

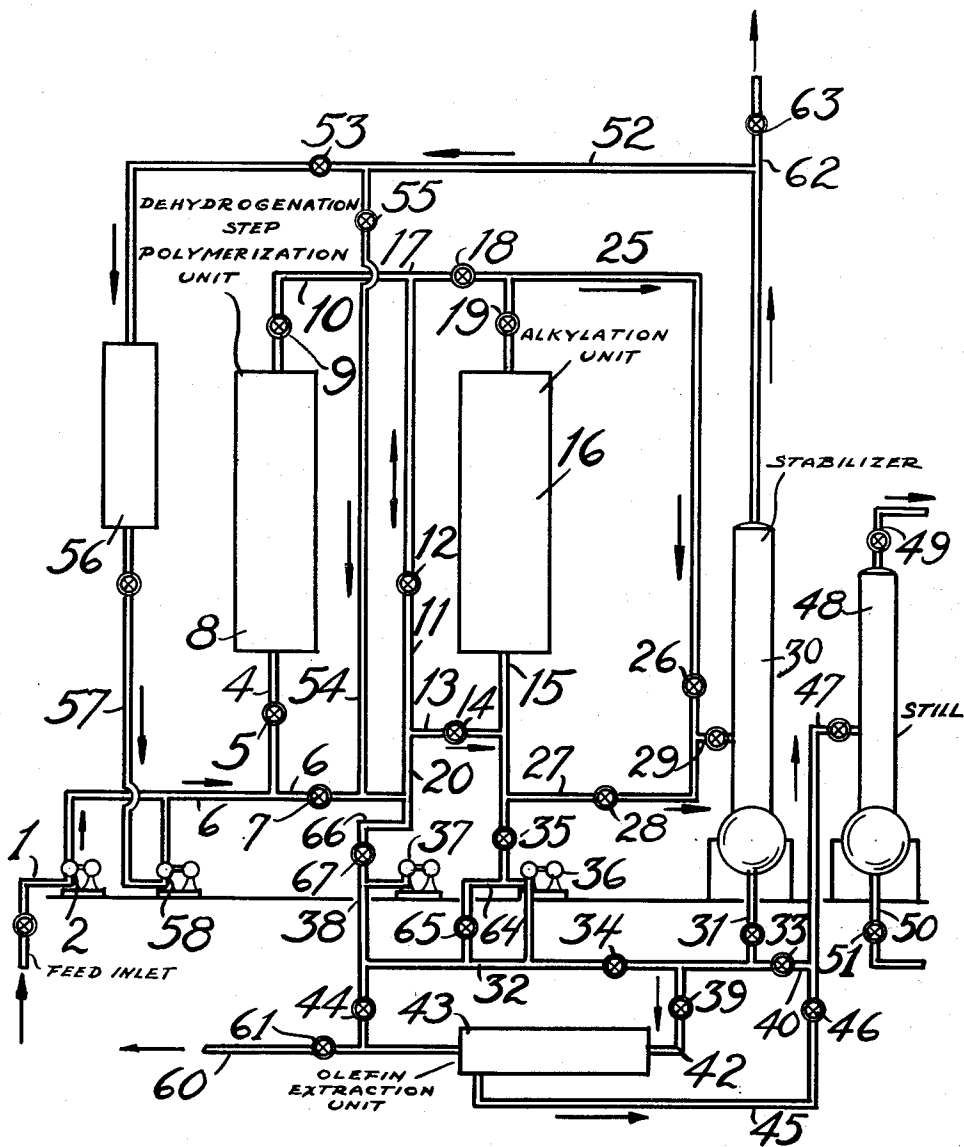
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CATALYTIC REACTION OF OLEFINS

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CATALYTIC REACTION OF OLEFINS

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This invention relates to improvements in the production of highly desirable components of motor fuels and pertains particularly to the production of relatively low boiling saturated aliphatic hydrocarbons which have been found to be useful in the preparation of gasolines.

It has heretofore been proposed to produce motor fuel constituents according to a number of well known processes, such as thermal and catalytic cracking of heavier hydrocarbons, polymerization of normally gaseous olefins, followed by hydrogenation, treating of crude and straight run gasoline cuts with olefins to improve their octane number by alkylating the aromatic constituents thereof with normally gaseous olefins in the presence of various catalysts, such as phosphoric acid impregnated on kieselguhr, etc. It has been found possible to react paraffinic hydrocarbons with olefins to produce saturated hydrocarbons boiling within the motor fuel range. This reaction has been denoted as alkylation. It is therefore readily apparent that a saturated branched chain normally liquid paraffin may be produced directly by alkylating a normally gaseous branched chain paraffin, for example, isobutane, by treating such a compound with a normally gaseous mono-olefin without the necessity of including a hydrogenation treatment in the process as would be the case where a polymerization process had been used.

Another advantage of the use of an alkylation process in contrast to the well known polymerization process lies in the fact that the petroleum industry is constantly seeking methods whereby it may be possible to utilize by-products of the industry and in this respect, alkylation is a distinct advance in the art. Large supplies of field butanes, refinery C₄ cuts from cracking units, debutanizer units, etc., each containing substantial quantities of gaseous olefins and paraffins, both straight and branched chain, are available. Such gaseous mixtures have heretofore been fed to polymerization processes which are able to utilize only the olefinic content of the mixtures. As a consequence, the industry has found it expedient to dehydrogenate the inert paraffinic gases coming from such units, to convert them to olefins and to then refeed these gases to the polymerization processes. Such a procedure is unnecessary in a comparable alkylation process, and the expensive dehydrogenation of paraffins and subsequent necessary hydrogenation of the condensation products is

eliminated, yet a gasoline of substantially the same or higher octane value is produced.

It is an object of the present invention to provide a process for the production of normally liquid acyclic hydrocarbons boiling in the gasoline range by reacting mixtures containing at least one mono-olefin under condensation conditions in the presence of catalysts containing binary and ternary inorganic compounds of uranium.

It is an object of the present invention to provide a process for the production of premium grade motor fuels by the reaction of saturated hydrocarbons containing at least one tertiary carbon atom and boiling below the desired gasoline range or mixtures thereof with monomeric mono-olefins boiling below the desired gasoline range, dimers, trimers, tetramers, etc. of said monomeric mono-olefins or mixtures thereof.

It is a further object of the invention to so carry out the alkylation of isoparaffins that it is possible to obtain substantially twice as large a yield of premium grade gasoline based upon the amount of olefins reacted as would be obtained in the conventional olefin polymerization processes.

To accomplish such objects, it is a feature of the present invention to carry out the reaction under optimum reaction conditions in the presence of alkylation catalysts which have been found to be particularly effective in promoting the desired reactions. It is obvious that the optimum reaction conditions will vary for each particular catalysts employed. In general, it may be said that alkylation conditions predominate when the feed rates are adjusted to provide longer contact time than would be used in corresponding polymerization reactions. Better yields of saturated products are obtained, however, where the olefin concentration is somewhat lower than in the corresponding polymerization reactions. This phenomenon is probably correlated with the fact that, in general, lower temperatures, i. e., about room temperature or slightly lower, have been found to be most advantageous in obtaining completely saturated products. It was found that when the olefinic content of the feed stock was too high, the temperature rose too rapidly to be accurately controlled within the desired range and a lower yield of alkylated products resulted. Also a very low temperature led to the formation of high molecular weight polymers while tem-

peratures above 0° F. led to the formation of lower boiling alkylation products.

Likewise, it was found that the more intimate the contact between the reactants, the higher the yields of alkylated products. The feed stock is preferably introduced into the reaction zone through jets, porous thimbles, turbo-mixers and the like.

It was sometimes found to be advantageous in the production of high octane saturated fractions suitable for motor fuels, to return to the reaction zone the heavier alkylated fractions and the fractions boiling intermediate the reactants and the desired motor fuel fraction removed from the system as the final product. It is sometimes desirable to leave the products formed intermediate the reactants and the C_5 cut in the final product since the C_5 , C_6 and C_7 saturated hydrocarbons are readily volatilized and have excellent anti-knock properties. Likewise, any unreacted reactants are separated from the desired fraction and returned to the reaction zone. Increased yields of the desired fraction result whether this recycle step is carried out continuously or intermittently, whether only part or all of the recycle stock is fed into the alkylation zone together with the fresh feed.

The invention is not limited to the use of reactants from any particular source. Single paraffinic and olefinic hydrocarbons may be employed. However, it is commercially more feasible to use mixtures of hydrocarbons containing the necessary paraffins and olefins as part of their constituents. The reacted mixture from catalytic and thermal polymerization units, including the gaseous constituents thereof, may be directly introduced as reactants into the alkylation process of the invention. Gaseous mixtures evolved from catalytic and thermal cracking units may also be advantageously employed. It is an essential requirement in the practice of the invention that the feed stock contain at least one paraffin containing a tertiary carbon atom and preferably boiling below the boiling point of the desired final product fraction, and that the feed stock contain at least one mono-olefin. Partially dehydrogenated field butanes, refinery C_3 - C_4 and C_4 cuts, saturated paraffinic mixtures containing isoparaffins and which have been enriched with mono-olefins from extraneous sources, etc. are suitable. The invention contemplates the reactions of isobutane, isopentane, etc. with ethylene, propylene, normal or isobutylenes, normal and branched chain amylene, the dimers, trimers, tetramers, etc., codimers, cotrimers, cotetramers, etc., cross polymers, interpolymers, etc. of these olefins and the like. In particular, it is contemplated to react an isoparaffin with a lower mono-olefin such as ethylene or propylene. It is preferable, to insure high yields of alkylation products, to have between about 2 and about 12 mols of isoparaffin in the reactor per mol of mono-olefin present. Likewise, from 4 to 24 mols of isobutane should be contacted with each mol of diisobutylene, etc. This avoids excessive formation of high boiling compounds.

It has been found that certain novel catalysts effectively promote the alkylation reaction. In particular, the inorganic compounds of uranium have been found to promote the alkylation reaction. The compounds which have been found to be suitable are the oxides of uranium, the halides of uranium, such as the chlorides and bromides, sulfates and phosphates. Uranium oxide, U_3O_8 , has been found to be particularly effective, not

only in promoting the formation of saturated compounds, but also in the formation of polymers. The addition of small amounts of alkyl halide, such as ethyl chloride, the propyl chlorides or bromides, tertiary or secondary amyl chloride, have been found to promote the activity of these catalysts, their effects being to increase the formation of C_5 - C_8 saturates at the expense of initially formed olefin polymers.

The catalyst may be deposited or impregnated on carriers such as clays, either natural or acid activated gels, such as alumina gel or silica gel. Bauxite, fuller's earth, bentonite, kieselguhr, pumice, celite, montmorillonite, Marsil, Tonsil, Super Filtrol, activated Floridin, etc., serve as excellent carriers for the uranium compounds above mentioned. Also, it is contemplated to use these uranium compounds deposited upon the carriers above mentioned in conjunction with the use of the alkyl halide promoters.

The percentage of uranium compounds deposited upon the various carriers may vary from between about 1% and about 15%, but it is preferable to use from about 5% to about 10% of the weight of the carrier of uranium salts impregnated or deposited thereon. The optimum percentage of uranium salts or oxides, deposited upon the various carriers depends, to a large extent, upon the amounts of the various salts used and also to some extent upon the surface available of the particular carrier. Thus, a larger percentage by weight of uranium sulfates would be required than would be the case where the uranium halides were used, all other conditions remaining the same; also, the acid activated clays, where a siliceous body, such as celite, is used, would require a smaller quantity of the uranium compounds than would be the case where the uranium compounds were used alone or impregnated upon or carried upon substances having a less degree of porosity and surface area.

The catalyst may be used in any suitable conventional form; that is, it may be deposited upon a gel carrier of extremely high porosity, said porosity having been increased by the admixing of the gel with carbonaceous substances such as cane sugar or carbon, and heating to oxidize off these substances, thereby leaving a gel of increased porosity for any given density; or the catalyst may be admixed or impregnated upon highly activated clays, which are then pillled or finely comminuted and used in the reaction medium in the form of a powdered catalyst, or the catalyst may be admixed with a carrier and extruded through orifices of predetermined size. The catalyst mass may also be made up in the form of small pellets having the uranium compounds deposited thereon, or, if desired, the uranium salts may be used by themselves without the aid of any carrier, although this is not usually the preferred procedure.

The process may be carried out either as a batch or continuous run. It may be carried out in either the gas or liquid phase. Where it is carried out in the liquid phase, it is desirable to maintain sufficient pressure so that the reactants are in the liquid state and are allowed to flow over the beds of the catalyst mass, preferably in vertical reactors with a downward flow of the reactants through that mass. From a commercial standpoint the continuous process lends itself more readily to economical operation and is to be preferred over the conventional batch-type operation. It is to be distinctly understood, however, that the particular mode of operation of

the invention is not limited in any respect but that any of the conventional forms of apparatus used in polymerization or analogous alkylation processes may be employed with highly satisfactory results.

Pressures ranging from about 0 lb./sq. in. to about 200 lbs./sq. in. gauge may be used, but in general, it may be said that the higher pressures, coupled with the higher temperatures, are not conducive to the formation of alkylation products. Atmospheric pressures have been found to give good results, as well as moderately elevated pressures in the neighborhood of about 50 lbs./sq. in. gauge. Temperatures of between about 10° F. and about 500° F. have been used, but it is preferred to operate within a temperature range between about 15 and about 80° F. Temperatures much below 10° F. are distinctly detrimental to the production of the desired products; that is, those products boiling within the motor fuel range. Thus, the use of a temperature around -80° F. would produce a heavy, viscous liquid wholly unsaturated in character and of little value so far as the production of motor fuels from olefins and isoparaffins is concerned.

Reaction times of between about ½ and about 7 hours have been found to be desirable, but it is preferred when operating to secure optimum yields of saturated products to allow a contact time of between about 2½ and about 5 hours. This allows for a substantially complete alkylation of the paraffinic constituents and, at the same time, results in the production of relatively little of the higher boiling constituents.

The amount of alkyl halide added ranges from about 1% to about 15%, depending upon the specific alkyl radical and the specific halide present. Thus, in the case where a propyl radical is present, less weight per cent of the compound is required to accomplish the same results than in the case where a butyl radical was employed. The same also holds true where the halide portion of the added compound is of less molecular weight than another halide of greater weight, for example, chloride would require a less weight per cent to be added than would bromide and yet, the same results could be accomplished so far as qualitative yields are concerned. In cases where tertiary butyl chloride is used as the alkyl halide, it is preferred to add between about 3 and about 6.5% by weight based on the total amount of feed stock used in the run.

Experimental evidence points to the theory that the mechanism of the alkylation reaction involves the intermediate formation of polymers or copolymers by the mono-olefins followed by the degradation of these higher polymers and copolymers into an active or nascent state which permits their reaction with the paraffins present. The same mechanism appears to hold true where the reaction occurs between a preformed polymer or copolymer and a paraffin. While the theory of the invention appears to be as described, it is to be distinctly understood that the invention is not limited to any theory of operation and is restricted only insofar as indicated by the appended claims.

No specially designed apparatus is necessary for the successful carrying out of the invention. Conventional apparatus now customarily used in effecting solid catalytic polymerizations is quite suitable. In the case of effecting alkylations under pressures of 1800 to 3000 lbs./sq. in, it has been found to be advantageous to introduce the

reactants into the catalyst chamber from the top and allow them to flow through the reactor in a downward direction, whereas, in cases where the alkylation proceeds at room temperatures or slightly below and under substantially atmospheric pressure, it is advantageous to introduce the reactants into the catalyst chamber at the bottom and remove the reacted mixture from the top of the reactor. The fractionation of the resultant products is accomplished in conventional fractionating towers. Any excess or unreacted reactants may be recycled to the catalytic reactor after first being mixed with the fresh feed just prior to its introduction into the catalyst chamber. If desired, the recycled reactants may be subjected to a dehydrogenation treatment prior to their mixing with the fresh feed. It is apparent that the process and apparatus may be readily adapted to the carrying out of a continuous operation. Such a type of process and apparatus is familiar to those working in the art and it is to be understood that conventional and/or minor variations therefrom may be made without departing from within the spirit or scope of the present invention.

For illustrative purposes and not with the intention of restricting the invention thereto, there is shown in the accompanying drawing a diagrammatic illustration or flow sheet representing one specific process adapted to carrying out the invention. The process may be adapted to the use of liquid, gaseous, or solid catalysts as the exigencies of each particular case might require. It is to be distinctly understood, however, that the invention in its broader aspects is not restricted to any particular type of apparatus or to any particular flow plan, but that the flow plan hereinafter described is only intended to aid in a further understanding of applicants' invention.

Referring to the drawing, the olefins and paraffins to be subjected to alkylation are introduced into the system through charging line 1 by means of pump 2. The olefinic-paraffinic hydrocarbon mixture may then be passed through pipes 6, 20, 13 and 15 controlled by valves 7 and 14 directly into alkylation reactor 16 which is filled with the desired catalyst and provided with suitable heating or cooling jackets (not shown), or, if it is desired to polymerize a portion of the olefinic content of the feed prior to the alkylation reaction, valve 7 may be closed and the feed passed through line 4 controlled by valve 5 into any suitable conventional polymerization reactor such as that represented by reactor 8. The olefinic portion of the feed is converted into polymers thereof while the paraffinic portion remains unchanged. The resultant mixture of polymers and paraffins exits through pipe 10 controlled by valve 9. Valve 18 is closed and the polymerized mixture enters the alkylation zone through lines 10, 13 and 15 controlled by valves 12 and 14.

The reacted mixture is discharged from the alkylation reactor through pipes 19 and 25 controlled by valve 26. Valve 28 is closed and the reacted mixture is passed into the stabilizer 30 by means of pipe 29.

It is sometimes advantageous from the standpoint of yields, quality of product, etc. to carry out the alkylation reaction by directing the reactants into the alkylation reactor so that they will flow through the catalyst in the opposite direction from that described above. In such a case, reactants not passing through polymerization zone 8 are flowed through pipe 6 controlled

by valve 7 into pipes 20, 11, 17 and 19, valves 12 and 18 being open and valves 5, 9, 14 and 26 being closed. The reaction mixture discharged from the alkylation zone 16 passes by means of lines 15, 27 and 28 into stabilizer 30. If it is desirable to first subject the olefinic-paraffinic hydrocarbon mixture to a preliminary polymerization treatment, valves 7, 12, 14 and 26 remain closed and valves 5, 9, 18 and 28 are opened so that the flow is through pipes 4, 10, 17 and 19 into the alkylation reactor 16.

It is sometimes found advantageous to allow the ultimately complete alkylation reaction to proceed in small increments of the total final conversion and to recycle the reactants continuously through the alkylation zone 16 through the circuit of pipes 15, 19, 17, 11, 20, 38 and 32 provided with pump 37. Pump 38 is by-passed by allowing the flow through pipe 64 and valve 65. It is contemplated to so recycle whether the reactants are contacted with the alkylation catalyst downflow or upflow, pump 38 being provided for the reversed direction of flow from that above indicated. For downflow operation pump 37 is by-passed by allowing the flow through pipe 66 and valve 67. A portion of the reacted mixture so recycled is withdrawn either intermittently or continuously, the rate of withdrawal being adjusted to insure adequate time for substantial completion of the alkylation.

Any suitable method of dispersing the reactants into reactor 16 is contemplated, such as jets, turbo-mixers, atomizers, porous thimbles, etc.

The reacted mixture enters stabilizer 30 through pipe 29. The temperature of the stabilizer is maintained at such a point as to insure the removal of normally gaseous hydrocarbons from the normally liquid hydrocarbons. The gases comprising paraffins, and at times some olefins, leave the stabilizer by means of pipe 52 controlled by valve 53. If desired, these gases coming from the stabilizer may be reintroduced directly into the fresh feed pipe 6 by closing valve 53 and opening valve 55 so that the gases are conducted to pipe 6 by means of pipe 54, or the gases may be bled from the system through pipe 52 and valve 63, valves 53 and 55 being closed. A compressor unit (not shown) is inserted in line 54 to liquefy the gases prior to their introduction into feed line 6. It is preferable, however, to close valve 55 and open valve 53 so that these gases which are predominantly paraffinic in character are passed to a dehydrogenation step 56 of any suitable conventional design, wherein the gaseous paraffins are converted into gaseous olefins either in whole or in part, the gases are liquefied, hydrogen, methane, C₂ and C₃ fractions are removed, and the remaining gases leaving the dehydrogenation step 56 by pipe 57 are pumped by means of pump 58 into the fresh feed line 1.

The normally liquid products leaving the bottom of stabilizer 30 by means of pipe 31 may be conducted directly to the gasoline still 48 through lines 40 and 47 by opening valve 33 and closing valve 46, conducted directly back to the alkylation unit 16 by means of pipe 32 by closing valves 33, 39 and 35 and opening valve 34, thereby effecting further conversion before final treatment, or conducted by means of pipe 42, closing valves 33 and 34 and opening valve 39, to an olefin extraction unit 43 wherein by any suitable conventional treatment, such as treatment with a solvent, for example, sulphur diox-

ide, any normally liquid olefinic polymers that might be present are removed. Free olefins from this unit may be removed to storage (not shown) through line 60 controlled by valve 61 or they may be directly returned by means of pipe 44, 38 and 28 to the alkylation unit 16. It is to be understood that when the bottoms from stabilizer 30 are conducted back to the alkylation unit 16, only a portion of these bottoms are so recycled at any one time, the remainder of the bottoms being treated according to any of the alternative methods above suggested.

The liquid paraffinic fraction is conveyed to gasoline still 48 by means of pipes 45 and 47 upon opening valve 46.

Gasoline still 48 is maintained at suitable controlled temperatures such that a desired gasoline fraction is removed to storage through pipe 49 and the heavier fraction removed through line 50 by opening valve 51.

It is convenient when using a solid catalyst to have a plurality of reactors, such as alkylation reactor 16, arranged in parallel so that while two or more are being actively used a similar number may be subjected to a regeneration process. Such a procedure thereby provides for intermittent reactivation of solid catalysts without interruption of the continuous process.

Example 1

A feed stock containing 10.5% of isobutylene, 0.4% normal butylene and 81.9% of isobutane in an amount of 14 grams of catalyst per 100 grams of feed was subjected to a 10% U₃O₈ impregnated on silica gel catalyst at a pressure of 50 lbs./sq. in. gauge and a temperature of about 77° F. in a batch operation for a few hours. A product was obtained in an amount of about 73% yield based upon the total unsaturates in the feed stock, had a bromine number of about 100, and contained 25% paraffinic hydrocarbons. The product consisted of 70% by weight of a C₆ fraction and 30% by weight of a C₇ and heavier fraction, and had a final boiling point of 397° F.

Example 2

A feed stock composed of 10.8% of isobutylene, 0.4% normal butylene, 85.5% of isobutane and 5.5% of tertiary butyl chloride was contacted with a catalyst composed of 4% U₃O₈ impregnated on activated alumina in an amount corresponding to 11 grams of catalyst per 100 grams of feed stock for 5 hours at atmospheric pressure and at a temperature of about 15° F. The product was obtained in a yield of about 70% based upon the isobutylene content of the feed stock, and had a bromine number of 65. It was composed of 2% by weight of a C₆ fraction, 10% of a C₇ fraction, 30% of a C₇ fraction, 20% of a C₈ fraction and 38% of a C₉ and heavier fraction.

In a similar experiment conducted under substantially identical conditions of operation, but wherein no tertiary butyl chloride was added to the feed stock, no reaction occurred, hence it may be concluded that the tertiary butyl chloride quite actively promotes the latent ability of uranium catalysts in influencing their alkylation activities. Apparently, when an activated alumina is used as the carrier for the uranium oxide catalyst, the addition of an alkyl halide is required in order for the uranium oxide to effectuate an alkylation reaction. It will be noted that in the case of Example 1, a catalyst of uranium oxide on silica gel promotes alkylation without the presence of an alkyl halide, whereas

in the case of uranium oxide on activated alumina, apparently no alkylation resulted unless an alkyl halide was present.

Example 3

A feed stock composed of 10.2% isobutylene, 1.6% normal butylene and 88.2% of isobutane in an amount of 11.8 grams of catalyst per 100 grams of feed was contacted with a 10% uranium oxide impregnated on Super Filtrol for 5½ hours with vigorous agitation at atmospheric pressure and at a temperature of about 15° F. A product was obtained which had a bromine number of 65 and contained 60% by weight of saturated hydrocarbons. The product had an A. P. I. gravity of 48.8°.

Example 4

Using the same catalyst as in the preceding example, and contacting the same continuously with a feed stock composed of 9.4% isobutylene, 1.8% normal butylene and 88.8% isobutane at a temperature of about 404° F. and a pressure of 2000 lbs./sq. in. gauge at a continuous throughput of about 3.6 volumes of feed/volume of catalyst/hour, resulting in a yield of 97% based upon the isobutylene content of the feed stock. The product had a bromine number of 150 and contained no paraffinic hydrocarbons. The product was composed of 60% by weight of a C₈ fraction and 40% by weight of a C₉ and heavier fraction, and illustrates an example wherein an uranium oxide catalyst effectively promoted a polymerization reaction rather than an alkylation reaction. The olefin reduction during the reaction amounted to 88%.

The above examples are intended to be but illustrative of the concept of the invention and they are not to be considered as limiting in any way the scope of the invention.

The nature and objects of the present invention having been thus fully described what is claimed as new and useful and desired to be secured by Letters Patent is:

1. A process for the production of normally liquid acyclic hydrocarbons boiling in the gasoline range and containing substantial amounts of saturated, branched chain hydrocarbons, which comprises contacting a mixture comprising essentially isoparaffins and mono-olefins with at least one binary compound of uranium under alkylation reaction conditions.

2. A process as in claim 1 wherein the mixture contacted contains no constituent of more than 16 carbon atoms per molecule.

3. A process as in claim 1 wherein the process is carried out continuously under at least sufficient pressure to maintain the reactants in the liquid phase.

4. A process for the production of substan-

tially saturated acyclic hydrocarbons boiling chiefly in the gasoline range which comprises reacting a mixture containing at least one paraffin containing at least one tertiary carbon atom with at least one member of the group consisting of normally gaseous monomeric mono-olefins, dimers, trimers, tetramers, and higher polymers thereof, co-, inter-, and cross-dimers, trimers, tetramers and higher analogous polymers thereof, in the presence of at least one binary compound of uranium under alkylation reaction conditions.

5. A process as in claim 4 wherein the uranium compound is deposited on a clay in the presence of a lower alkyl halide.

6. A process as in claim 4 wherein the uranium compound is deposited on silica gel.

7. A process as in claim 4 wherein uranium compound is deposited on activated alumina and wherein the feed stock contains tertiary butyl chloride.

8. A process as in claim 4 wherein the uranium compound is deposited on an acid-treated bentonitic clay and the reaction is carried out in the presence of between about 1% and about 15% of a lower alkyl halide.

9. A process as in claim 4 wherein the reaction is carried out at temperatures between about 15 and about 80° F. and at pressures between about 0 and about 50 lbs./sq. in. gauge.

10. A process for the production of saturated acyclic hydrocarbons boiling chiefly in the gasoline range which comprises reacting a mixture containing isobutylene and isobutane in the presence of an oxide of uranium under alkylation reaction conditions.

11. A process which comprises reacting a mixture containing about 10.5% of isobutylene and about 89.5% of isobutane at about 50 lbs./sq. in. gauge at a temperature of about 77° F. in the presence of a 10% U₃O₈ impregnated on silica gel.

12. A process which comprises reacting a mixture of about 11% of isobutylene and about 85.5% of isobutane and about 5.5% of tertiary butyl chloride at a temperature of about 15° F. for about 5 hours in the presence of a 4% U₃O₈ impregnated on activated alumina.

13. A process which comprises reacting a mixture of about 10% of isobutylene, about 2% of normal butylene and about 88% of isobutane at a temperature of about 400° F. and a pressure of about 2000 lbs./sq. in. gauge at a throughput of about 3.6 volumes of feed/volume of catalyst/hour in the presence of a catalyst composed of a 10% U₃O₈ impregnated on an acid-treated bentonitic clay.

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