PATENT SPECIFICATION

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COMPLETE SPECIFICATION

Synthesis of Hydrocarbons

We, Standard Oil Development Company, a Corporation duly organized and existing under the laws of the State of Delaware, United States of America, having an office at Elizabeth, New Jersey, United States of America, do hereby declare the nature of this invention and in what manner the same is to be performed, to be particularly described and ascertained in and by the following statement:—

The present invention relates to the synthesis of hydrocarbons and oxygenated organic compounds from carbon oxides and hydrogen in the presence of suitable catalysts. More specifically, the invention relates to an improvement of this process, which affords a convenient means for adjusting the molecular weight distribution of the synthesis product in accordance with market demands.

The synthesis of hydrocarbons and other valuable products from gas mixtures containing various proportions of hydrogen and carbon oxides, particularly carbon monoxide in fixed bed as well as in dense phase fluid catalyst operation is well known in the art. The character and quality of the synthetic product depends largely on the temperatures, pressures, H_2:CO ratios of the feed gas and the type of catalyst used, the latter being usually an iron group metal catalyst promoted with such promoters as various alkali metal compounds, rare earth metal oxides, magnesia, alumina, and/or other compounds in amounts of about 0.5—10%.

For example, cobalt supported on an inert carrier and promoted with thoria and/or magnesia may be used in combination with relatively low pressures (atmospheric to about 5 atmospheres), low temperatures (about 375°—425° F.) and high H_2:CO ratios of 2 or more, to produce a substantially saturated hydrocarbon material from which valuable diesel fuels, lubricating oils and waxes may be obtained. Iron-type catalysts usually promoted with a suitable alkali metal compound such as the chlorides or fluorides, carbonates, oxides, phosphates, silicates, acetates, etc. of potassium or sodium may be used in combination with relatively high pressures (about 5—50 atmospheres), high temperatures (about 500°—750° F.) and low H_2CO ratios of not above 2, to produce a predominantly unsaturated material from which large proportions of high octane motor fuels may be recovered.

The synthesis feed gas may be produced either by suitable conversions, such as oxidation or reformation of hydrocarbon gases such as natural gas or by suitable water gas processes from solid carbonaceous materials such as coal or coke. In either case, the normally liquid synthesis products are more valuable than the hydrocarbon gases which may be synthetically produced. Prior to the present invention, therefore, the research and development work as well as commercial operation have, with very minor exceptions, been directed to an improvement of the liquid product yields of the invention. The exceptions were mainly concerned with the development and use of specific catalysts promoting the formation of gases.

The market demands for normally liquid and normally gaseous fuels fluctuate considerably depending on various foreseeable and unforeseeable factors. The most marked and consistent fluctuations of the market demands are seasonal changes. The demand for liquid fuels, particularly motor fuels, is largest during summer and lowest during winter, while the demand for fuel gases is highest during winter and lowest during the summer. These fluctuations are of course of rather irregular intensity depending on weather conditions and other factors. In addition there are other fluctuations of various frequencies for which the foreseeable changes depending on the times of the day are one example.

These fluctuations in market demands require highest flexibility of the synthesis operation and this particularly when the synthesis is based on carbonaceous solids as the raw material. In this case, normally little or no gas is available to supply peak demands exceeding the normal gas output of the plant. However, the problem is appreciable even in synthesis plants based on natural gas, because the yield of liquid products is substantially fixed by the original design of the plant and may not be conveniently varied as a function of unforeseeable fluctuations in the market demand.

The liquid/gas ratio in the synthesis product may, as previously mentioned, be influenced to a certain extent by changing the character and/or composition of the catalyst, for example by using nickel as the catalytically active catalyst component or by changing the promoter content of iron catalysts, decreasing promoter concentrations being generally conducive to an increasing gas yield. However, the changes which may be secured in this manner are minor and rather cumbersome to achieve. There remains therefore a strong need for a convenient and efficient means for changing the liquid/gas ratio of the synthesis product as a function of fluctuations in market demand of any conceivable magnitude and frequency. The present invention fills this need.

It is therefore the principal object of the present invention to provide means for improving the catalytic synthesis of hydrocarbons from gas mixtures containing carbon oxides and hydrogen.

A more specific object of the invention is to provide means for increasing the flexibility of this process with respect to the liquid/gas ratio in the synthesis product.

Other objects and advantages will appear hereinafter.

It has been found that the selectivity of the synthesis reaction towards formation of gases or liquids is a function of the composition of the feed gas, provided all feed gas components participating in the synthesis reaction that is H_2, CO, H_2O and CO_2 are taken into consideration. In other words, extensive research work has demonstrated that the liquid/gas distribution of the synthesis product may not be controlled by a mere adjustment of the H_2CO ratio in the fresh feed but that all reacting gas components must be brought into a proper relationship.

More specifically it has been found that the gas/liquid distribution in the synthesis product at otherwise comparable reaction conditions is a function of the value of the ratio

$$R = \frac{H_2 + H_2O}{H_2 + H_2O + CO + CO_2}$$

where H_2, H_2O, CO and CO_2 are respectively the molecular proportions of hydrogen, water, carbon monoxide and carbon dioxide in the feed gas. This ratio may be defined by

$$R = \frac{H_2}{H_2 + C}$$

where the H_2 and C represent the molecular proportions of the total hydrogen and total carbon in the feed in whatever form the hydrogen and carbon are present. The effect of this relationship is that the selectivity to gaseous hydrocarbons increases as the value of R increases and decreases as the value of R decreases, while of course a similar but opposite relationship exists with respect to the production of liquid hydrocarbons.

Therefore, in accordance with the present invention the gas/liquid ratio in the synthesis product is controlled by controlling the value of R in the gas mixture entering the reactor. In the case of iron type catalysts these values of R may vary, for example, from about 0.2 for lowest gas and highest liquid production to about 0.9 for highest gas and lowest liquid production. The relationship is not a straight line function; its slope increases slightly as the value of R increases.

The existence of the above described functional relationships, which forms the basis of the present invention, is clearly demonstrated in Figures 1 and 2 of the accompanying drawing. The curves shown are the result of an evaluation of some 50 hydrocarbon synthesis runs carried out on different iron-type catalysts in dense phase fluid operation at reaction conditions varying relatively widely and falling within the approximate ranges given below:

Temperatures, °F.	600—690
Pressures, Psig	235—400
Fresh Feed Rates, V/W/Hr.	8—70
Recycle Ratio, V/V Fresh Feed	0.5—6

H_2:CO Ratio, Total Feed	1—7
Superficial Velocity	0.3—0.9
Average Catalyst Particle Size of fresh catalyst, Microns	20—200

Referring now to Figure 1, the curve shown therein is the correlation between R and the selectivity of the reaction to C_1+C_2 hydrocarbons in the product, i.e., the percentage of carbon contained in the product hydrocarbons and oxygenated compounds accounted for by C_1+C_2. It will be seen that gas formation increases rapidly as R increases. For example gas formation at a value of R of about 0.85 is approximately four times that at a value of R of about 0.25. It is of interest to note in this connection that no useful correlation between any other gas composition parameter and the amount of gas formed could be found. In particular there is no clear functional relationship between the simple H_2:CO ratio in the feed and the gas content of the product. Also, the ratio $H_2:H_2+CO+CO_2$, while correlating well in a large number of cases, does not satisfy all situations. For example when large quantities of water are added to control carbon formation or oxygen content of the catalyst, no satisfactory correlation exists between this ratio and the selectivity to low molecular weight hydrocarbons.

In Figure 2, selectivity to C_1+C_2 hydrocarbons is plotted against selectivity to liquid hydrocarbons, i.e., cc. of C_4+ hydrocarbons formed per cubic meter of H_2+CO consumed. R-values corresponding to some values of selectivity to C_1+C_2 hydrocarbons are likewise given. It can be observed that liquid product formation falls off sharply with rising values of R or selectivity to C_1+C_2 hydrocarbons. For example at a selectivity to C_1+C_2 of about 11, corresponding to a value of R of about 0.26 the liquid product formation is about twice as high as that at a C_1+C_2 selectivity of about 40 corresponding to an R value of about 0.87.

It follows that control of R is a reliable means of controlling gas/liquid distribution in the synthesis product. Any means suitable for changing the value of R may be used to establish the desired gas/liquid distribution in the product according to the market demand.

If it is desired to raise gas production and lower liquid production the hydrogen over CO ratio may be increased or steam may be added to the feed in once-through or recycle operation or CO_2 may be removed from the recycle gas in recycle operation until the value of R corresponding to the desired gas or liquid production is established. If it is desired to lower gas production and increase liquid production CO or CO_2 may be added in once-through operation or CO_2 scrubbing of the recycle gas may be reduced by partially or completely by-passing the CO_2 scrubber in recycle operation. Any suitable combination of these means may be employed.

Suitable values of R for high gas production for example for the production of volatile gasolines for use in winter operation are those above about 0.6, preferably above 0.65, and up to about 0.85 which permit an increase in gas production of about 100—300% and a decrease in liquid production of about 10—30% over conditions of peak liquid and low gas production. For high liquid and low gas production, for example for the production of gasolines for use during summer time R values of less than 0.65 preferably of about 0.3—0.6 should be used.

It has further been found that the effect of a control of the value of R on the gas/liquid distribution in the synthesis product will be even more pronounced when the promoter and oxygen contents of the catalyst, particularly of iron catalysts are properly adjusted and optimum temperatures are employed. Quite generally, low promoter contents, high oxygen contents and high temperatures have been found to be conducive to increased gas formation in combination with the higher ranges of R-values specified above. Any change of these variables in a direction opposite to those indicated will tend to influence the reaction toward a decrease in gas formation and an increase in liquid product formation. Manipulation of these additional variables is of greatest advantage in connection with the use of iron-type catalysts in fluid operation. For this case, conditions favorable to high gas formation are as follows:—

(1) Promoter Content, % K_2O on Pure Iron	<0.5
(2) Oxygen Content, % O_2 on Pure Iron	>20 and <32
(3) R	>0.6 and <1.0
(4) Temperature, ° F.	>650 and <800

These conditions may be met with a minimum of change in any hydrocarbon synthesis plant. In regard to item 1, the promoter content in the reactor may be controlled because catalyst will be added more or less continuously to the unit, and when it is desired to make a maximum amount of $C_1 + C_2$ hydrocarbons the catalyst with little or no promoter may be added to the unit. Of course, when a shift to low C_1 and C_2 production is desired catalyst with higher than average promoter content may be used to fortify the catalyst in the reactor. Regarding item 2, the iron catalyst normally becomes oxidized in operation, and reduction with hydrogen or other gases is employed to keep the catalyst in the reduced state. It is very simple to raise the oxygen content of the catalyst either by omitting hydrogenation or feeding an oxidizing gas, such as steam, CO_2, or O_2 to the reactor in any manner known *per se*. The desirable oxidation range is in the order of 25% oxygen on carbon and oxygen free catalyst. While the oxygen will tend to deactivate the catalyst this may be offset by employing higher hydrogen partial pressures and higher temperatures. Moreover, lower feed gas conversions are desirable because they themselves tend to give more $C_1 + C_2$ gases and any residual CO and H_2 will be desirable fuel constituents in the resulting fuel gas. Regarding item 4, higher temperatures of the order of 700° F. may be easily obtained by decreasing the cooling of the reactor.

The effects which may be accomplished by the present invention will be further demonstrated by the following specific example wherein some representative fluid-type synthesis runs are reported in detail.

EXAMPLE.

In the table below the catalyst designated " Ammonia Synthesis " was a fused and reduced high purity magnetite containing a small amount of alumina and the potassium promoter indicated. The catalyst designated " Pyrites " was a sintered and reduced pyrites ash promoted as indicated.

Run No.	I	II	III	IV	V	VI	VII	VIII
Catalyst	Pyrites + 2.5% KF	Pyrites + K_2CO_3	Pyrites + 2.5 KF	Ammonia Synthesis		Pyrites + K_2CO_3		Ammonia Synthesis
Reaction Conditions								
Temperature, °F.	587	644	651	666	655	634	632	650
Pressure, Psig	252	395	400	395	235	213	395	400
V./Hr./W.	6.7	23	14	18	45	55	28	38
Recycle/Fresh Feed	4.5	3.0	3.7	3.3	1.9	0.5	2.9	2.0
Superficial Vel., Ft./Sec.	0.27	0.84	0.32	0.88	1.4	0.82	0.85	0.52
Fresh Feed Composition								
% H_2	50.8	60.1	52.4	58.2	60.4	59.2	70.2	73.2
% CO	45.3	30.4	24.5	29.0	29.2	30.8	22.3	23.8
% CO_2	0.5	8.1	7.4	10.0	7.5	8.7	6.4	0.26
% H_2O	0.2	0.1	11.5	0.1	0.2	0.2	0.1	0.1
Total Feed, before reaching equilibrium conditions								
R	0.285	0.32	0.365	0.42	0.54	0.59	0.78	0.867
Hydrogen Partial Pressure, Psia	59	100	89	138	110	130	169	273
Total Feed, after reaching equilibrium conditions								
% H_2	20.8	21.6	20.8	31.0	41.7	56.1	40.4	65.5
% CO	16.7	13.5	8.0	15.7	16.9	27.5	7.6	8.95
% CO_2	38.3	39.2	29.3	30.2	21.0	12.1	3.9	1.05
% H_2O	1.32	2.8	3.4	2.7	2.3	1.1	0.93	0.35
H_2/CO Ratio	1.24	1.60	2.6	1.98	2.47	2.04	5.31	7.32
CO Conversion, Mol. %	95	98.4	99	93	92	97.2	99.7	99.5
H_2+CO Conversion, Mol. %	93	97.8	97	90	85	79	94.3	81
Yields, cc/m³ of H_2+CO Consumed								
C_3+	249	244	321	230	231	227	192	175
C_4+	209	198	184	176	185	172	123	119
400° F. Bottoms	59	35	25	24	—	30	11	—
Oxygenated Compounds in Aqueous Phase	27	39	49	38	23 (1)	27	38	—
Total Oxygenated Compounds	—	75	—	74	48 (1)	54	50	—
Selectivity to C_1 + C_2	10.5	12.15	13.7	14.82	17.95	18.8	32.6	39.5
% O on C— and O—Free Catalyst		16		18	12—20	13—8	10	
% C on C— and O—Free Catalyst		30		60.0	36—72	38—56	15—20	
% (0—20) Micron Fraction in the spent catalyst		55		50	11—50	61—64	54—74	
% K_2O on C— and O—Free Catalyst	1.8—1.4	1.2—1.0	2.8—1.9	1.8—1.4	1.7—1.0	0.9—1.3	—1.2	

The above detailed data prove the perfect correlation of the gas/liquid distribution in the synthesis product to the ratio R, largely independent of changes in other reaction variables.

In order further to illustrate the invention, a preferred embodiment thereof will now be described with reference to Figure 3 of the drawing, which shows in a simplified manner an essentially conventional system for carrying out a fluid hydrocarbon synthesis operation of the type employed in the runs of the above example.

In operation, fresh feed is supplied through line (1) to the bottom of reactor (10) which it enters through a distributing device such as a grid (3). Reactor (10) contains a finely divided iron-group metal catalyst, preferably an iron-type catalyst having a particle size of about 20—200, preferably 30—100 microns. The catalyst is fluidized by the upflowing gasiform reactants and reaction products to form a dense highly turbulent mass having a well defined upper level (L) and an apparent density of about 30—150 lbs. per cu. ft. Superficial linear gas velocities of about 0.3—3 ft./sec. are suitable for proper fluidization of the type of catalyst specified. Suitable temperatures of about 600°—750° F. are maintained by any conventional cooling means such as a cooling coil (5).

Gasiform reaction product is withdrawn overhead from reactor (10) through line (12), cooled in cooler (14) and passed to a liquid-gas separator (16). Water and oil are withdrawn downwardly from separator (16) through line (18) to be further treated by conventional separation and product recovery means (not shown).

The tail gas is passed through line (20) to a CO_2-scrubber (22) wherein its CO_2 content may be adjusted downwardly in accordance with the invention by scrubbing with water or any other suitable absorbent. The absorbent enriched with CO_2 is removed through line (24) and passed to a conventional regenerator, such as a flash reactivator (26) from which CO_2 may be recovered through line (28) if desired. Reactivated absorbent is pumped back by pump (27) through line (29) to scrubber (22) under any suitable pressure.

Tail gas of reduced CO_2 content is withdrawn overhead from scrubber (22) through line (30). It may either be passed to city gas mains or stored. At least a portion of the tail gas, however, is recycled through line (32) to reactor (10) for proper fluidization and adjustment of the desired R-value. Any desired portion of the tail gas in line (20) may be recycled directly to reactor (10) by means of bypass line (34).

An important feature of the invention is the scrubbing of the recycle gas to remove CO_2 to adjust the value of R whenever high gas yields are desired. When feed gas is produced in a coal gasification plant it will have about 1—1.5H_2:CO ratio. As a result, the ratio R will be below 0.5 at a recycle rate of about 1.3. However, this ratio may be improved by removing the greater part of the CO_2 from the recycle gas in scrubber (22). Then a ratio of 0.6—0.7 may be obtained and the selectivity to C_1 and C_2 hydrocarbons will be increased from about 20 to 40—50% of the CO converted. Since it is undesirable to have in the exit fuel gas large quantities of CO_2, which is made in substantial amounts at a low H_2:CO feed *ratio*, the CO_2 scrubbing described for the recycle gas is also preferably applied to the exit gas. Since the exit gas is also the recycle gas, the two scrubbing treatments may be accomplished in one scrubbing unit. This unit need not completely remove the carbon dioxide but may be an inexpensive flash CO_2 scrubber which will remove large quantities of CO_2 at a low investment cost. The CO_2 is removed from the synthetic fuel gas to increase the heating value and decrease the specific gravity of the gas.

A study of reaction rates in hydrocarbon synthesis using iron catalysts in a fluid reactor has shown that the rate of reaction is not proportional to the ratio of weight of catalyst to the feed gas but rather to the surface area of the catalyst. Various proposals have been made in the past, as in catalytic cracking, to vary the reaction rate by either increasing the temperature, replacing old catalyst with fresh catalyst or regulating the catalyst level in reactors. These proposals are not applicable to the hydrocarbon synthesis reaction because, here, the catalytic fluid reaction has a rate dependent on the surface area. In commercial operations it is desirable to hold the temperature constant to maintain product quality and heat transfer; the feed gas rate must be constant so as to maintain production, the pressure is set by the operating limits of the reactor, and the recycle rate is fixed by equipment requirements. This leaves the rate of reaction to be controlled by the amount of catalyst surface in the reactor.

It is undesirable to have too high conversion of the feed because this lowers the hydrogen partial pressure and causes carbon formation and catalyst disintegration. On the other hand, low conversion lowers the yield of product because less feed gas is converted and more of what is converted goes to methane. So, a narrow range of conversions is desirable say 93 to 96% H_2+CO converted. Again, if catalyst quality changes, there should be a compensation for this change.

It is pointed out that if carbon formation is initiated, it continues as a chain reaction. The finer catalyst of more surface formed by disintegration due to carbon formation causes higher conversion, lower hydrogen partial pressure and again more carbon formation and more disintegration and more catalyst surface. So, the cycle is repeated and soon the bed will be out of control because of the catalyst becoming too fine.

It has been found that this condition may be controlled by maintaining such a constant catalyst surface area in square feet in the reactor as is consistent with the desired conversion. If the conversion is too high and the hydrogen partial pressure of the reactor too low, as indicated by a recording analyzing instrument, the top catalyst may be removed through line (40). Segregation takes place in the reactor and the top catalyst will be the finest and have the greatest surface. This removal may be continued until the average catalyst sample from the middle of the reactor shows the desired screen size. This can also be detected by the density of the catalyst as indicated by pressure tap determinations up and down the reactor.

When catalyst is to be replaced to raise the overall activity, the coarse catalyst at the bottom may be removed through line (42) and replaced by equally coarse catalyst through line (44). Again, if the conversion is too low, coarse catalyst from the bottom of the reactor may be removed and replaced by finer catalyst. The coarse catalyst removed may be ground and used as future catalyst replacement. Fortunately, the bulk density also changes with changes in particle size, smaller particles having a lower bulk density. So, for constant conversion a constant volume of catalyst is desired. This results in about constant area of catalyst in the reactor. To accomplish this effect the level in the reactor is held constant.

Considerable emphasis has been placed in the preceding description on the use of iron-type catalyst in fluid operation. While these are the preferred conditions of the invention it is noted that the relationships explained and the embodiments of the invention described hold fully also for fixed bed operation and in a substantially analogous though quantitatively different manner for other iron group catalysts, particularly those of the cobalt type.

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

1. A process for synthesizing normally liquid and gaseous hydrocarbons by the catalytic conversion of a gas mixture containing carbon oxides and hydrogen in synthesis proportions under synthesis conditions of temperature and pressure which comprises adjusting the gas liquid distribution in the reaction products to a desired ratio by adjusting the ratio of hydrogen to hydrogen plus carbon, i.e.

$$R=\frac{H_2}{H_2+C}$$

as defined herein, in the feed gas mixture.

2. A process as claimed in claim 1 wherein the gas content of the reaction products is increased by increasing the ratio of hydrogen to hydrogen plus carbon in the feed gases.

3. A process as claimed in claim 1 wherein the liquid content of the reaction products is increased by decreasing the ratio of hydrogen to hydrogen plus carbon in the feed gases.

4. A process as claimed in claims 1 or 2 wherein the ratio is adjusted by reducing the carbon dioxide content of the feed gas mixture.

5. A process as claimed in claim 4 wherein the feed gas also contains recycled gas and the carbon dioxide content is reduced by removing the carbon dioxide from the tail gas recycled.

6. A process as claimed in claims 1 or 2 wherein the ratio of hydrogen to hydrogen plus carbon is adjusted by adding water to the said gases.

7. A process as claimed in any of the preceding claims wherein the catalyst is iron and the ratio of hydrogen to hydrogen plus carbon in the feed gas is maintained between 0.3 and 0.9.

8. A process as claimed in claims 1 or 2 or any of claims 4 to 7 wherein a high gas content in the reaction product is obtained by adjusting the ratio of hydrogen to hydrogen plus carbon in the feed gases to be greater than 0.6, preferably over 0.65.

9. A process as claimed in any of claims 1, 3 or 7 wherein a high liquid content in the reaction product is obtained by adjusting the ratio of hydrogen to hydrogen plus carbon in the feed gases to be between 0.3 and 0.6.

10. The process as claimed in claims 1 or 3 or any of claims 4 to 8 in which the

catalyst contains an alkali metal promoter and an appreciable amount of oxygen combined with the catalyst metal and the amount of hydrocarbon gas in the product is increased by decreasing the amount of promoter present and/or increasing the amount of oxygen in the catalyst and/or the temperature of the reaction.

11. The process as claimed in any of claims 1, 3, 7 or 9 in which the catalyst contains an alkali metal promoter and an appreciable amount of oxygen combined with the catalyst metal and the liquid content of the reaction products is increased by increasing the amount of promoter present in the catalyst and/or decreasing the amount of oxygen combined with the catalyst and/or the temperature of the reaction.

12. A process as claimed in claim 10 wherein the ratio of hydrogen to hydrogen plus carbon in the feed gas is maintained above 0.6 and a fluidized iron catalyst is used containing less than 0.5% of promoter based on the weight of the pure iron and more than 20% by weight of oxygen combined with it and the reaction is carried out at a temperature above 650° F.

13. The process as claimed in any of the preceding claims in which said catalyst is present in the form of a dense turbulent fluidized mass of finely divided solids.

14. The process for adjusting the gas liquid distribution in the reaction products in the synthesis of hydrocarbons from mixtures of carbon oxides and hydrogen as hereinbefore described.

Dated this 31st day of December, 1948.
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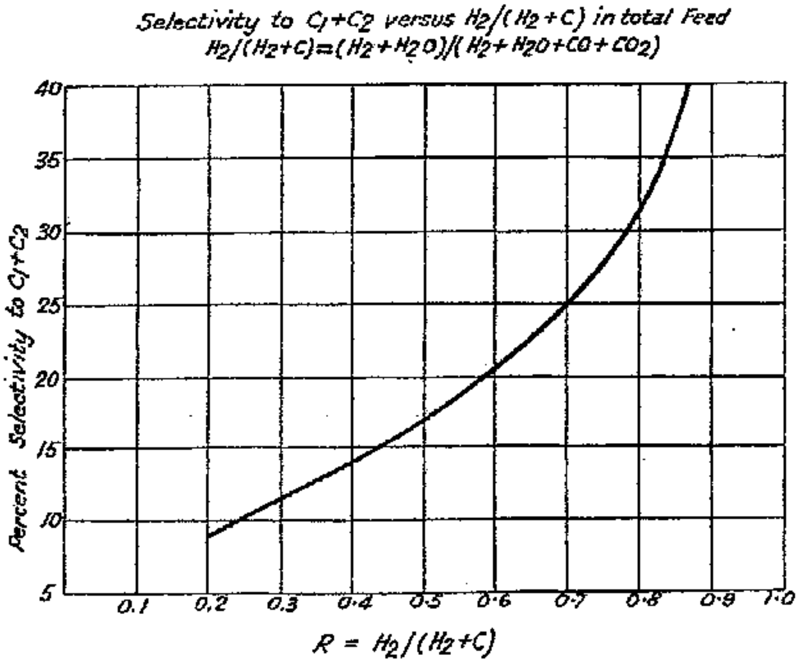
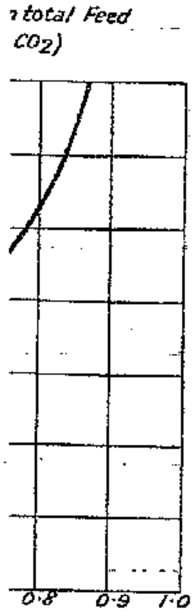

Selectivity to C_1+C_2 versus $H_2/(H_2+C)$ in total Feed
$H_2/(H_2+C)=(H_2+H_2O)/(H_2+H_2O+CO+CO_2)$
Percent Selectivity to C_1+C_2
40
35
30
25
20
15
10
5
0.1
0.2
0.3
0.4
0.5
0.6
0.7
0.8
0.9
1.0
$R = H_2/(H_2+C)$

FIG. 1.



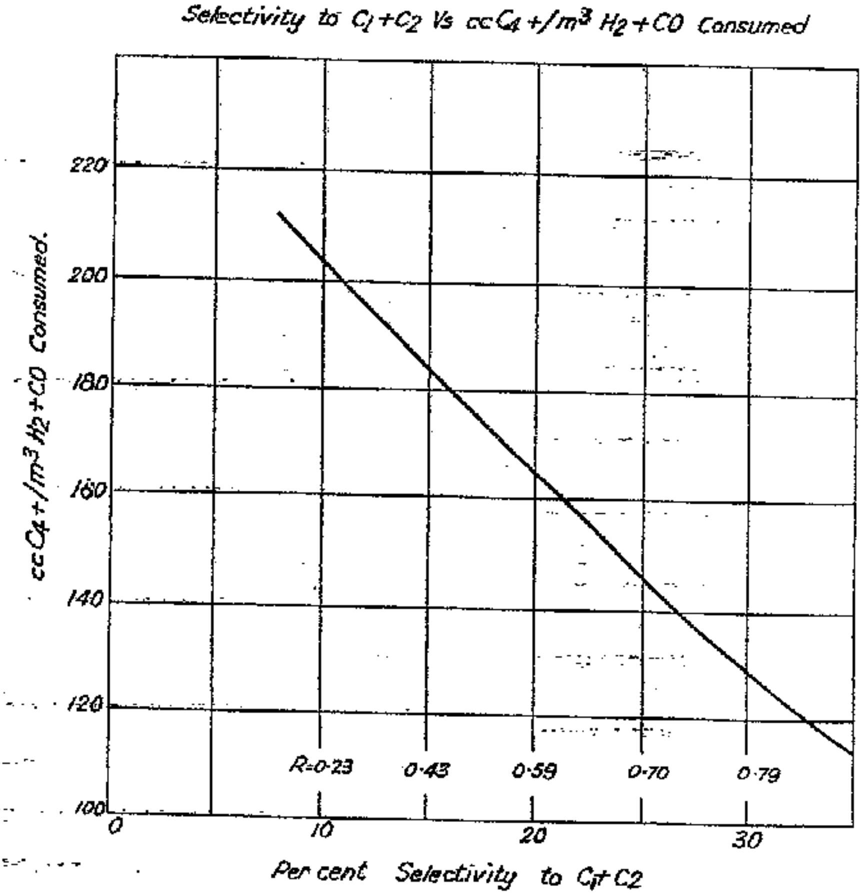


FIG. 2.

H.M.S.O. (M.F.P.

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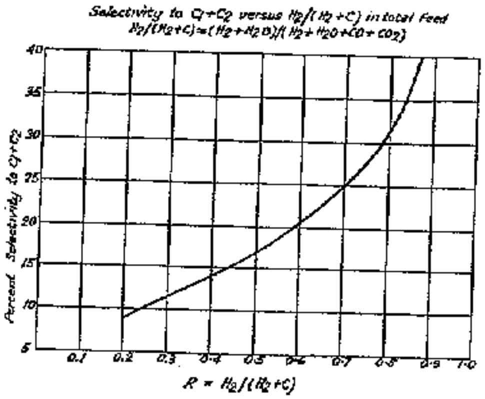

FIG. 1.

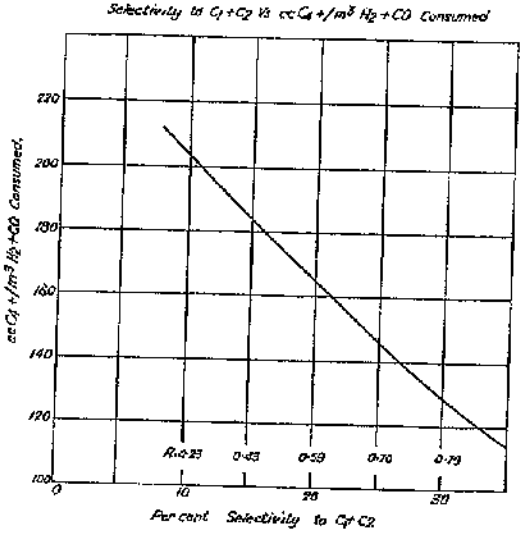

FIG. 2.

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FIG. 3.