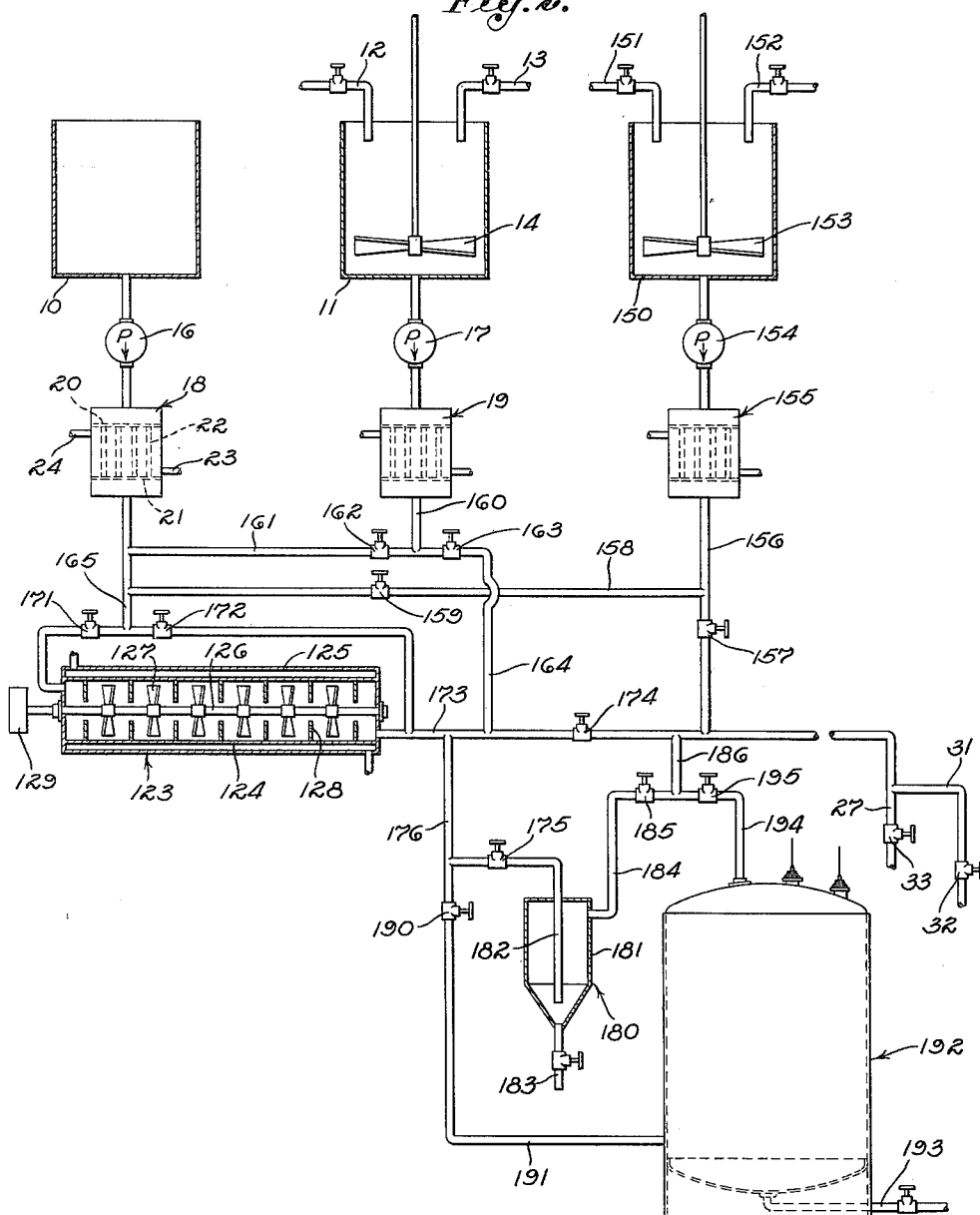
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2 Sheets-Sheet 2

Fig. 2.



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UNITED STATES PATENT OFFICE

2,447,529

METHOD FOR REMOVING IMPURITIES
FROM HYDROCARBONS

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19 Claims. (Cl. 204—190)

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My invention relates to an electrical process and apparatus for removing impurities from oils and is particularly adapted to the purification of hydrocarbon oils before or after distillation.

Oils, such as petroleum or its distillates, contain various impurities which are more or less susceptible to the action of chemical refining agents. In many instances, however, it is difficult to obtain satisfactory contact between the refining agent and the impurities, or to obtain a sufficient degree of reaction to effect substantial purification. Again, in many instances, it is difficult to separate the reacted impurities and/or the unconsumed reagent from the treated oil. The present invention is designed to overcome these difficulties and remove various impurities from oils.

In the present specification, the following definitions of general terms will be used: By "hydrocarbon oils," I have reference to oils which are composed predominantly of hydrocarbons, such as crude petroleum, coal tar, shale oil, and the like, or fractions thereof, which are fluid at the temperature of treatment. By "refining agent," I have reference to a chemical agent adapted to react with or modify the impurities in the oil. By "reacted impurities," I have reference to materials which separate from the oil, or which are caused to separate in my process, by virtue of the action of the refining agent on the impurities originally present in the oil. By the term "gathering agent," I have reference to a liquid present, as a dispersed phase in the oil, at the time of electric treatment.

General objects of the invention are as follows:

It is an object of my invention to provide a process and apparatus for the chemical refining of oils whereby impurities therein are subjected to the action of refining agents under controlled conditions leading to the desired degree of contact and reaction.

Another object of my invention is to provide a process and apparatus for electrically refining oils in which oils are subjected to the action of an electric field subsequent to their mixture with a chemical refining agent, whereby a separation of the treated oil from the refining agent and/or the reacted impurities is induced, and whereby some further refining action may take place.

Another object of my invention is to provide a process and apparatus for electrically refining oils in which a gathering or collecting agent is dispersed into the oil prior to electric treatment and, if desired, at the same time that the refining agent is added to the oil, whereby reacted impurities are gathered or associated with the dispersed phase in such manner as to be eliminated therewith when the mixture or emulsion is sub-

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jected to electrical treatment to resolve the dispersion.

It is, furthermore, an object of the invention to provide such a process in which the oil is maintained in contact with the dispersed reagent and/or gathering agent during a conditioning period prior to electrical treatment, whereby a conditioning effect is obtained which may be beneficial both in facilitating the subsequent electric treatment and in increasing the percentage removal of impurities.

Still another important object of the invention is to remove corrosive materials from an oil by electric treatment, for example hydrogen sulphide, or materials which are potential sources of hydrogen sulphide or other corrosive substances when subjected to heat. For the purpose of this specification, I have grouped, under the term "sulphur-containing substances," both hydrogen sulphide and those potential sources of hydrogen sulphide which, upon heating, will liberate this objectionable compound.

Another object of the invention is to treat an oil to remove sulphur-containing substances. Generally stated, it is an object of my invention to decrease the sulphur content of oils and/or distillates.

More specific objects of the invention will be apparent from the following general statement of the invention and the subsequent detailed illustrations and examples set forth.

In general, the invention resides in treating an impure oil with a chemical refining agent adapted to react with the impurities in the oil, maintaining such conditions of contact as give the desired degree of reaction, and then subjecting the reaction mixture to the action of an electric field, preferably of an alternating high potential type, the reaction mixture as subjected to the field being in the form of an oil-continuous emulsion containing a dispersed phase comprising or adapted to collect reacted impurities and/or the spent or remaining refining agent.

The present process and apparatus are adapted to the removal of various types of impurities from oils, either before or after distillation, and the character of the refining agent is chosen to correspond with the impurity which it is desired to remove. For example, in removing acidic or saponifiable impurities, I preferably use an alkaline reagent, such as caustic alkali, alkaline earth oxides or hydroxides, alkali carbonate, alkali phosphate, solutions (usually aqueous and/or alcoholic) of caustic alkali and the like, etc. Impurities of an unsaturated or resinous character can be removed, according to my process, by the use of reagents having a polymerizing or precipitating action on the impurities, such as strong acids, for example sulphuric acid; see the subject matter claimed in my copending application Se-

rial No. 312,222. Aqueous solutions of various materials may be used as refining agents in my process, for example, solutions of NaOCl, the halogen acids, phosphoric acid, nitric acid, and the like. In general, I propose to use refining agents known in the art to react with and remove or modify impurities which are objectionable and, for the most part, my invention is not concerned solely with the character of the reagent but rather with the mode of its use.

My process is of particular value in treating crude oils prior to distillation, and I find that pre-refining, as practiced according to my invention, is advantageous in many respects, among which are the suppression of corrosion of metallic equipment in contact with the crude oil, for instance, the crude pipe lines and, in particular, the stills in which the crude petroleum is distilled into fractions.

The present process can be used to remove present or potential hydrogen sulphide by treatment of the crude stocks before distillation. In other instances, the process can be advantageously used to remove hydrogen sulphide or materials engendering hydrogen sulphide from a hydrocarbon which has previously been subjected to fractionation, for example, one or more of the various fractions obtained from a distillation process.

My process is also adapted to decrease the sulphur content of oils generally, without particular reference to the specific type of sulphur-bearing material which is removed. Desulphurizing agents, in general, comprise alkalis, acids, and the like.

In addition, some crude petroleum stocks and some distillates produced therefrom contain various acidic materials which are detrimental to the quality of the distillates, and which may also give rise to corrosion difficulties during distillation. Examples of such materials are naphthenic acids, phenols, and saponifiable complexes yielding such acids on distillation or saponification. Such acidic or acid-engendering materials may be reacted and removed by my process, preferably by the use of an alkaline reagent. Here again, the process is applicable either to a crude oil or its distillates, and I am able to obtain from crude oils which have been so treated distillates which contain materially smaller quantities of acidic material than would otherwise be the case.

In some instances, I prefer to perform the process by use of the following steps: first, contacting the impure oil with a reagent adapted to react upon or transform the impurities present in the oil, e. g., dissolved therein; second, dispersing in the oil a phase adapted to collect the reacted impurities and/or the spent or unused reagent, e. g., a gathering agent; third, providing a sufficient conditioning period during which the character of the dispersion may be modified and during which further reaction with impurities may take place; fourth, subjection of the treated oil containing the dispersed gathering phase to the action of an electric field, preferably a high potential alternating field; fifth, separating the purified oil from the gathering phase and the impurities associated therewith.

Various of these steps may be combined into one step, or their order may be changed, or one or more steps may be omitted entirely, as hereinafter set forth. In many instances, I find it advantageous to combine the first two steps. For example, in treating an oil to remove acidic and acid-engendering compounds, I may use a

caustic alkali as my reagent and water as my collecting agent, and add them simultaneously to the oil in the form of an aqueous solution of caustic alkali. This aqueous solution is dispersed in the oil, preferably under conditions which are favorable to the formation of an oil-continuous emulsion, for example, at temperatures not substantially above atmospheric temperature. Some or all of the desired reactions may take place in this step. This dispersion may then be conditioned by maintaining it, usually at an elevated temperature, either in a quiescent state or with agitation for a period sufficient to condition it for electric treatment, usually for a period of an hour or more. During this conditioning period, additional desired reactions may take place, namely, reactions of the reagent upon the impurities and the conditioning of the emulsion for subsequent steps.

As an alternative to the process just described, I may add the reagent directly to the oil. For example, in the case just illustrated, I might have added a very concentrated solution of caustic alkali to the oil, preferably with sufficient mixing to insure good contact, and might have thereafter dispersed a gathering agent, such as water, in the oil thus commingled with the concentrated caustic alkali. Again, I may add an oil-soluble, or partially oil-soluble, reagent to the oil, for example, an alcoholic solution of caustic alkali, and thereafter disperse water in the oil thus treated. Various other methods can be used for adding the refining agent and the gathering agent to the oil.

In some instances, it may be advantageous to reverse the sequence of the first steps mentioned above. Thus, the water may be added to the oil and dispersed therein, an emulsifying agent being added or present, if desired. The emulsion thus formed may then receive the refining agent.

In some instances, I may partially separate the impurities before proceeding to the final separating stage. Thus, I may add alcoholic KOH containing sufficient alcohol of an appropriate strength to form a dispersed alcoholic phase. All or a portion of this phase may be then separated from the oil as by an electrical process, or by other means, and the oil thus treated then commingled with water to form a water-disperse emulsion which is then subjected to the further steps of separation. This mode of operation is of particular advantage when it is desired to avoid any return of impurities to the oil by the diluting effect of the water. Thus, high molecular weight alkali reacted mercaptans, in the example illustrated, are removed with the alcohol due to their solubility in the concentrated alcohol and are thus not subjected to the precipitating and/or hydrolyzing action of the water.

The gathering or collecting agent is a material which is either immiscible with the oil or, at least, sufficiently immiscible to form a separate second phase in the presence of the oil. It is, furthermore, a material which is adapted either to dissolve the reacted impurities or to associate with the reacted impurities in such a manner, for example, by adsorption of these impurities at the interface, as to remove the reacted impurities from the treated oil in the final step of separation. It is not necessary, however, that all of the impurities be removed in this fashion, for some of the impurities may have been merely modified by the reaction into inoffensive oil-soluble compounds which still remain in the oil,

As stated above, by reacted impurities I mean those impurities which have been so changed that they dissolve in, or become associated with, the gathering or collecting agent. The gathering agent may furthermore aid in the separation of the spent reagent from the treated oil. Water and alcohol have been illustrated as suitable gathering or collecting agents, but I do not wish to be limited to these materials.

In many instances, the gathering or collecting agent will form a suitable oil-continuous emulsion with the oil. In other instances, I find it advantageous to add a modifying agent to insure the formation and suitable stability of the desired oil-continuous emulsion. Such modifying agent may be any of the well known class of emulsifying agents which promote the formation of oil-continuous emulsions, for example, alkaline earth soap and the like. In still other instances, I may keep the disperse phase suspended by continuous agitation or turbulence until the dispersion is subjected to the action of the electrical field or other separatory means, and thereby make use of dispersions which are so unstable as to rapidly separate on standing.

In operating on crude oils, I usually find it unnecessary to add any emulsifying agent of the type described. On distillates, however, and in particular the lighter distillates, it is sometimes impossible to obtain a satisfactory emulsion without the aid of an emulsifying agent of the type described. In the case of heavy lubricating oil distillates or of long residuums, there are frequently present sufficient amounts of naturally occurring asphaltic bodies to aid in the formation of the desired emulsion. I may, if desired, however, add a small amount of an emulsifying material to a distillate or lubricating oil distillate in order to bring this effect into greater prominence.

The conditioning step, wherein the dispersion of the gathering agent in the oil is allowed to age, and wherein further reaction between the refining agent and the impurities may take place, is an advantageous and, in many instances, a necessary step, although under certain circumstances it may be omitted or shortened to merely a nominal period. During this period, a change in the character of the emulsion usually takes place, the mechanism of which is obscure and not understood, the first effect of which is usually to increase somewhat the ease with which the emulsion may be treated or broken by the action of the electrical field.

The second conditioning effect which involves further reaction of the reagent with impurities, may or may not be of importance, depending upon the character of the refining agent used and on the character of the impurities to be removed. When a rapidly reacting reagent has been used, the additional refining effects, as induced by the conditioning period, are small or even negligible. However, in certain other instances, the desired effects are obtained only in conjunction with the conditioning period. For example, in treating a crude petroleum with an aqueous solution of caustic alkali comprising, if desired, an alkali disulphide, the refining effects of the reagent on the sulphurous material comprising or forming potential sources of hydrogen sulphide (including sulphur and sulphur compounds, such as mercaptans, disulphides, and the like) are usually realized to a suitable extent only if the emulsion is maintained for a period of an

hour or more and preferably at an elevated temperature, for example, from 120 to 180° F.

In separating the components of the conditioned emulsion so as to obtain the purified oil on the one hand and the reacted impurities and gathering agent on the other, I prefer to employ the action of a coalescing electric field, preferably a high potential alternating field. Among other things, this field produces a separation or resolution of the components of the emulsion, or it at least so changes the character of the emulsion, as by partial coalescence of the droplets, that the components thereafter rapidly separate by gravity. In addition to these effects, however, I find further unexpected effects arising from the action of an electrical field which make it particularly applicable to the separation of the emulsions as produced by my process. These effects, which are not well understood in their character or mechanism, are manifested by the greater degree of purification which is usually obtained when the electrical field is used than when other methods of separation are relied upon.

Various explanations may be advanced for the beneficial effect of the electrical field. There is some evidence tending to show that an electrical field is more selective in its action on very small droplets highly charged with impurities than are other methods of separation. On the one hand, the smallest droplets are most highly charged with impurities because of their high surface-to-volume ratio which results in the small drops producing a greater amount of interfacial reaction with respect to their quota of reagent than is produced by the larger drops. On the other hand, the smallest droplets are those most difficult to separate when gravitational methods of separation are relied upon solely, as in the case of separating by settling or by centrifuging. In ordinary methods of separation, therefore, these small droplets, which are heavily charged with impurities, are separated with difficulty or not at all. In using an electrical field, however, I find that these small impure droplets are very substantially removed from the oil.

Moreover, the effect does not seem to be one which can be explained merely upon the basis of a greater degree of resolution, since, in many instances, I find that, when the emulsion is separated to the same extent, i. e., to produce a purified oil of the same water content, by an electrical process on the one hand and a gravitational or centrifugal process on the other, the emulsion treated by the electrical field will be found to contain less impurities than the emulsion of the same water content separated by gravitational or centrifugal separation. This may arise from a selective tendency of the very small impure droplets to coalesce with larger droplets under the action of the electric field so that whatever droplets of water are retained in the emulsion are substantially less concentrated in impurities than those selectively acted upon.

Referring to the drawing,

Fig. 1 illustrates a pipeline diagram of a system embodying my invention, with the electric treatment shown in elevation.

Fig. 2 illustrates a diagram alternative to part of Fig. 1.

Fig. 1 is illustrative of a flow sheet adapted to the use of an aqueous reagent capable of serving as a gathering agent. Referring particularly to Fig. 1, the numeral 10 indicates a tank for storage of the hydrocarbon, and 11 indicates a tank containing the aqueous reagent. If desired, this

reagent can be mixed in the tank 11 by introducing proportioned quantities of water and chemical through pipes 12 and 13 and mixing these by any suitable means, such as an agitator 14.

Pumps 16 and 17 respectively pump the hydrocarbon and reagent from tanks 10 and 11 and may develop sufficient pressure to force these constituents into or through the conditioning means to be hereinafter described.

Heaters 18 and 19 may be used to heat the liquids before mixing if such heating is desirable. Various types of heaters can be used, the ones shown being of the tube type provided with headers 20 and 21 between which extend pipes 22 for carrying the liquid to be heated. Steam or other heating medium can be circulated through the space between the headers and around the tubes 22, being introduced, for instance, through a pipe 23 and withdrawn through a pipe 24.

The hydrocarbon moves from the heater 18 through a pipe 27, and the aqueous reagent may be introduced into a flowing stream of this hydrocarbon through a pipe 28. A preliminary mixture is formed when these liquids are thus brought together.

If desired, this preliminary mixture can be further heated by moving through a heater 30 similar to the heaters 18 and 19 previously described. Alternatively, it can by-pass this heater flowing through a pipe 31 when a valve 32 thereof is opened and a valve 33 in the pipe 27 is closed. During flow through such a heater 30, some additional mixing may take place due to turbulent flow conditions therein.

To further mix the hydrocarbon and aqueous reagent, the preliminary mixture may move from the heater 30 by means of a pipe 34 through an emulsifying means 35. Various types of emulsifying means can be used to form the desired type of emulsion, such as baffle mixers or spring-loaded valves. An emulsifying means which has been found particularly advantageous is illustrated and comprises a weighted valve, such as shown in detail in the patent to Harold C. Eddy, No. 2,182,145.

The resulting emulsion moves through a pipe 45 to a header 46 and thence to one or more of a plurality of conditioning means. Three of these conditioning means are indicated by the numerals 50, 51, and 52, and the resulting emulsion can be moved into one or more of these by proper control of valves 53, 54, and 55.

The simplest form of conditioning means includes a tank 57 into which the resulting emulsion is discharged and in which this emulsion is permitted to stand for the desired conditioning period. The conditioning means 50, 51, and 52 may be supplied with suitable stirring means, not shown, which can be continuously operated or put into operation just before the conditioned emulsion is withdrawn. However, such a stirring means is not always essential to the process.

It is often desirable that the emulsion be conditioned at elevated temperature. To prevent excessive cooling of the emulsion during conditioning, or to add additional heat thereto, a jacket 58 may be provided around each tank 57. A hot medium may be circulated therethrough by use of pipes 59 and 60. In other instances, the heat initially in the resulting mixture may be conserved by heat-insulating each of the tanks 57. In other instances, such heating or heat-insulating may be dispensed with, allowing the mixture to cool slightly during the conditioning step.

Various other conditioning means may be employed instead of the illustrated system of tanks. For example, the conditioning means may comprise an elongated tank with an intake connection at one end and a discharge connection at the other end, of such a capacity that the emulsion in its flow therethrough is properly conditioned; i. e., the time consumed in the travel through the tank corresponds to the desired conditioning period. If desired, such a tank may be provided with stirrers or other agitating means, preferably used in connection with baffles designed to prevent indiscriminate mixing of conditioned and unconditioned emulsion. Such a tank may be elongated either vertically or horizontally, and, if desired, a plurality of such tanks may be used, either in series or in parallel.

A horizontally elongated tank serving as a conditioning means is shown in conditioner 123. This conditioner comprises a horizontally extended inner pipe 124 closed at the ends and surrounded by a steam jacket 125. The inner pipe 124 is provided with a plurality of baffle means such as baffle 128 intermediate which are a plurality of paddle means such as paddle 127 rotatable by a shaft 126. The shaft extends beyond one end of the conditioner, and the extended portion is keyed to a pulley 129 whereby it may be suitably driven.

The unconditioned emulsion is brought to the conditioner 123 from the header 46 by means of valve 121 and line 122, the latter opening into one end of the interior pipe 124. The conditioned emulsion is withdrawn from the other end of the conditioner through a pipe 130 provided with a valve 132, and conducted into the line 142. The conditioner 123 is shown broken in the middle; the unindicated horizontal extension should be such as to give the conditioner a total volume substantially equal to the rate of flow of the emulsion multiplied by the desired conditioning period.

The elongation of the passageway in which conditioning takes place may, if desired, be emphasized to such a degree that a turbulent flow is occasioned by the diminished cross section of the passageway. The cross section may be chosen in such relation to the rate of flow of the emulsion that turbulent flow is induced to a degree which insures the maintenance of the emulsion or mixture in the desired state of uniformity.

The conditioned emulsion may be withdrawn from the plurality of conditioning means 50, 51, and 52 shown in the drawing in succession, or it may be simultaneously withdrawn from two or more of these means. As shown, a pump 62 may be utilized for withdrawing the conditioned emulsion from any or all of the conditioning means, as determined by the settings of the valves concerned. The pump 62 may be dispensed with in some instances, or by-passed by a valve 140, utilizing the pumps 16 and 17 for the purpose of forcing the emulsion through the conditioning means and through a pipe 67 into an electric treater 70. However, if used, the pump 62 may serve to further emulsify the constituents of the conditioned emulsion.

In some instances it is desirable to re-heat the conditioned emulsion before passing it to the electric treater, or to raise it to a higher temperature than the conditioning temperature for the purposes of electrical treatment. To do this I may close a valve 133 in the line 142 and open a valve 135 leading to a heat interchanger 136.

The conditioned emulsion is brought to the desired treating temperature therein and thence conducted by means of lines 137 and 138 to a point in the line 142 beyond the valve 133, at which point it is picked up by the action of the pump 62. If desired, the pump 62 may be by-passed by closing the valve 134 and opening the valve 140, the emulsion then passing through the line 139, valve 140, and line 141 into the line 67 entering the treater.

Various types of electric treaters can be used, the type of electric treater shown in the Eddy patent, supra, being particularly advantageous. As the details of this treater are not, per se, a part of the present invention, no detailed description will be here given. Suffice it to say that this treater includes a closed tank 71 and performs the dual functions of coalescing the dispersed water droplets in the conditioned emulsion and separating the coalesced masses to leave a purified hydrocarbon. It will be clear, however, that these functions can be performed in separate means, if desired.

As disclosed in the patent to Eddy, supra, the treater includes suitable live electrodes for establishing a main field, as well as one or more auxiliary fields, one of the auxiliary fields being bounded by the body of coalesced aqueous material, indicated by the numeral 97, which separates following electric treatment and which provides a rather definite surface, indicated by the numeral 98.

It is often desirable to use two or more emulsifying means successively. If desired, the means for discharging the emulsion into the main field may be utilized as one of these, or as supplementary thereto, to effect a final emulsification just before the emulsion is subjected to the action of an electric field.

The action of the one or more electric fields is to treat the emulsion to effect separation, e. g., to coalesce the dispersed droplets into masses distributed in the oil and of size sufficient to cause them to settle from the oil to form the body 97. The treated hydrocarbon will occupy the space in the tank 71 above the body 97.

The action of the electric field or fields is several-fold. In the first place, it may cause additional portions of the sulphur-containing materials to become associated with the aqueous reagent. In the second place, it coalesces the dispersed aqueous droplets. In the third place, this coalescing action may be particularly effective with regard to the small droplets highly charged with impurities, especially under the alternating electrostatic stresses induced by the use of an A. C. type treater. The coalesced masses thus contain sulphurous materials or other reacted impurities which were originally present in the hydrocarbon.

The coalesced aqueous material can be continuously or intermittently withdrawn from the lower end of the tank 71 through a pipe 110, as controlled by a valve 111. Similarly, the purified hydrocarbon can be continuously or intermittently withdrawn through a pipe 112 as controlled by a valve 113 and may move to suitable storage or directly to the stills if desired.

Fig. 2 illustrates a diagram which is alternative in part to Fig. 1, and is illustrative of the practice of my invention when using separate refining agents and collecting agents. The diagram of Fig. 2 is designed to replace or be alternative to that portion of the diagram in Fig. 1 which is ahead of valves 33 and 32.

Referring more particularly to Fig. 2, the tank 19 contains impure oil which is picked up by the pump 16 and passed through the heat interchanger 18 to bring it to the desired temperature. The tank 11 is a tank wherein the refining agent is compounded or stored and is provided with a paddle 14 whereby such chemicals as are introduced through the pipes 12 and 13 may be properly mixed. The reagent is picked up by the pump 17 and passed through the heat interchanger 19 wherein it is brought to the desired temperature. A tank 150 is designed to contain the collecting or gathering agent, such as water or alcohol or mixtures thereof, or any of these in combination with a modifying agent. The constituent parts of the gathering agent may be added through pipes 151 and 152 and mixed by means of a paddle 153. The gathering agent is picked up by means of a pump 154 and passed through a heat interchanger 155 to bring it to the desired temperature.

When it is desired to subject the oil to the action of the refining agent prior to the introduction of the gathering agent, a valve 162 is opened and the refining agent is brought from the heat interchanger 19 through lines 160 and 161 into the stream of oil flowing from the heat interchanger 18 through the line 165. With a valve 171 in the line 165 opened, the commingled stream of oil and reagent is brought into the conditioner 123, the structure of which may be identical with that previously described in connection with Fig. 1. The commingled streams may be passed through either with or without the application of heat and/or agitation, depending upon the conditions desired for the refining action. If desired, the conditioner 123 may be made very small in dimensions, serving under such circumstances more as an agitating means than as a conditioning means. If desired, the conditioner 123 may be by-passed entirely by opening a valve 172 and closing the valve 171.

The conditioned or by-passed dispersion of reagent in oil then enters a line 173, and, after passing a valve 174, is commingled with gathering agent introduced through a line 156 and valve 157. If desired, partial or substantial separation of the dispersed refining agent and/or reacted impurities may be caused to take place prior to introduction of the gathering agent. Thus the valve 174 may be closed and a valve 175 opened, whereby the dispersion of refining agent in oil is brought by means of a line 176 into a decanter 180 comprising a down pipe 182 for the entering emulsion and a closed shell 181 defining a settling space. The oil rising to the top of the shell 181, being more or less freed from reagent and/or reacted impurities by the settling thereof, is removed by means of a line 184 having a valve 185 into a line 186 communicating with the line 173 at a point beyond the valve 174. This partially settled emulsion is then commingled with a gathering agent as before, if desired.

If desired, a dispersion of reagent in oil may be substantially freed from reagent and/or reacted impurities prior to contacting it with the gathering agent by means such as electrical precipitation. An electric treater 192 is illustrated very schematically in Fig. 2 and may be of a type similar to that shown in the patent to Eddy, supra. By closing the valves 174 and 175 and opening a valve 190 in the line 176, the dispersion of reagent in oil is brought through a line 191 into the electric treater 192 wherein the reagent

and/or reacted impurities are caused to settle out by the action of the electric field. The treated oil is removed through a line 194 having a valve 195 into the line 186, and thereafter treated with the gathering agent as described above.

The materials settling in the decanter 180 may be removed continuously or intermittently through a line 183, or the materials settling in the treater 192 may be removed continuously or intermittently through a line 193.

When it is desired to introduce the gathering agent into the oil prior to the introduction of the reagent, the valve 157 may be closed, and the gathering agent may be pumped through the line 158 into the stream of oil flowing through the line 165, the valve 159 being open. The commingled stream may be agitated and/or conditioned in the conditioner 123, or it may be bypassed around this conditioner.

Reagent is introduced to the dispersion thus formed by opening the valve 163, the valve 162 being closed, and conducting the reagent through the line 164 into the stream of oil containing dispersed gathering agent flowing through the line 173.

In the case of any of the above flow alternatives, or of others which will be apparent from a consideration of the diagram, a dispersion of gathering agent in oil is finally introduced into the line 27 controlled by the valve 33 and communicating with the line 31 controlled by the valve 32. From this point the handling of the mixture is such as was described and illustrated in connection with Fig. 1.

It has been found that the process in general works best if the following conditions are fulfilled, though it will be understood that the following statements are made for purposes of illustration and direction as to how the process can best be performed and that the limits hereinafter defined can, in many instances, be departed from, particularly by proper correlation of the various steps.

As previously stated, the process may be used with any suitable refining agent, and the modifications which may be desirable according to the various characters of the refining agent will be obvious to those skilled in the art. For the purposes of exposition a more detailed discussion of the refining steps in my process will be limited to the use of an aqueous alkaline solution for the removal of hydrogen sulphide, both present and potential, here classed together as sulphur-containing materials, and various acidic and acid-engendering materials such as mercaptans, phenols, naphthenic acids, and saponifiable complexes of the latter. Furthermore, for the purposes of illustration, the discussion will be largely restricted to the use of an aqueous reagent in which the refining agent and the gathering agent are jointly present.

In removing such materials, it is desirable that the aqueous reagent be of such character as to have an affinity for the sulphurous or acidic materials or of such character as to tend to become associated therewith. Best results have been secured from use of an aqueous alkaline reagent. Sodium hydroxide and sodium sulphide have been found particularly advantageous though materials such as sodium carbonate, potassium hydroxide, potassium carbonate, sodium phosphate, magnesium hydroxide, or other alkaline reagents can be used in this connection. In other instances, various mixtures of these materials can

be used, for instance, a mixture of sodium hydroxide and sodium sulphide or various other mixtures of such materials as mentioned above. Partially spent caustic as produced in various refinery operations, as in the caustic washing of gasoline, is quite suitable for this purpose, particularly when the spent caustic contains appreciable quantities of sodium sulphide. In general, the reagents may be either dissolved or dispersed in the water, depending on their solubility.

The concentration of the aqueous alkaline reagent which I use to remove sulphurous and acidic materials is usually quite low, and 0.1 M solutions have been found very satisfactory. Usually, best results are obtained by solutions from 0.01 M to 0.8 M or 1.0 M. The optimum concentrations from the standpoint of the stability and electric treatability of the emulsions which are formed depends on the character of the stock. Thus, with a crude petroleum in which the impurities were predominantly sulphurous materials, I obtained best results with a 0.1 M solution of NaOH, whereas with another crude in which the impurities were predominantly naphthenic acids, I obtained best results using 0.5 M NaOH. In the instance of still other crudes I have found it advisable to use NaOH solutions less than 0.025 M to remove naphthenic acids. It has been found that, if the aqueous alkaline solutions are too concentrated, there will be a tendency to form reverse-phase emulsions, i. e., water continuous emulsions, which are not susceptible to the electric treatment contemplated, and for this reason the concentration of the aqueous alkali solution should not be so high as to form material quantities of such water continuous emulsion.

Best results have been obtained by using such aqueous reagents in quantity about 20% by volume of the hydrocarbon, and the pumps 16 and 17 may be controlled to introduce this quantity of the aqueous reagent into the flowing stream of the hydrocarbon. However, in some instances, larger or smaller quantities of the aqueous reagent can be utilized, for instance, between 10% and 30% by volume.

It is not always essential to form the preliminary mixture by bringing together flowing streams of the two liquids though this has been found very convenient. Other mixing means can be utilized in this capacity.

If the oil is very viscous, it may be preferable to have the preliminary mixture at an elevated temperature before it moves through the emulsifying means, though this is not always essential if heat is supplied to other portions of the system. It will be found that good results are obtained by heating the constituents of the preliminary mixture either before or after they are brought together. In general, it is advantageous to emulsify at as low a temperature as the viscosity of the oil permits, in order to avoid a tendency toward the formation of oil-in-water type emulsions which sometimes appears at the more elevated temperatures. In obtaining suitable temperatures, heat can be supplied to the ingredients in the heaters 18 and 19 before formation of the preliminary mixture, or the preliminary mixture can be heated in the heater 30. In some instances, it has been found possible to apply all of the necessary heat to the individual constituents, by-passing the heater 30 by opening the valve 32 and closing the valve 33.

As to the degree of mixing, it can be said in general that this process contemplates a some-

what more intimate mixture than proposed in the Eddy patent, supra. The resulting emulsion must, in all instances, be of such character that the hydrocarbon forms the continuous phase, the aqueous reagent being dispersed therein in the form of droplets. If the incoming hydrocarbon contains a small percentage of water in the form of dispersed droplets, herein termed "original droplets," it is desirable that the mixing action should not be sufficiently violent to cause homogenization. The original water droplets should not be subdivided to any material extent in the mixing step. If such original water droplets are present, the mixing action should be of such character as to disperse in the hydrocarbon droplets of the aqueous reagent or water or other gathering agent to coexist with the original droplets, though some combination of the droplets may be allowed to take place in the mixing step. It will be clear, however, that the process is not limited to the treatment of hydrocarbons containing such original water droplets.

The particle size of the droplets in the emulsion discharged from the emulsifying means is not particularly critical, though a heterogeneous particle size has been found to give the best results. In general, however, due to a more intimate mixing action, the average size of the droplets of aqueous reagent will be somewhat smaller than those proposed in the Eddy patent, supra.

It is one of the concepts of the present invention to condition the resulting emulsion before subjecting it to the action of an electric field. This conditioning step, as stated previously, involves essentially an aging of the emulsion which serves several desirable purposes. For instance, it permits additional association of the refining agent and/or the collecting agent with the impurities and better prepares the emulsion for subsequent electric treatment. In general, when using an aqueous alkaline refining agent, this conditioning should extend for a period of one hour or more, and satisfactory results have been obtained by a conditioning period of from one to twelve hours or somewhat more. With most hydrocarbons, it has been found that a conditioning period of from three to five hours is best. The optimum conditioning time will depend upon the particular hydrocarbon and upon the impurities present therein.

It is usually desirable that this conditioning take place while the gathering agent is dispersed in the hydrocarbon. In at least some instances, however, I prefer to condition the mixture of oil and refining agent prior to the addition of any gathering agent, and in still other instances I prefer to add part of my gathering agent prior to the conditioning and part subsequent to the conditioning. In any of the instances leading to further addition of material after the above described conditioning step, I may use a further conditioning step subsequent to the final introduction of gathering agent.

In some instances, the degree of mixing may be sufficient to form a sufficiently tight emulsion which will not settle to too great an extent if allowed to stand quiescent during the conditioning period. If some such settling does take place, the emulsion can be mildly mixed in the conditioning means before being withdrawn therefrom. In other instances, the emulsion can be gently mixed throughout this conditioning period by use of paddles or agitators in the conditioning zones.

It has also been found desirable, in most in-

stances, to condition the emulsion while at elevated temperature. This temperature may be between 120° F. and 180° F. with an optimum range of from 130° F. to 165° F. A conditioning temperature of 140° F. has been found particularly advantageous on certain hydrocarbons. In maintaining this temperature, the conditioning zones can be suitably jacketed or heat-insulated. In most instances, lower temperatures can be used in the preliminary stages of the process and additional heat applied during this conditioning.

Any number of conditioning zones can be utilized. If the process is to be intermittently performed, it is possible to utilize a single zone. In some instances, a single zone can be used in a continuous process by continuously flowing the emulsion thereinto and continuously withdrawing the conditioned emulsion from another portion of the zone. However, the preferred mode of operation provides for the conditioning of the emulsion in a plurality of zones. The emulsion may be supplied thereto in sequence or may be supplied to several of the zones at the same time. The conditioned emulsion can be similarly withdrawn in sequence or from several of these zones.

With hydrocarbons requiring rather low temperatures in the conditioning step, heat can be added to the conditioned emulsion before it is introduced into the electric treater. Desirable temperatures of the emulsion, when introduced into the treater, will vary with different hydrocarbons, but in general this temperature will be between 120° F. and 180° F.

The conditioned emulsion is forced into the treater at superatmospheric pressure by the pump 62 or by the pumps 16 and 17. If the pump 62 is used, it may be of such character as to further mix the constituents of the emulsion, though, here again, it is not desirable to homogenize or form an emulsion which is characterized by an extremely small particle size, for such emulsions are often very difficult to treat electrically. As disclosed in the patent to Eddy, supra, a further gentle mixing may take place in the means through which the emulsion is discharged into the field if desired, and the expedient of using this gentle mixing immediately preceding introduction into the electric field is often advantageous.

The electrically treated hydrocarbon will rise to the upper end of the treater tank substantially free from reacted impurities. Usually, the electric treatment will not remove all of the dispersed droplets, and the treated hydrocarbon will usually contain some rather small droplets dispersed therein. The quantity will not be more than a few per cent and usually will be below one per cent and will not interfere with later distillation if it is desired to remove fractions from the treated hydrocarbon by the use of conventional stills or fractionating means. The treated hydrocarbon can be moved directly to such a fractionating means or it can be moved to storage and withdrawn as desired for subsequent treatment. If thus stored, some additional quantities of the refining agent and/or gathering agent may separate therefrom during storage.

Electric fields of the alternating current type are preferred in effecting this coalescence though the process contemplates the use of any electric field which will coalesce the dispersed droplets.

Various wave forms can be used in establishing such an electric field, but the simplest system involves the use of one or more transformers. In the electric treater of the patent to Eddy, supra, very satisfactory results have been obtained by using transformers which will each develop a potential of 12,000 volts. This will establish a potential difference across the main treating space of 24,000 volts.

By way of illustration and without intent of limiting the invention thereto, the following examples will be given:

Example I

A certain crude stock containing substantially no hydrogen sulphide was tested and found to evolve upon distillation at 600° F., 23.6 grams of hydrogen sulphide per barrel. This crude stock was emulsified with 20% of 0.1 M sodium hydroxide and allowed to stand at 140° F. for three hours to condition the emulsion. A centrifuge test on the resulting emulsion showed 25% B. S. (bottom settlings). The temperature of the conditioned emulsion was raised to 150° F., and the emulsion was electrically treated at this temperature. A centrifuge test was made on the purified hydrocarbon and showed only 0.5% B. S. This purified hydrocarbon was distilled, and it was found that only 9.4 grams of hydrogen sulphide per barrel were evolved at 600° F., a reduction of 60.2% in the amount of hydrogen sulphide evolved during distillation.

Example II

Another crude stock was tested and found to contain 4.9 grams of dissolved hydrogen sulphide per barrel and to evolve 73.3 grams of hydrogen sulphide per barrel when heated to a temperature of 660° F. After treatment by the process herein disclosed, it was found that the treated hydrocarbon contained no dissolved hydrogen sulphide and that, up to a temperature of 660° F., the evolution of hydrogen sulphide was reduced by 53%. In this example, the aqueous reagent was a 0.1 M sodium hydroxide solution, and one volume thereof was dispersed in four volumes of the crude oil. This emulsion was conditioned by allowing it to stand at 160° F. for three to five hours and was electrically treated at 140° F.

Example III

A crude petroleum yielded lubricating oil fractions on distillation which had acid numbers from 1.1 to 1.3 mg. KOH per gram, arising from the naphthenic acid content of these distillates. When the crude oil was treated according to my process, using a caustic soda solution about 0.5 molal in NaOH as the refining agent, and then distilled, the lubricating oil distillates thereby obtained showed acid numbers of 0.05 or less, corresponding to a reduction in naphthenic acid content of about 95%. The naphthenic acids could be recovered from the spent alkali solution by acidification.

Example IV

It will be clear that various changes and modifications can be made without departing from the spirit of this invention as defined in the appended claims.

I claim as my invention:

1. A process for the chemical refining of a hydrocarbon oil containing oil-associated acidic impurities capable of reaction with an alkaline refining agent to remove said acidic impurities from said oil, which process includes the steps of:

mixing with said oil a sufficient amount of said alkaline refining agent to react with said acidic impurities and form reacted impurities dispersed therein; subjecting the treated oil to the action of an emulsion-resolving high-intensity coalescing electric field; emulsifying into the oil prior to subjection to the action of said electric field an aqueous gathering agent capable of association with the reacted impurities whereby said electric field receives an oil-continuous emulsion and coalesces the oil-dispersed material comprising the gathering agent and reacted impurities; and separating the coalesced material from the oil.

2. A process for the removal from hydrocarbon oils of dissolved acidic impurities capable of reacting with an aqueous alkaline solution to form water-soluble reaction products, which process includes the steps of: mixing with the impure oil a sufficient amount of said aqueous alkaline solution to react with said acidic impurities, said mixing being of sufficient intensity to form an emulsion in which the oil is the continuous phase and the aqueous alkaline solution is the dispersed phase whereby said alkaline solution can react with said impurities to form dispersed water-soluble reaction products; resolving said emulsion by subjecting same to the action of a high-potential coalescing electric field, the amount of water in said emulsion being at least about 10%; and separating the coalesced aqueous material from the oil along with reaction products resulting from the reaction between said alkaline solution and said impurities.

3. A process for the electrical-chemical purification of an oil containing acidic impurities which are reactable with an aqueous alkaline refining agent to remove said impurities from said oil, which process includes the steps of: mixing with the oil a sufficient amount of said aqueous alkaline refining agent to react with said acidic impurities, said mixing being of sufficient intensity to disperse the aqueous alkaline refining agent throughout the oil to form an oil-continuous emulsion; subjecting said emulsion to the action of a high-potential coalescing electric field, prior to the time that the reaction between said aqueous alkaline refining agent and said acidic impurities is complete, to carry forward said reaction and coalesce the dispersed aqueous phase; and separating the coalesced aqueous material from the oil.

4. A process for the chemical-electrical purification of hydrocarbon oils containing oil-associated acidic impurities reactable with an alkaline refining agent to produce water-soluble reacted impurities to remove the acidic impurities from said oil, which process includes the steps of: intimately mixing with the oil and dispersing therein a sufficient amount of said alkaline refining agent to react with said acidic impurities and form said water-soluble reacted impurities dispersed in said oil; mixing water with said oil while said reacted impurities are dispersed therein, the amount of said water being sufficient to dissolve said reacted impurities and said mixing being of sufficient intensity to form an oil-continuous emulsion; and substantially coalescing the dispersed water and the dispersed water-soluble reacted impurities under electric stress by subjecting the emulsion while containing the dispersed water and dispersed reacted impurities to the action of a high-potential coalescing electric field; and separating the coalesced material from the oil.

5. A process for the chemical purification of a

hydrocarbon oil to remove therefrom oil-associated acidic impurities capable of reaction with an alkaline refining agent to form water-soluble reaction products, which process includes the steps of: mixing water with the oil in such amount and with sufficient intensity to form an oil-continuous emulsion containing droplets comprising said water; mixing with said emulsion a sufficient amount of said alkaline refining agent to react with said acidic impurities and form dispersed water-soluble reaction products; subjecting the resulting emulsion to the action of a high-voltage coalescing electric field to coalesce the dispersed water and water-soluble reaction products; and separating the coalesced material from the oil.

6. A process as defined in claim 5, including the additional step of dispersing an additional amount of water into said emulsion after incorporation of said alkaline refining agent and before subjection of said emulsion to the action of said electric field.

7. A process for removing from a hydrocarbon oil acidic impurities capable of reacting with an aqueous alkaline solution to form water-soluble reacted impurities, which process includes the steps of: mixing with said oil an aqueous alkaline solution of a strength between 0.01 M. to 1.0 M. and in amount between 10% and 30% by volume, said mixing being of sufficient intensity to form an emulsion of the water-in-oil type and to disperse the aqueous alkaline solution throughout the oil to provide droplets thereof for reaction with said acidic impurities and to produce a dispersed phase of said emulsion comprising water-soluble reacted impurities; substantially completely resolving said emulsion by subjecting same to the action of a high-voltage electric field of sufficient intensity to coalesce the dispersed aqueous phase thereof; and separating the coalesced aqueous material from the oil while containing said reacted impurities, whereby said reacted impurities are recovered from the oil in association with the separated aqueous material.

8. A method of removing dissolved acidic impurities from a hydrocarbon oil, which comprises: mixing the oil containing acidic impurities with an alcoholic alkali solution containing sufficient water to be dispersible in the oil in the form of small droplets and containing sufficient alkali to react with said acidic impurities, said mixing being of sufficient intensity to form an emulsion of the water-in-oil type and to disperse the alcoholic alkali solution throughout the oil to provide droplets thereof for reaction with said acidic impurities and to produce a dispersed phase of said emulsion comprising reacted impurities; subjecting this emulsion to the action of a high-tension electric field to aid in the resolution of the emulsion through coalescence of the dispersed droplets to masses readily susceptible to gravitational separation, said masses comprising alcohol and reacted impurities; and separating the coalesced aqueous masses containing the alcohol and reacted impurities from the purified oil.

9. A method of refining a hydrocarbon oil containing acidic impurities, comprising: emulsifying said oil with an alkali dissolved in alcohol containing sufficient water to prevent complete miscibility with the oil thereby forming an oil-continuous emulsion in which the alcoholic alkali comprises the dispersed phase, the amount of said alkali being sufficient to react with said

acidic impurities to form reacted impurities; subjecting the emulsion to the action of an electric field of sufficient intensity to coalesce the dispersed alcoholic phase to form a coalesced alcoholic phase containing reacted impurities; separating from the oil the coalesced alcoholic phase containing reacted impurities; mixing water with the separated oil to form an emulsion comprising a continuous oil phase with dispersed aqueous droplets therein; subjecting the emulsion thus obtained to the action of an electric field of sufficient intensity to coalesce the dispersed aqueous droplets to form aqueous masses responsive to gravitational separation; and separating said coalesced aqueous masses from the purified oil.

10. A process for removing naphthenic acids from hydrocarbon oils, comprising: intimately mixing with such hydrocarbon oil and dispersing therein a sufficient amount of an alcoholic alkali to substantially neutralize said naphthenic acids and form reacted impurities dispersed in the oil; mixing water with the oil containing said dispersed reacted impurities while using such intensity of mixing as will produce an oil-continuous emulsion; subjecting the emulsion to the action of a high-tension electric field to coalesce the dispersed aqueous phase into aqueous masses comprising the reacted impurities; and separating the aqueous masses containing reacted impurities from the purified oil.

11. A process as in claim 8 in which the purified oil is subsequently contacted with water to remove any residual alcohol.

12. A process for removing acidic impurities such as mercaptans and naphthenic acids from petroleum fractions, comprising: mixing the fraction with an alcoholic solution of alkali with sufficient intensity to form an oil-continuous emulsion, said alcoholic solution of alkali containing sufficient water to constitute the dispersed phase of said emulsion and the amount of alkali being sufficient to react with said acidic impurities; subjecting the emulsion to a high-tension electric field to coalesce the alcoholic phase; separating the coalesced alcoholic phase to produce a partially purified oil; dispersing water into the partially purified oil; and subjecting the resulting dispersion to the action of a high-tension electric field to coalesce the aqueous phase and produce a purified oil.

13. A process for removing acidic impurities such as hydrogen sulphide, phenols, naphthenic acids, and mercaptans from hydrocarbon oils containing same, which process includes the steps of: mixing with the oil containing one or more of said acidic impurities an alcoholic alkali solution containing sufficient water to prevent complete miscibility with the oil and to form therein a dispersed phase, the amount of alkali in said alcoholic alkali solution being sufficient to react with substantially all of the alkali-reactable acidic impurities in said oil, said mixing being sufficient to produce an emulsion; subjecting this emulsion to the action of an electric field of sufficient intensity to coalesce the dispersed phase; and separating the coalesced material from the oil.

14. A process for removing acidic impurities such as hydrogen sulphide, phenols, naphthenic acids, and mercaptans from hydrocarbon oils containing same, which process includes the steps of: mixing with the oil containing one or more of said acidic impurities an alcoholic alkali solution containing sufficient water to prevent com-

plete miscibility with the oil and to form therein a dispersed phase, the amount of alkali in said alcoholic alkali solution being sufficient to react with said acidic impurities in said oil, said mixing being sufficient to produce an emulsion; retaining a mass of said emulsion in mildly agitated state for a period of time to permit continued reaction between the dispersed alcoholic alkali phase and said acidic impurities; then subjecting this emulsion to the action of an electric field of sufficient intensity to coalesce the dispersed phase; and separating the coalesced material from the oil.

15. A process for removing from hydrocarbon oils acidic impurities contained therein, which acidic impurities are reactable with an alcoholic alkali solution to produce water-soluble reacted impurities dispersed in the oil, which process includes the steps of: separately adding to the oil and mixing therewith two liquids, one liquid being an alcoholic alkali solution capable of and added in amount sufficient to react with said acidic impurities to produce said water-soluble reacted impurities, the other liquid being water and being added in sufficient amount to dissolve said water-soluble reacted impurities; subjecting this oil-continuous emulsion to the action of a coalescing electric field of a character to coalesce the dispersed phase of the emulsion into masses of sufficient size to gravitate from the oil, said masses comprising the reacted impurities; and separating these masses from the oil.

16. A process as defined in claim 10, in which residual alcohol is removed from the purified oil by mixing this purified oil with water to disperse the water into the oil, subjecting the resulting dispersion to the action of an electric field of sufficient intensity to coalesce the water, this coalesced water containing residual alcohol, and separating this coalesced water from the oil.

17. A process for removing from hydrocarbon oils acidic impurities contained therein, which acidic impurities are reactable with an alcoholic alkali solution to produce water-soluble reacted impurities dispersed in the oil, which process includes the steps of: intimately mixing with such hydrocarbon oil a sufficient amount of alcoholic alkali to react with said acidic impurities and form said water-soluble reacted impurities dispersed in the oil; mixing water with the oil while said dispersed reacted impurities are present therein, the amount of said water being sufficient to dissolve said reacted impurities and the water-mixing step being of sufficient intensity to form an oil-continuous emulsion; subjecting this oil-continuous emulsion to the action of a coalescing electric field of a character to coalesce the dispersed phase of the emulsion into masses of sufficient size to gravitate from the oil, said masses comprising the reacted impurities; and gravitationally separating said masses from the oil.

18. A continuous process for the removal of acidic impurities from hydrocarbon oils, said acidic impurities being reactable with an alcoholic alkali solution to produce reacted impurities dispersed in the oil, which process includes the steps of: continuously bringing together and mixing a stream of said oil and a stream of alcoholic alkali solution, the amount of alcoholic alkali being sufficient to react with said acidic impurities to form said reacted impurities dispersed in the oil and said alcoholic alkali solution being incompletely oil-soluble so as to be dispersible in the oil in the form of small droplets, said mixing being of sufficient intensity to

produce a stream of oil-continuous emulsion; continuously introducing said stream of oil-continuous emulsion into an electric field of sufficient intensity to coalesce the dispersed phase to produce aqueous masses comprising said reacted impurities dispersed in the oil but of sufficient size to settle therefrom; and continuously separating said aqueous masses from the oil.

19. A process for removing from a hydrocarbon oil acidic impurities capable of reacting with an aqueous alkaline solution to form water-soluble reacted impurities, which process includes the steps of: mixing with said oil a sufficient amount of said aqueous alkaline solution to react with said acidic impurities, said mixing being of sufficient intensity to form an emulsion of the water-in-oil type and to disperse the aqueous alkaline solution throughout the oil to provide droplets thereof for reaction with said acidic impurities and to produce a dispersed phase of said emulsion comprising water-soluble reacted impurities; conditioning said emulsion by maintaining same for a period of at least one hour while preventing substantial separation of the constituents of said emulsion during said period to permit the dispersed aqueous alkaline solution to react with said impurities; then substantially completely resolving said emulsion by subjecting same to the action of a high-voltage electric field of sufficient intensity to coalesce the dispersed aqueous phase thereof; and separating the coalesced aqueous material from the oil while containing said reacted impurities, whereby said reacted impurities are recovered from the oil in association with the separated aqueous material.

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