April 30, 1940. H. GOETHEL ET AL 2,199,200

MANUFACTURE OF LUBRICATING OILS

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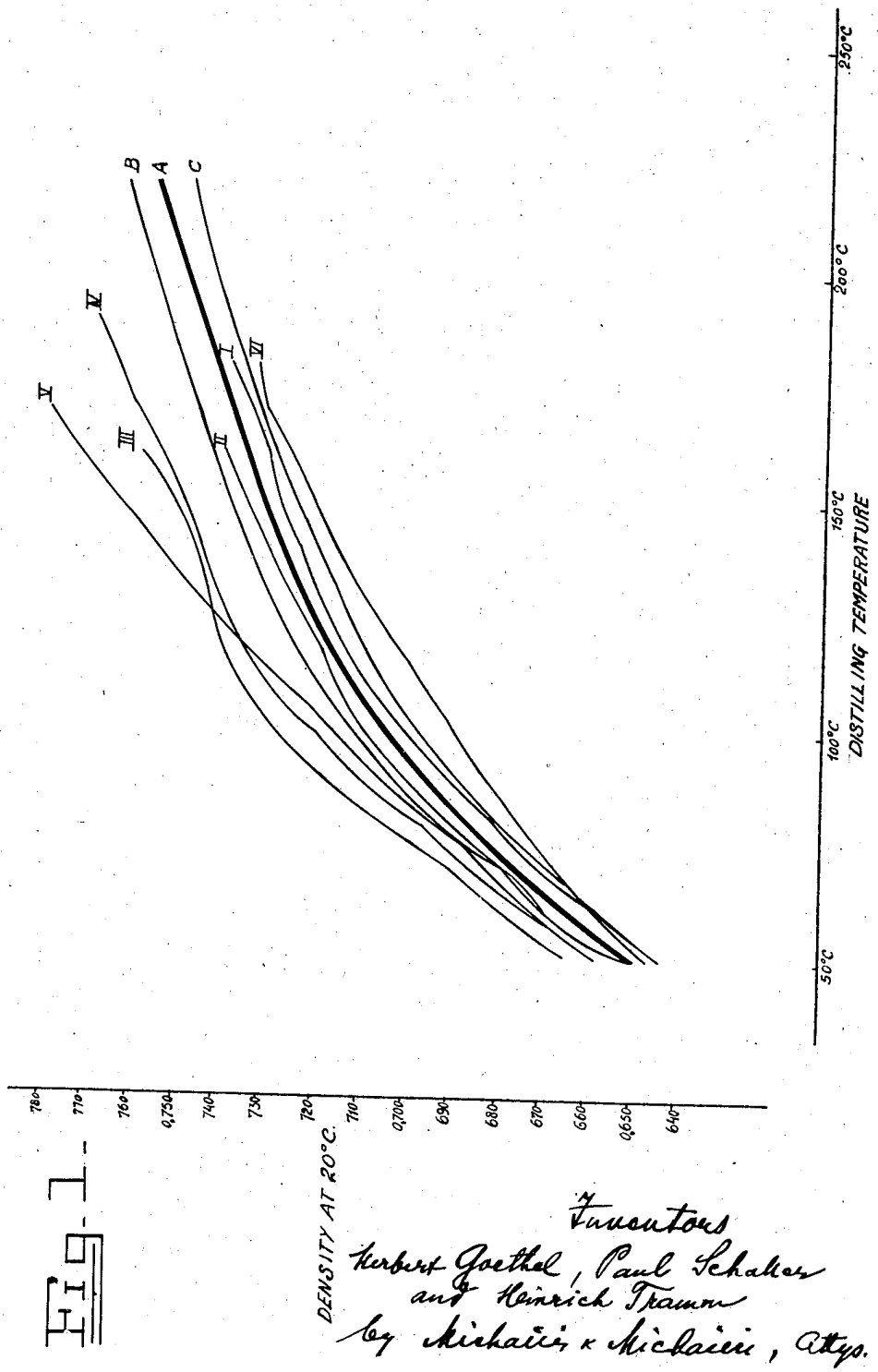

FIG. 1

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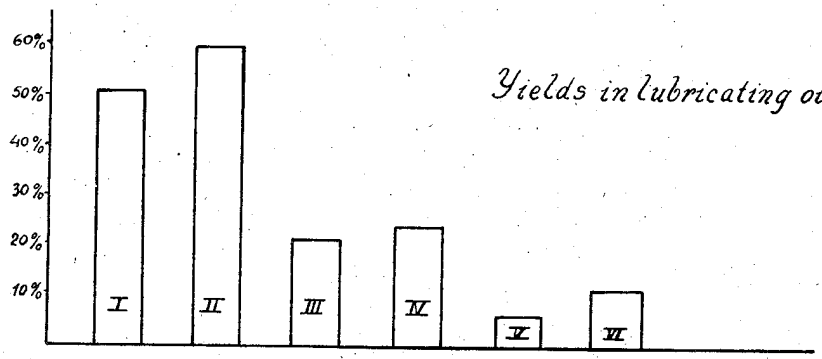
Fig. 2



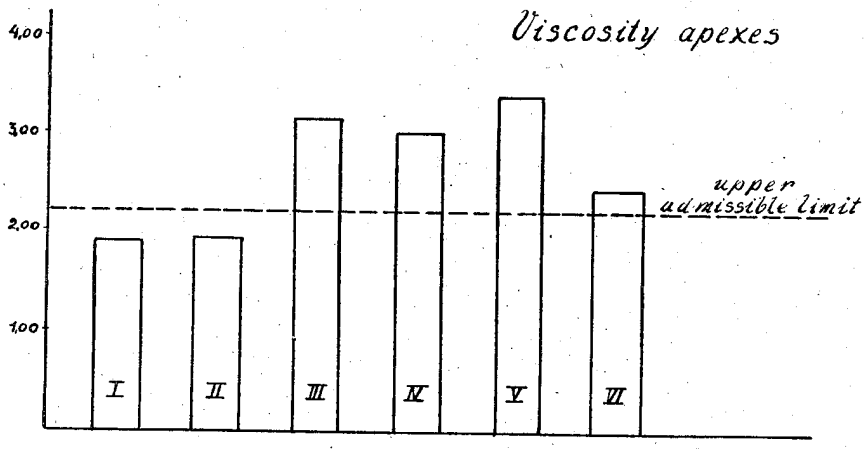


Fig. 3.

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UNITED STATES PATENT OFFICE

2,199,200

MANUFACTURE OF LUBRICATING OILS

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Application October 13, 1937, Serial No. 168,794
In Germany October 24, 1936

5 Claims. (Cl. 196—78)

Our invention relates to lubricating oils and more particularly to an improved method of manufacturing same.

It is an object of our invention to produce lubricating oils by polymerizing unsaturated hydrocarbons, such as olefines, or hydrocarbon mixtures containing same, by means of a polymerizing catalyst.

It is another object of our invention to improve the manufacture of lubricating oils as disclosed in the copending applications for U. S. Letters Patents Serial No. 115,950 filed December 15, 1936, by Nikolaus Geiser and Herbert Goethel; Serial No. 115,951 filed December 15, 1936, by Heinrich Tramm, and Serial No. 165,566 filed September 24, 1937, by Carl Clar and Herbert Goethel.

It is known to produce lubricating oils by condensing or polymerizing, by means of a suitable condensing agent or polymerizing catalyst such as a metal halide and more particularly aluminium chloride, hydrocarbon mixtures, such as synthetic benzines, which contain unsaturated hydrocarbons. In order to increase the content of unsaturated hydrocarbons in such hydrocarbon mixtures it has also been proposed to subject the mixtures of hydrocarbons serving as starting material for the manufacture of the lubricants to a cracking or dehydrogenating treatment.

In the course of our investigations we have now found that it is not the only nor the most important point to use as starting material in these processes a benzine as rich in olefines as possible. We have found that a benzine suitable for synthetically producing lubricating oils therefrom must correspond, with deviations not exceeding plus or minus 1%, to a well defined curve of density. This curve of density establishes the densities of the individual boiling fractions, into which the benzine may be divided, in the following manner: if a benzine suitable for the production of lubricating oils by polymerization is divided into boiling fractions, each of which is distinguished from the other by 10° C., and if the density of the individual benzine fractions, each of which corresponds to a boiling range of 10° C., is ascertained, the values of density thus obtained must correspond within a limit of error not exceeding 1%, to a curve determined by the following figures, wherein the mean boiling or distilling temperatures of the individual fractions are plotted as abscissae against the corresponding specific gravities as ordinates:

Fraction	Mean boiling temperature	Density at 20° C.	+1%	−1%
	Degrees centigrade			
45–55°	50	0.6510	0.6575	0.6445
55–65°	60	0.6635	0.6701	0.6569
65–75°	70	0.6755	0.6823	0.6687
75–85°	80	0.6860	0.6929	0.6791
85–95°	90	0.6960	0.7030	0.6890
95–105°	100	0.7055	0.7126	0.6984
105–115°	110	0.7120	0.7191	0.7049
115–125°	120	0.7180	0.7252	0.7108
125–135°	130	0.7225	0.7297	0.7153
135–145°	140	0.7275	0.7348	0.7202
145–155°	150	0.7315	0.7388	0.7242
155–165°	160	0.7360	0.7434	0.7286
165–175°	170	0.7400	0.7474	0.7326
175–185°	180	0.7430	0.7504	0.7356
185–195°	190	0.7465	0.7540	0.7390
195–205°	200	0.7500	0.7575	0.7425
205–215°	210	0.7530	0.7605	0.7455
215–225°	220	0.7555	0.7631	0.7479

Hydrocarbon mixtures which contain unsaturated hydrocarbons, but do not obey the law above mentioned, have been found to be less fit for the synthetic production of lubricating oils. The use of such benzines resulted on the one hand in a low yield of lubricating oil, and on the other hand the lubricating oils obtained showed an unsatisfactory viscosity apex, which means that the viscosity of these lubricating oils depended to a very high degree on the temperature*. From benzines, the curves of density of which did not correspond to the above mentioned conditions, there were obtained yields of from 3 to 28% in lubricating oils, calculated on the quantity of starting benzine employed, and these oils showed a viscosity apex of from 2.12 to more than 4. The yield in lubricating oil as obtained from benzines, the curve of density of which corresponded to the above mentioned values, was 42 to 62% and the lubricating oils obtained had a viscosity apex ranging between 1.82 and 1.95.

In the process according to this invention the proportion of unsaturated hydrocarbons in the benzine is of minor importance. The mixtures to be subjected to polymerization may contain for instance 30% or 50% or more olefines. Mixtures which contain considerably smaller quantities, for instance less than 20%, can also be employed with advantage, provided that they possess the correct curve of density explained above.

Our invention may be illustrated more in detail

*(Cf. pages 7 to 11 of "Zur Viskosimetrie" by L. Ubbelohde; 2nd edition, Berlin 1936.)

by the drawings affixed to this specification and forming part thereof and by the following examples. Examples 1 and 2 show the behaviour of benzines with curves of density according to our invention, while Examples 3 to 6 show the behaviour of benzines with different curves of density. Examples 7 and 8 show the manner in which starting benzines with a curve of density according to our invention may be obtained.

In the drawings Fig. 1 shows curves of density as defined above. Curve A corresponds exactly to the requirements established by our invention, while curves B and C represent the admissible deviations from the values of density. Curves I to VI show the values of density of the fractions of the benzines which were employed as starting materials in Examples 1 to 6, respectively.

Fig. 2 is a diagram showing in columns I to VI the average yields in lubricating oil as obtained according to Examples 1 to 6, respectively, calculated in per cents of the starting benzine. The superiority, as to yield, of the process according to the present invention can easily be estimated from this figure.

Fig. 3 is a diagram showing in columns I to VI the viscosity apexes of the lubricating oils obtained according to Examples 1 to 6, respectively, while the upper limit of the viscosity apex admissible for a good lubricating oil is indicated by a dotted line. It will be seen that the columns which correspond to Examples 3 to 6, due to the inferior quality of the lubricating oil obtained according to these examples, remarkably exceed this admissible upper limit of the viscosity apex.

Example 1

As starting material there was used a cracking benzine produced from the fractions, boiling above 150° C., of a hydrocarbon mixture obtained by the reaction of carbon monoxide with hydrogen, and which showed the following curve of density.

Mean boiling temperature	Density at 20° C.	Mean boiling temperature	Density at 20° C.
° C.		° C.	
60	0.656	140	0.724
70	0.671	150	0.728
80	0.681	160	0.730
90	0.692	170	0.734
100	0.700	180	0.738
110	0.708	190	0.741
120	0.715	200	0.744
130	0.720	210	0.746

50 grams anhydrous aluminium chloride were stirred 24 hours at 20° C. in an autoclave with 1000 grams of this dried cracking benzine. The mixture obtained by the reaction consisted of two layers, viz. of the so-called "upper layer," which contains the lubricating oil in dissolved state, and of a lower layer, the so-called "contact layer," consisting of double-compounds of aluminium chloride with portions of cracking benzine. The upper layer weighed 766 grams; it was washed with caustic soda solution, sulfuric acid and water and thus freed from the remainder of the contact layer and neutralized. After drying the not converted cracking benzine was distilled off up to a boiling point of 200° C. The remainder of the distillation was subsequently distilled at 200° C. in vacuo at an absolute pressure of 5 mms. mercury column, whereby the lubricating oil was recovered as distillation residue. There were obtained 407 grams lubricating oil with a density of 0.859 at 20° C. and a viscosity of 17.3° E. (Engler) at 50° C. The viscosity apex was 1.9, while the solidifying point was −23°.

The "contact layer" weighing 283 grams was reacted in the same manner at 55° C. in an autoclave with 1000 grams fresh cracking benzine of the same quality. The upper layer thus obtained weighed 803 grams and contained 450 grams lubricating oil. The contact layer the weight of which had increased to 480 grams, was reacted anew at 95° C. in the autoclave with 1000 grams fresh cracking benzine. There followed similar reactions at 110°, 130° and 150° C.

The following table shows the details of these tests:

	Reaction No.—					
	1	2	3	4	5	6
Temperature degrees	20	55	95	110	130	150
Quantity of cracking benzine started from grams	1,000	1,000	1,000	1,000	1,000	1,000
Contact layer before conversion grams	(50 $AlCl_3$)	283	480	510	470	520
Contact layer after conversion grams	283	480	510	470	520	550
Upper layer do	766	803	968	1,040	945	969
Quantity of lubricating oil obtained grams	417	450	550	610	480	520
The same in percents of the quantity of starting benzine	41.7	45	55	61	48	52
Density at 20° C	0.859	0.856	0.865	0.868	0.866	0.865
Viscosity at 50° C. ° E.	17.3	16	19.1	18	19.6	18.6
Viscosity apex	1.9	1.82	1.88	1.95	1.88	1.92

Example 2

As starting material there was used again a benzine obtained by cracking the fractions, boiling above 150° C., of a hydrocarbon mixture produced by the reaction of carbon monoxide with hydrogen. This cracking benzine showed the following density curve.

Mean boiling temperature	Density at 20° C.	Mean boiling temperature	Density at 20° C.
° C.		° C.	
50	0.652	110	0.716
60	0.668	120	0.721
70	0.679	130	0.726
80	0.690	140	0.731
90	0.701	150	0.735
100	0.710	160	0.739

15 kilograms of this cracking benzine were stirred, in an autoclave of 50 liters, first 4 hours at 20° C. and thereafter 8 hours at 50° C. together with 200 grams fresh aluminium chloride and 11,150 grams of a contact layer, which had been formed in the course of 30 preceding single reactions and contained aluminium-chloride double-compounds. The upper layer formed after the mixture had been allowed to settle, weighed 14,800 grams; it was separated and neutralized. From this layer there were obtained, by distillation under ordinary pressure and subsequent distillation in vacuo, a distillation residue consisting of 9,060 grams lubricating oils, which corresponds to a yield of 60.4% calculated on the quantity of the starting cracking benzine. The contact layer present after the reaction (11,550 grams) was, after an addition of 200 grams aluminium chloride, reacted anew under the same conditions of temperature and time with 15 kilograms fresh cracking benzine.

Any optional number of conversions may be carried out with fresh quantities of cracking benzine, the contact layer obtained in the preceding conversion treatments being reused.

The following table shows the details of five subsequent conversions of this kind, wherein the conversion was carried out 4 hours at 20° C. and subsequently 8 hours at 50° C.:

	Reaction No. —				
	1	2	3	4	5
Quantity of cracking benzine started from....grams..	15,000	15,000	15,000	15,000	15,000
Addition of fresh $AlCl_3$...grams..	200	200	200	200	200
Contact layer before conversion.....grams..	11,150	11,750	12,450	12,800	12,600
Contact layer after conversion.....grams..	11,550	12,250	12,600	12,400	13,050
Upper layer.....do....	14,800	14,500	14,850	15,400	14,550
Quantity of lubricating oil obtained....grams..	9,060	8,760	8,700	9,240	8,720
The same in per cents of the quantity of starting benzine.....	60.4	58.4	58	61.6	58.1
Density at 20° C.....	0.860	0.859	0.860	0.858	0.860
Viscosity at 50° C....° E..	13.38	14.79	17.42	13.88	12.2
Viscosity apex.....	1.9	1.88	1.92	1.9	1.88

Example 3

As starting material was used a cracking benzine obtained according to the so-called TVP-process* from the higher boiling hydrocarbons produced in a benzine synthesis according to Fischer and Tropsch, and which showed the following values of density.

Mean boiling temperature	Density at 20° C.	Mean boiling temperature	Density at 20° C.
°C.		°C.	
50	0.663	110	0.736
60	0.678	120	0.740
70	0.690	130	0.743
80	0.706	140	0.747
90	0.718	150	0.752
100	0.728	160	0.759

In the manner described in connection with Example 1 there were successively carried out three conversions with this cracking benzine at 20°, 40° and 70° C., respectively. The reaction lasted in each case 24 hours. There were obtained only small yields of lubricating oil having too high viscosity apexes. The details of the tests are compiled in the following table:

	Reaction number		
	1	2	3
Quantity of cracking benzine started from.....grams..	1,000	1,000	1,000
Contact layer before conversion grams..	(50 $AlCl_3$)	220	249
Contact layer after conversion.do....	220	249	295
Upper layer.....do....	825	970	950
Quantity of lubricating oil obtained grams..	223	171	232
The same in per cents of the quantity of starting benzine.....	22.3	17.1	23.2
Density at 20° C.....	0.882	0.895	0.892
Viscosity at 50° C.....°E..	49	56	22
Viscosity apex.....	3.03	3.25	3.12

Example 4

As starting material was used again a cracking benzine produced according to the TVP-process from the higher boiling hydrocarbons obtained in a benzine synthesis according to Fischer-Tropsch, and which showed the following values of density:

Mean boiling temperature	Density at 20° C	Mean boiling temperature	Density at 20° C.
° C.		° C.	
50	0.656	130	0.741
60	0.672	140	0.745
70	0.684	150	0.749
80	0.697	160	0.755
90	0.711	170	0.760
100	0.720	180	0.765
110	0.730	190	0.770
120	0.736		

In the manner described in Example 1 there were carried out with this cracking benzine conversions at 20°, 40° and 70°, respectively, and there was again obtained an unsatisfactory yield of lubricating oils which moreover showed too high viscosity apexes. The following table shows the details of the test:

	Reaction number		
	1	2	3
Quantity of cracking benzine started from.....grams..	1,000	1,000	1,000
Contact layer before conversion.. grams..	(50 $AlCl_3$)	179	193
Contact layer after conversion.do....	179	198	245
Upper layer.....do....	875	980	951
Quantity of lubricating oil obtained. grams..	280	186	229
The same in per cents of the quantity of starting benzine.....	28	18.6	22.9
Density at 20° C.....	0.864	0.906	0.907
Viscosity at 50° C.....° E..	34	172	51
Viscosity apex.....	2.12	3.7	3.25

Example 5

Cracking benzine of the following density, produced from hydrocarbons obtained in a benzine synthesis according to Fischer and Tropsch, served as starting material:

Mean boiling temperature	Density at 20° C.	Mean boiling temperature	Density at 20° C.
°C.		°C.	
60	0.668	120	0.737
70	0.677	130	0.747
80	0.694	140	0.756
90	0.706	150	0.764
100	0.717	160	0.773
110	0.727	170	0.780

The conversions took place, in the same way as in Example 1, at 20°, 55° and 95° C. respectively. The yield of lubricating oil was however particularly small and the oils showed a viscosity apex ranging from 2.45 to more than 4. The details of the tests may be estimated from the following table:

	Reaction No. —		
	1	2	3
Quantity of cracking benzine started from.....grams..	1,000	1,000	1,000
Contact layer before conversion grams..	(50 $AlCl_3$)	160	90
Contact layer after conversion grams..	160	90	90
Upper layer.....do....	888	1,069	1,000
Quantity of lubricating oil obtained.....grams..	77	53	37
The same in per cents of the quantity of starting benzine.....	7.7	5.3	3.7
Density at 20° C.....	0.889	0.931	0.9486
Viscosity at 50° C.....°E..	17.5	77.6	39
Viscosity apex.....	3.6	2.45	Above 4

*(The "True-vapour-phase"-process as described for instance in pages 430–431, vol. 15, of "Refiner and Natural Gasoline Manufacturer," or in British Patent 340,021, French Patent 682,328, and German Patent 587,899.)

Example 6

In the same manner as in Example 1 there were carried out successive conversions at 20°, 50° and 80° C., respectively, with a benzine having the following density values:

Mean boiling temperature	Density at 20° C.	Mean boiling temperature	Density at 20° C.
°C.		°C.	
50	0.647	120	0.704
60	0.657	130	0.711
70	0.667	140	0.717
80	0.675	150	0.721
90	0.684	160	0.726
100	0.690	170	0.730
110	0.697	180	0.733

The starting material was a hydrocarbon mixture boiling between 150° and 180° C., obtained as primary product in a synthesis according to Fischer and Tropsch. The yield of lubricating oils was small and the oils obtained showed a viscosity apex of approximately 2.5. The details of the tests are compiled in the following table:

	Reaction No. —		
	1	2	3
Quantity of cracking benzine started from grams	1,000	1,000	1,000
Contact layer before conversion grams	($50\ AlCl_3$)	140	210
Contact layer after conversion do	140	210	230
Upper layer do	910	930	980
Quantity of lubricating oil obtained grams	100	75	130
The same in per cents of the quantity of starting benzine	10	7.5	13
Density at 20° C	0.846	0.844	0.846
Viscosity at 50° C °E	12.7	12.8	5.4
Viscosity apex	2.32	2.5	2.44

We will now describe more in detail some ways according to which hydrocarbon mixtures may be obtained which possess curves of density as prescribed in the process of our invention. The mixtures are obtained as a rule by a cracking reaction from hydrocarbon oils produced by the hydrogenation of carbon monoxide.

The benzine synthesis takes place in the usual manner, for instance by contacting at 180–200° C. and under ordinary pressure a mixture of one part CO and two parts H_2 with a cobalt-thorium-kieselguhr catalyst composed for instance of 37.3% Co, 6.7% ThO_2 and 56% kieselguhr. A cobalt-magnesium-catalyst or a cobalt-catalyst activated by an addition of thorium and magnesium may be used as well. The proportion of CO to H_2 may vary in the gas used for the synthesis. If its content of carbon monoxide is increased, a benzine is obtained which is richer in unsaturated hydrocarbons than the benzines which are usually obtained from a synthesis gas with a proportion of CO to H_2 equal to 1:2. In any case the reduction of the carbon monoxide yields, in addition to gaseous hydrocarbons, a product of reaction consisting of liquid hydrocarbons and solid hydrocarbons which are dissolved in the liquid ones. Slightly increased pressure, for instance of 7–8 atmospheres, may be employed instead of ordinary pressure; in this case the proportion of solid hydrocarbons in the product of reaction is increased.

Benzines having a curve of density such as required in our process may be obtained from these synthetic hydrocarbon mixtures by a cracking reaction. Cracking temperatures of between 450 and 550° C., preferably of 460 to 510° C., are preferred. The period of time during which the hydrocarbons are caused to remain in the cracking zone depends on the boiling range of the starting hydrocarbons and on the cracking temperature used and varies between about 3 and 15 minutes. For hydrocarbon mixtures of a similar boiling range the time of cracking is the shorter, the higher the cracking temperature chosen. Suitable benzines may be obtained according to the following two examples.

Example 7

A hydrocarbon mixture boiling above 180° C. and obtained in the catalytic hydrogenation of carbon monoxide according to Fischer and Tropsch is led under a pressure of 10 atmospheres above normal through cracking tubes heated to 490° C. The products are caused to remain in the cracking zone 6–7 minutes. The return-proportion, i. e., the proportion of the freshly introduced hydrocarbon oils coming from the hydrogenation of the carbon monoxide, to the not cracked portions of hydrocarbon which are recycled, is 1 to 4.5. From the cracking benzines thus obtained there is separated a fraction boiling from 30° up to 180° C. The cracking benzines contained in this fraction correspond to the curve of density according to our invention, as may be seen from the following data:

Mean boiling temperature	Density at 20° C.	Mean boiling temperature	Density at 20° C.
° C.		° C.	
50	0.657	120	0.720
60	0.668	130	0.726
70	0.677	140	0.732
80	0.688	150	0.736
90	0.699	160	0.740
100	0.707	170	0.745
110	0.714	180	0.749

Example 8

A fraction, boiling from 140° C. up to 240° C. of the hydrocarbons obtained in the benzine synthesis according to Fischer and Tropsch is led under a pressure of 10 atmospheres above normal through cracking tubes heated to 500–510° C. The hydrocarbons are caused to remain in the cracking zone 9.5–10.5 minutes. The proportion of fresh hydrocarbons added to the recycled non-converted hydrocarbons is 1 to 3.7. Of the cracking benzines thus obtained the fraction which boils from 30° up to 150° C., corresponds to the curve of densities according to our invention, as may be estimated from the following figures:

Mean boiling temperature	Density at 20° C.	Mean boiling temperature	Density at 20° C.
° C.		° C.	
50	0.658	110	0.713
60	0.666	120	0.720
70	0.675	130	0.725
80	0.685	140	0.728
90	0.695	150	0.730
100	0.705		

When producing lubricating oils in the presence of a polymerization catalyst, such as aluminium chloride, from a benzine or hydrocarbon mixture which contains a sufficient proportion of unsaturated hydrocarbons, such as olefines, and possesses the curve of density defined by the present invention, all the details of operation may be made use of which were disclosed in the above-mentioned copending applications Serial Nos. 115,950, 115,951 and 165,566. Thus during

polymerization the temperature may vary within wide limits, for instance between about 0° and 160° C. It is preferably raised, in stages or continuously, in the course of polymerization as described more in particular in the copending applications Serial Nos. 115,950 and 115,951. The polymerization treatment may last about 12–24 hours for a charge to be polymerized at practically constant temperature, but it may last as much as 120 hours if the continuous treatment disclosed in the copending application Serial No. 115,951 is employed. The quantity of aluminium chloride may amount up to about 6%, calculated on the hydrocarbon mixture treated in the first charge; small quantities, about 0.5–2%, may be added in order to revive the aluminium chloride sludge formed in the polymerization treatment of one charge of benzine and used as catalyst for another charge of the same or a different benzine. In dependency on the viscosity desired of the lubricating oil to be produced the temperature during polymerization is preferably the higher, the smaller the quantity of polymerizing catalyst present and the shorter the duration of the polymerization treatment, as explained more in detail in the copending application Serial No. 165,566. Since polymerization is carried out as a rule in an autoclave, the pressure of operation is substantially the total vapor pressure of the substances under treatment at the operating temperature. In the course of polymerization the pressure drops in proportion as larger molecules are formed from the defines.

Various changes may be made in the details disclosed in the foregoing specification without departing from the invention or sacrificing the advantages thereof.

We claim:

1. The method of producing lubricating oil from a cracked benzine mainly consisting of aliphatic hydrocarbons and rich in olefines, which comprises subjecting for at least about 12 hours to the action of a metal halide polymerizing catalyst, present in a starting proportion of about up to 6%, at temperatures not materially above about 160° C., a cracked benzine of the aforenoted type, the ten degrees C. range fractions of which have densities which, with deviations not exceeding 1% in either direction, are related to the respective mean distilling temperature as follows:

Mean distilling temperature	Density at 20° C.	Mean distilling temperature	Density at 20° C.
° C.		° C.	
50	0.6510	140	0.7275
60	0.6635	150	0.7315
70	0.6755	160	0.7360
80	0.6860	170	0.7400
90	0.6960	180	0.7430
100	0.7055	190	0.7465
110	0.7120	200	0.7500
120	0.7180	210	0.7530
130	0.7225	220	0.7555

2. The method of producing lubricating oil from a cracked benzine mainly consisting of aliphatic hydrocarbons and rich in olefines, which comprises subjecting for at least about 12 hours to the action of a metal halide polymerizing catalyst, present in a starting proportion of about up to 6%, at temperatures not materially above about 160° C., a fraction of a cracked benzine of the aforenoted type, the ten degrees C. range fractions of which have densities which, with deviations not exceeding 1% in either direction, are related to the respective means distilling temperature as follows:

Mean distilling temperature	Density at 20° C.	Mean distilling temperature	Density at 20 °C.
° C.		° C.	
50	0.6510	140	0.7275
60	0.6635	150	0.7315
70	0.6755	160	0.7360
80	0.6860	170	0.7400
90	0.6960	180	0.7430
100	0.7055	190	0.7465
110	0.7120	200	0.7500
120	0.7180	210	0.7530
130	0.7225	220	0.7555

3. The method of claim 1, wherein aluminium chloride is employed as polymerization catalyst.

4. The method of claim 1, in which the benzine subjected to the action of the polymerizing catalyst is produced by the steps of reacting hydrogen with carbon monoxide in the presence of a hydrogenating catalyst to produce a hydrocarbon mixture of the type of synthetic benzine and of cracking the hydrocarbon mixture thus obtained at a temperature ranging between about 450 and 550° C. under slightly increased pressure.

5. The method of claim 1, in which the benzine subjected to the action of the polymerizing catalyst is produced by the steps of reacting hydrogen with carbon monoxide in the presence of a hydrogenating catalyst activated by cobalt to produce a hydrocarbon mixture of the type of synthetic benzine and of cracking the hydrocarbon mixture thus obtained by maintaining same about 5–15 minutes at a temperature ranging between about 460 and 510° C. under a pressure of approximately 10 atmospheres.

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