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HYDROCARBON SYNTHESIS WITH AN IRON CATALYST By Kolbel

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Introduction

It is hardly necessary for me to explain to my audience the importance of this topic, nor the intensive work and the great interest which has been created since the gasoline synthesis by Franz Fischer and mans Tropsch has resulted in a successful industrial application. The excessively rapid scientific and industrial development of the cobalt catalyst has not made us forged that the use of cobalt as a catalyst makes us dependent on its importation from abroad or the colonies, and is uneconomical. I don't know whether the 10% loss of cobalt used as a catalyst in a single circuit is covered by the German production, but it is certain that the total German cobalt requirements go 'principally in the steel industry, and are in no way covered by domestic production. As long as this is the case, dependence on cobalt is a handicap and the further development of the Fischer-Tropsch synthesis, which is a key process, and a starting point for an entirely new organic chemical industry, the development which we may be able to measure by the immense development of the inqustry in America based on a further applications of petroleum for the production of the greatest variety of products. A further danger, caused by war conditions, consists in the necessity of centralization of the manufacture of the catalyst because of the high value of cobalt and of thorium, which also will be eliminated by changing to iron. The catalyst developed by us is simple to prepare with simple equipment and can be done in the future at the synthesis works.

It must further be remembered that changing the catalyst from cobalt to iron will greatly reduce the cost of the catalyst. These are not the only reasons for the trend of the development of the gasoline synthesis catalyst in the direction of the application of iron. The greatest attraction does not consist in the simple substitution of cobalt but rather in the possibility of synthesizing more readily convertible and cheaper products than can be done with cobalt. This applies also to the gasoline synthesized with iron, which can be considered as being useful as such, unlike the cobalt gasoline.

The problem of synthesis of hydrocarbons with iron catalyst is by no means as novel as it is assumed by many. Franz

Fischer himself has continued the development of iron catalyst with his characteristic stubbornness ever since his conception of the idea of preparation of oil from coal by synthesis of CO and H₂—containing gases. In fact he was first to produce in 1925 at atmospheric pressure the homologs of methans from carbon monoxide and hydrogen using iron catalyst. The work with the iron catalyst has been continued even today, in spite of the high stage of development of the cobalt-thorium catalysts, and without regard to the

existence of fundamental disadvantages of iron catalyst against cobalt and nickel catalyst which were believed to exist. efforts were continuously started anew, because of the realization that iron plays in fature as well as in the arts a more important role as a catalyst, than cobalt and nickel. Finally, many industrial catalytic processes involving the use of costly. metals have been changed over in the course of the development to the use of iron. Numerous patents of the I. G. Farbenindustrie lead one to assume that they have worked for a long time on the use of iron catalyst for the reduction of carbon monoxide and the formation of hydrocarbons. The results there recorded can, however, not be experimentally substantlated in spite of the many earnest efforts, nor do they equal as far as is known to the results obtained by Fischer and his students. We further are familiar with different work in Japan which consisted in almost painfully accurate reproduction of the principal features of Fischer's publications, but which have brought out nothing new beyond the results obtained by KWI. French scientists have occupied themselves intensively with the physical investigation of iron catalyst. The X-ray and thermo-magnetic methods used by them are highly original and very interesting, but they must be used with the greatest circumspection according to the results of our experimental work in this field.

Fundamental Problems with the Iron Catalyst

The reason for the slower development of the iron catalyst in comparison to the nickel and cobalt catalyst is found basically

in specific problems and difficulties offered by iron in comparison with the other two metals when used as catalysts. We know that in the series iron-cobalt-nickel, the activity increases with respect to reduction of CO to hydrocarbons. Of these three elements, when considered for the technical hydrocarbon synthesis, iron offers the greatest attraction, but also fundamentally presents the greatest difficulties because it has the least activity. This finds its expression in the reaction temperatures with the different catalyst metals: at atmospheric pressure, the hydrocarbon synthesis can be carried out at 160°C with nickel, at 170-180°C with cobalt, and only at 225-250°C with the iron catalysts known today. The necessity of using higher temperatures with iron introduces a multitude of difficulties and problems:

- 1) The formation of gaseous products is greatly favored by rising temperatures, i.e., the liquefaction becomes poorer.
- 2) The formation of secondary products, e.g., through the decomposition of the high molecular weight primary paraffins, is greatly favored by rising temperatures. The types of secondary products, e.g., from the high decomposition molecular weight primary paraffins affect very unfavorably the life of the catalyst.
- 3) The separation of carbon from the carbon monoxide, the so-called carbon deposition, becomes greatly increased at higher reaction temperatures, which will threaten great technical difficulties, especially in the synthesis in the middle pressure range.
- 4) The synthesis equipment is the more difficult to construct and is the more expensive, the higher the reaction temperature. Here we to persist in leading away the heat of the reaction

with water, definite technical limitations will be imposed by the reaction temperatures. The empirical experience accumulated with cobalt and nickel as catalysts, cannot be used for the solution of these basic problems, because iron catalyst differs from the two brother elements by special conversion properties during the reaction.

The cobalt and nickel can cause two kinds of reduction on carbon monoxide:

- 1) Oxygen of the carbon monoxide may be split off as water during the formation of hydrogarbons. (CO + 2 H₂ = CH₂ + H₂O).
- 2) The oxygen in the carbon monoxide can be used for the production of carbon dioxide with the formation of the unwanted methane. (2 CO + 2 H_3 = CH_6 + CO_2).

Iron, however, offers a third and principal kind of reaction, in which oxygen is used in the formation of carbon dioxide, with the simultaneous formation however of hydrocarbons (2 CO + H₂ = CH₃ + CO₃). We might mention here in parenthesis that Fischer and Tropsch's statement in their collected work on the study of coal, that the iron and copper catalyst always cause the reaction to proceed by way of formation of carbon dioxide, and without formation of water, does not agree with our observations. We have developed iron catalyst in which there was a considerable amount of water formed during the carbon monoxide reduction, which therefore approached quite closely to the ideal case already stated by Fischer and Tropsch in 1926 of the use of water was products, using equal parts of carbon monoxide and water according to the equation 3 CO + 3 H₂ = CO₃ + H₂O + CH₃. We do not wish to discuss this

any closer at the present, because our work in this direction has not yet been completed.

The composition of the gas must always correspond to the reaction; when water is formed, the proportion of CO:H₂ must be 1:2; 2:1 when carbon dioxide is formed, and when both reactions occur the proportion should be 1:1, i.e. the proportion such as exists in the usual water gas (blue gas). Naturally the nature of the hydrocarbons formed will be different depending on the operation. When an iron catalyst is used with carbon monoxide rich gas, with the production carbon dioxide, products very high in olefines are obtained; when water is formed with hydrogen-rich gas, the products assume a more saturated character. These possibilities of variations differentiate iron synthesis from the cobait and nickel catalyst, and are in themselves exceedingly desirable, but require overcoming considerable difficulties.

After having briefly discussed the problems met during the development of the iron catalyst, I will proceed with communicating some results of our own work in this field, carried out in Rheinpreussen with Dr. Ackermann and later with Dr. Ruschenburg. I will not overload you with details of the work which has lasted for nearly four years, and will limit myself for the sake of brevity principally to the communication of the results.

Investigation

The purpose of our work did not consist merely in finding a substitute of iron for cobalt, but to develop the hydrocarbon
synthesis with iron to such an extent that it would no longer be
a mere substitution for the synthesis with cobalt, but would

represent actual technical and economic improvements. Our work began in Rheinpreussen in the spring of 1937, initially in two directions, in the continuation of the work begun at KWI at atmospheric pressure, and connecting it to the "synthol" work of Franz Fischer done in 1922 to 1924, using higher pressures for the production of anti-knock gasoline. The resumption of the work with iron catalyst appeared to us very promising because in the past ten years of development of synthol, considerable progress had been made with iron catalyst, which had not yet been used at increased pressures.

At the time we started our work, the status of development was given in the publications of Franz Fischer and in the work of K. Meyer with an iron - copper catalyst containing 25% copper, using a synthesis gas containing 28% CO and 56% He, and who obtained 55 g liquid hydrocarbons/nobm during 4 - 5 weeks operation. The conversion without exception proceeded by way of carbon dioxide formation, the temperatures were 228-235°C. The. GO: Ha proportion of 1:2 used in these tests was the most unfavorable imaginable for work with this catalyst, and the reaction by way of carbon dioxide required the reciprocal proportion of 2 00 to 1 H2. 95 g/obm was the theoretical maximum yield with the gas of composition selected, CO:H2 = 1:2. No success was obtained working with carbon monoxide-richer gas, because the action of the catalyst dropped off very rapidly after an initial high yield at normal pressures. Attempts to conduct the atmospheric pressures synthesis by way of water formation were also unsuccessful in the tasts performed at the KWI.

Our results with the atmospheric pressure synthesis with iron catalyst consisted at that time in attempts to increase its life and accordingly also the yield over longer periods, using a catalyst of much simpler composition, and accordingly also much cheaper. We were also successful in working with a gas containing more carbon monoxide, without harming the catalyst. This success was achieved by avoiding side reactions, with the production of substances which caused a gradual poisoning of the catalyst. However, even in spite of the appreciably higher yields we obtained in our work at a mospheric pressure, it still remained much below the liquid product yield of the catalyst. This stage of development of the atmospheric pressure synthesis with the iron catalyst is of little interest to the outsider, and I will refrain from giving any details. The work was continued and no detailed information was given when positive results were obtained.

We considered at the beginning of our investigation that the iron catalyst could become of practical interest only after the yields could compete with the yields obtained with cobalt catalyst. This, however, was again possible only if the CO:H2 ratio was selected to correspond to the operation of the iron catalyst, or conversely when the work of the iron catalyst was adapted to the hydrogen-rich synthesis gas. I.e., our task became to find an iron catalyst with which operations with carbon monoxide - rich gas became possible, or else operating conditions in which the iron catalyst operated like the cobalt and nickel catalyst, i.e. by way of water formation.

The very first pressure experiments with our own iron catalyst, which we began in the spring of 1937 and were admittedly based upon Fischer's synthol work, showed two striking results, which opened the possibilities of satisfying both requirements when working under pressure. With an appreciably less active catalyst we found during these first tests that when operating with water gas (1 CO:H₃) a surprisingly large amount of water was formed at pressures of 7-9 atmg and a temperature of 210°, with an almost complete utilization of the carbon monoxide. amount of water formed was up to 40 g/cbm, and the drop in efficiency of the oatalyst was barely perceptible in spite of using water gas. The higher pressure apparently overcame the harmful influence of higher concentration of CO and the hope seemed justifled that water formation will be considerably increased when water gas was used under pressure. On the strength of this experience, we continued our experiments within the pressure range between 5 and 100 atmg using high-carbon monoxide as well as high-hydrogen gases. We would obtain up to 80 g of water per cbm synthesis gas with hydrogen-rich gases at medium pressures of 7-15 atmospheres with a formation of 85 g of liquid and solid products, almost 30% of which consisted of paraffin. Up to 1/3 of the conversion proceeded by way of water formation. We have got still better results using other catalysts. I would, however, not like to talk about it at present, because the work has not been completed.

The work with carbon monoxide-rich gases was appreciably more successful. We have succeeded right from the start in obtaining

yields which were throughout equivalent to the cobalt yields, by changing over from atmospheric pressure to middle pressure and using a simple metal iron catalyst and a gas consisting of three parts 60 to two parts H.. The life of these catalysts seemed almost unlimited, and appeared even inconveniently long for a rapid development. Damage to the catalyst by the high carbon monoxide content was therefore overcome by the use of pressure. The products were water clear, colored slightly yellow and, always entirely free from crystallized paraffin. Franz Fischer made a report in December 1937 on the middle pressure synthesis which was the first preliminary communication on the iron catalyst pressure synthesis and which was in a surprisingly good agreement with our results.

We developed the following hypothesis from the unexpected fact that the harm done by the casalyst at high carbon monoxide concentrations and at atmospheric pressure disappears when the pressure is increased. The formation of hydrocarbons on the catalyst metal of the eighth group of the periodic system was caused according to Franz Fischer on two competing reactions of carbide hydrogenation. The carbide formation at atmospheric pressure is apparently easier with iron catalyst than the hydrogenation.

A minimum partial pressure of hydrogen seems to be required for a successful operation of the synthesis in order to hydrogenate the carbides formed from carbon monoxide and to re-form sufficiently rapidly the active centers of the catalyst through the re-formation of carbides. If the absolute hydrogen pressure; i.e., the hydrogen concentration is below the minimum, all the active centers of the

catalyst will become blocked by the carbide formation, and the activity will quickly drop. The minimum pressure of hydrogen is 0.5 atm abs when working at atmospheric pressure. When water gas is used, with 60% CO conversion, the pressure in the tail gas is already too low.

while using gas with still higher CO content the concentration is too low even in the synthesis gas, the catalyst becomes "carbided" or carbonized, and its activity drops rapidly. When operation at higher pressure, e.g., at 10 atmg, and with a 60% conversion of CO, the hydrogen pressure using water gas remains 3 atms abs. When the starting gas contains 3 CO:2 H2, 1 atm abs will still be present with a CO conversion of 91%; or with a gas of 2 CO:1 H2, C.5 atm abs will remain with 81% conversion. When operating with a hydrogen pressure or about I atm abs, as happens with a sas which consists of 3 CO:2 Ha at a pressure at 10 atmg, we may expect not merely a high conversion, a high amount of liquefaction, but also a long life of the catalyst. This hypothesis presupposes that the ability to form carbides does not increase to the same extent at increased pressure, as the hydrogenation ability. Such is the case when the iron catalyst is reduced (formed) at atmospheric pressure before operations at increased pressures. According to experience a maximum activity may also be attained under such conditions. If the catalyst is immediately started under full middle pressure, it hardly reaches full activity, and the carbide formation exceeds the hydrogenation. We realize that our hypothesis is not entirely free from contradictions, but it permitted us to explain the surprising differences

in the behavior of the catalysts at atmospheric and under middle pressures, and we owe to it many instances when using it resulted in a practical success.

Results

I. Catalyst

ment of a suitable catalyst, of which we have brought out several types suitable for different purposes. For the sake of brevity I will report here only on the single-substance catalyst with which we operated the longest. As the name implies, the catalyst contains none of the usual additions, such as copper, manganese, aluminum, etc. It consists essentially of iron, and its activity is the result of a special method of preparation which causes the formation of a definite modification of iron oxide. The discovery of this catalyst which excelled in simplicity and operation was the result of our application of the most modern methods of catalytic chemistry, which has to our knowledge not yet been applied to the study of the catalytic processes of gasoline synthesis.

Is. Mechanical Properties

Purely in outward appearance the catalyst looks somewhat like the silica gel in structure, and has the same extraordinary hardness and mechanical strength. The catalyst does not lose this strength during operations. The iron catalyst offers advantages over the present cobalt catalyst in its mechanical properties. Its filling weight is exceptionally large, and the reactor efficiency, referred to space, is 2 1/2-3 times as large with this iron single substance catalyst than with the present cobalt catalyst.

Preparation

The preparation of our iron catalyst is equally surprisingly simple as its composition. We already have prepared it on
a semi-technical scale in batches of 10 kg and over and obtained
reproducible highly active catalysts for several years. It is
obvious that with such cheap starting material and simple method
of preparation the catalyst costs play a very subordinate role.
Their importance sinks still further when considering the life
and the yields obtained with it. At the very start the reproducibility of the iron catalyst offered great difficulties, which
as far as 30 know have not yet been overcome in kWI for Coal Studies.
They depend on apparent accidents in the preparation of the catalyst, which makes the work much more difficult and its technical
realization still questionable. We have realized the cause of
these difficulties and eliminated them a long time ago. The reproducibility of good iron catalysts is no longer any problem to us.

Yields and Life

Other standards must be used when indicating the yield and the life of our iron catalyst than with the atmospheric pressure cobalt catalyst, because the life of our iron catalyst is not measured in weeks and months but in years. Referred to active gas ingredients the total yield in gasol, liquid and solid products is on an average 145 g/ncbm during the first one-half year. After one year's operation this total yield dropped on the average to 143 g. After one and one-half year's operation 132 g total products were still obtained per ncbm of active gas ingredients.

These data refer to a one stage process without circulation and without intermediate regeneration. When several stages are used, or the gas circulated, these yields can be still further increased according to our experience. The final life of our iron catalyst cannot yet be indicated, because our first catalysts have been seriously damaged in their activity after 15 months of operation by outside influence - failure of the temperature regulator, freezing of the gas line, etc. The catalyst with the longest operating time is still in use although it also has been damaged, and gives today after 19 months of operation a water clear liquid product including gasol of a total amount of 130 g/cbm active ingredients. The data and peak production have no meaning with the iron catalyst, but under special conditions a greater yield up to 160 g/cbm active gas ingredients can be obtained.

Degree of Liquefaction

Formation of Methane and CO Conversion

The carbon monoxide conversion could be maintained during the first 6 months of operation at 92%, with a degree of lique-faction of 73% and methane formation of 9% of the carbon monoxide converted. With increasing operating time the products formed by the iron catalyst are changed in the direction of formation of lighter boiling and gasol hydrocarbons. All these data are given for a single stage process.

Space Utilization or Space Velocity

A space velocity of up to 270 is obtained because of the great density of our iron catalyst with a normal gas load, i.e.

in one hour the gas volume is 270 times greater than the reactor volume. We may give for purpose of comparison the space velocity during the atmospheric pressure cobalt synthesis which reaches a maximum of only 150 with the single stage process, i.e. the gas volume is 150 times that of the reactor volume. The two stage process commonly used at present industrially reduces the space velocity to only 70 if the total gas converted is referred to all the reactors used. The space utilization is 3.5 to 3.8 times as high with the iron catalyst as with the cobalt catalyst at atmospheric pressure. This peak load is usually reduced in the course of operation, and the average space velocity is only equal to 170-190.

Synthesis and Operation Conditions

One great advantage of the iron catalyst in comparison with the cobalt catalyst is found in that it need not be reduced with hydrogen, which offered very great difficulties at first and can only be done today at a great expense. The starting of the iron single substance catalyst, as well as our other iron catalyst is done under certain readily re-producible conditions with the synthesis gas to be used in the synthesis. This "forming", as we call our starting, is very important for the activity of the catalyst. A perfectly definite carburization condition is important for the proper operation of the iron catalyst, and it differs quite considerably from that required for the atmospheric pressure synthesis.

The recognization of the proper degree of carburization is, however, very simple. I will emphasize here that we are not

forced to forming at low pressure with carbon monoxide and at high temperatures for the production of our better catalyst, as is done at the KWI.

Depending on the operating conditions, the synthesis temperature of the catalyst varies between 210 and 260°, the synthesis pressure between 5 and 20 atms, normally in a much narrower range of between 9 and 15 atms. The optimum gas composition, when operating with the iron catalyst by way of carbon dioxide formation, is approximately 3 parts CO:2 parts H₂.

I am giving here analyses of the starting gas and tail gas with a single substance catalyst when operating by way of formation of carbon dioxide.

	Starting Gas	Tail Gas
COa	4.5	64.2
Hydrocarbons	0.10	3.6
03	0.0	0.0
CO	54.6	6.2
H _a	35.4	6.5
CH ₄	0.6	9.7
C-number	1.0	1.78
Na	4.8	9.8

Properties of the Products

In my discussions of the products formed with the single substance catalyst I will limit myself to such which are formed in the operations by way of carbon dioxide formation. The data referred to the average product of a one year operating period. The specific gravity of the total yield of the liquid products was 0.710 to 0.715.

The boiling range was as follows:

54% 63%	ру	weight	of "	liquid	products	
67%	11	H	ŧŧ	, #	Ħ	-150°C -165°C
73-75%	12	Ħ	Ħ	t#	ti	-195°C

The olefine content of the fractions vary with the decreasing boiling point between 45 to over 70%. Up to 7% alcohollike substances are found in the fractions to 150°C. The motor properties, as expressed by the octane number of the stabilized fractions, are:

-120°C	o.n.	85
-150°C	Ħ 、	76
-165°C	Ħ	73
-195°C	ıi	66

These figures show that the gasoline with the iron catalyst may justly be called that, unlike the cobalt gasoline the octane number of which is incomparably lower. No cracking of the hydrocarbons obtained with the iron catalyst is required. We attribute these high octane numbers to the relatively low boiling point, to the higher iso -hydrocarbon content, to the presence of alcohol-like substances and to the high olefine content. The iron gasoline fulfills all requirements on purity as well as the cobalt gasoline. The gasoline clefines are as harmless with reference to gum formation when formed with the iron catalyst as the cobalt olefines. The motor behavior is perfect, I have used the gasoline obtained with iron catalyst for many months. The diesel fuels show good inflammability which varies depending on the boiling point of the fraction between 55 and 72 cetane number, which corresponds to 71 to over 100 cetene.

The gasol which comes over with the above products contains over 50% of Ce hydrocarbons and is composed of 60-75% of olefines.

Equipment

The results communicated here were obtained in laboratory as well as on a pilot plant scale. The pilot plant tests have been emphasized right from the very start, because the difference between the laboratory synthesis and large scale equipment is too great, according to the experience with cobalt synthesis, to permit drawing any conclusions. Our pilot plant equipment has an hourly production of around 10 cbm gas thruput, and offers therefore, already, the possibility to appreciate the technical difficulties to be experienced in large scale operations, if the construction of the pilot plant reactors is to be retained in industrial equipment. This experimental installation has now been used without interruption for over two years. The individual catalysts have not been used for longer than seven months to save time. During that time the results obtained were similar to those obtained in the laboratory. Moreover, the pilot plant tests gave us a better insight into the course of the reaction, in particular with respect to the heat effects. paid particular attention to the reactor construction in the development of our synthesis, because the technical ripeness of the iron catalyst synthesis is merely a question of the proper reactor construction. We have already mentioned that the high reaction temperature offers one of the principal problems in the synthesis with

iron catalysts. Mastery of these temperatures introduces difficulties which are aggravated by the high space velocity and the high catalyst thickness, making sufficiently rapid elimination of the heat of the reaction a very serious problem. This rapid leading away of the heat of the reaction is far more important with the iron catalyst than with the cobalt catalyst because the dangers of carbon deposition and of the reactor plugging up is here much greater. Were one to retain the usually used removal of the heat of the reaction by means of water, pressures of up to 50 atms gauge are to be expected in the cooling part of the reactors when using the same reactors as heretofore, which can be overcome industrially but will greatly increase the cost of the reactors. On the other hand it had been suggested to substitute oil cooling for the high cooling water pressures, as has been done by Franz Fischer in his first gasoline synthesis reac-This does not appear a satisfactory solution to us, because a heat exchange system cutside the reactor cannot materially reduce its costs in comparison to direct water cooling at high pressures; we were also afraid that even with high circulation velocities of the cooling oil its heat absorption properties would be insufficient to produce satisfactory and sufficient heat removal. started out with the idea that water was the ideal cooling agent in every respect, particularly for the gasoline synthesis reactors, and we have developed a reactor in which the heat of the reaction is taken away only by the evaporation of water, but in which the pressure of the synthesis reactor steam boiler system exceeds

10 atms gauge with a synthesis temperature of 210-260°. This type of reactor is still in the process of development, and I must therefore omit giving more detailed information on it.

Entirely independently from the above we have also tried another method which seems to be suitable to overcome the danger of the carbon deposition or reactor plugging up as well as for the mastering of the high temperature and high heats of the reaction. This method consists in using the iron catalyst synthesis in the liquid phase, i.e. in a sludge or emulsion of the catalyst in the synthesis oil. This process offers advantages both with respect to operations and other construction of the reactor: the intimate contact of the cooling medium with the catalyst with the complete elimination of the wall separating the two produces ideal heat exchange conditions, under which the overheating or the carbon deposition; as well as the plugging up are almost completely excluded. The suggestions for carrying out gasoline, synthesis in the liquid phase made by the KWI in Muelheim which are known to me, appear little promising at least from the standpoint of construction. We believe to have achieved important progress particularly in the construction of our liquid phase reactor because of its exceptional simplicity. Naturally, the synthesis in the liquid phase introduces numerous new problems, so that we cannot as yet make any definite reports on it, particularly because carrying out of these tests will require very much time with the exceptionally long life of our catalysts. We can, however, already say today that we have reached practically the

same conversion in a liquid phase synthesis in our reactors, as in the Vapor phase synthesis.

I have already mentioned that hydrocarbon synthesis with iron catalysts offers a few additional problems in gas production, purification, scrubbing, reintroduction of the off-gas carbon dioxide, etc. However, I seem to have used up too much time for discussion of these problems and must therefore come to a close.

I have already emphasized that the importance of hydrocarbon synthesis need no longer be discussed. The rapid development of gasoline synthesis by the Fischer-Tropsch methods, especially in the direction of working up of the primary product which may produce shortly gasoline as a by-product, deserves particular attention with the iron catalyst, because the products obtained from it are high in unsaturated compounds even in the high boiling fractions, which offer new possibilities in their working up in comparison with the cobalt products. I would not like to conclude my communication without acknowledging the support of our work by Director Kost or without expressing thanks to my collaborators, in particular to Dr. Ackermann.

/s/ Kölbel