necessary, may be accomplished by air-blowing after the hydrocarbon atmosphere has been replaced by an inert one.

REFINING OF BENZENE

Benzene derived from the pyrolysis of natural gas appears to differ from coal-tar benzene only in the relative proportions of its constituents, and the same general methods of refining are applicable in both cases. Where the product of pyrolysis is intended as a blending stock, the following are some of the methods of treatment available: inhibitors, sulfuric acid treatment, hydrogenation, fractionation and the separate treatment of the fraction, and vapor-phase refining.

Bound up with this question of refining is also the maximum permissible sulfur content of the benzene. The normal product using metal tubes contained 0.5 per cent sulfur, and this, for blending purposes, was considered satisfactory.

Pyrolysis benzene is a very suitable material for the testing of inhibitors, and considerable success can be expected by this method.

Sulfuric acid refining has proved satisfactory, in that the product, after treatment, is stable and of good quality, but this method is wasteful of what may be useful unsaturated material. Details need not be given as these follow the normal refining lines.

Hydrogenation may be applied to the benzene as a whole, and this has been tried with some success, but attention was concentrated on the hydrogenation of such highly unsaturated fractions of the benzene as the styrene fraction. The purpose of this was to produce extremely useful antiknock materials like ethyl benzene and so avoid loss of unsaturated hydrocarbons in refining. The more saturated parts of the benzene might then be refined using sulfuric acid.

Vapor-phase refining methods, using such substances as zinc chloride, did not prove to be highly successful in the case of pyrolysis benzene.

INFLUENCE OF PRESSURE ON PYROLYSIS OF HYDROCARBONS

Large-Scale Pyrolysis of Natural Gas. On the semitechnical scale, tests were carried out using metal tubes in the furnace section at a pressure of 30 pounds per square inch gage. This, unfortunately, was a limiting pressure owing to the construction of the plant. The heating system consisted of 12 × 2 inch i. d. tubes in series, of which the first eight were ordinary 2-inch steel tubes and corresponding return bends, while the four hottest tubes were of H. R. 4. steel with return bends of the same material. A lagged expansion box was used.

Operation at 30 pounds per square inch was compared with operation at 6 pounds per square inch using stripped gas of specific gravity I.00 to I.01. The throughput was increased from 1800 to 2800 cubic feet per hour at the higher pressure. The benzene production was little changed and was of the order of 1.1 gallon per 1000 cubic feet of nitrogen- and hydrogen sulfide-free gas.

Under 30 pounds per square inch pressure the plant was easier to run than at the lower pressure, heat transfer was improved, throughput increased, and yield remained the same.

ACKNOWLEDGMENT

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Thermal Decomposition of the "Coal Hydrocarbon"

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OAL is not a hydrocarbon. Even if we leave out of consideration the technically very important constituents ash, moisture, sulfur, and nitrogen, the data in Table I show that, in a high-rank bituminous coal, there may be one to nine oxygen atoms to every hundred atoms comprising the coal substance. Only in the anthracites, which are not of commercial interest from the viewpoint of use in any process of recovering by-products from thermal decomposition, do we approach a substance having the composition of a hydrocarbon. An attempt will be made later to show that a study of the thermal decomposition of anthracite may lead to a clearer understanding of the nature of the solid residue obtained in all normal destructive distillations of solid organic complexes.

TABLE I. CHEMICAL COMPOSITION OF COALS

	(In atomic per co	ent^a)	
COAL	C	H	0
Peat Lignite Bituminous Anthracite	38 ± 3 47 ± 4 56 ± 8 78 ± 12	45 ± 3 41 ± 4 39 ± 5 20 ± 12	16 ± 3 12 ± 2 5 ± 4 1 ± 1

a Calculated on the basis % C + % H + % O = 100. For each 100 atoms of C, H, and O, bituminous coals normally contain 1 to 2 atoms of N and 0.3 to 3 atoms of S.

Free hydrocarbons exist in coal in only relatively insignificant amounts, if at all. That certain solvents may

extract hydrocarbons from coal at temperatures from 80° to 260° C.—temperatures below active gaseous decomposition of coal—is frequently cited as evidence to the contrary. This argument appears to be insufficient. A substance dissolves in a solvent owing to the fact that its partial pressure in the solvent is less than its normal vapor pressure—in this respect solvent extraction is analogous to distillation. The fact that solvents extract materials from coal at lower temperatures than are effective in distillation does not necessarily signify their prior existence as such in the coal. For distillation a vapor-pressure gradient of an entirely different order of magnitude is necessary than for solvent extraction. In this connection consider, for example, sugar which may be distilled only at extremely low pressures (20) but may dissolve readily in the appropriate solvent. Also, it is clear that thermal decomposition in the presence of a solvent is easier than in a vacuum (or at atmospheric pressure). Elementary electrostatics states that the attraction between two oppositely charged bodies is less the higher the dielectric constant of the medium in which the bodies exist. Referring specifically to coal, the presence of a solvent therefore loosens the entire solid structure in such a way that less kinetic energy in the form of heat is necessary to cause rupture of the bonds holding the solid together than in the absence of the solvent. This explanation accounts for the greater effectiveness of

pyridine (dielectric constant 9.4) than of benzene (dielectric constant 2.1) as a solvent for coal at their normal boiling points, completely ignoring specific chemical reactions. Although coal is an organic solid showing macro- and microscopic heterogeneity, any attempt to distinguish between "chemical" bonds holding atoms together in a molecule and "physical" bonds holding atoms together between neighboring molecules seems academic and of no real value. It appears reasonable, therefore, to consider solvent extraction as a means for studying the mildest type of thermal decomposition.

CONSTITUTION AND FORMATION OF COAL

If coal is neither a hydrocarbon nor contains free hydrocarbons, how can the presence of a large proportion of aromatic hydrocarbons in coal tars be explained? A consideration of the chemical constitution and the mode of formation of coal offers an explanation. Since Hawley and Harris (6) have shown that the aromatic compound, lignin, may be prepared from the aliphatic compound, cellulose, we need not concern ourselves with which of the two is the mother substance of coal. Furthermore, Schrauth (17) has shown that the products obtained by Willstätter and Kalb (21) from the reduction of lignin, cellulose, and sugars

i. Hydrocarbon skeleton found by Schrauth (17) in the products obtained by Willstätter and Kalb (21) by the reduction of lignin, cellulose, and sugars.
i. Derivative of A suggested by Schrauth as the parent substance of coal.

FIGURE 1

may all be regarded as derivatives of the aromatic nucleus shown in Figure 1A. Schrauth postulates the formation of coal as a result of condensation and polymerization of compounds containing this same nucleus, as shown in Figure 1B. In this case, coal may be regarded as having as an essential element of structure the six-carbon ring characteristic of aromatic hydrocarbons and their derivatives. This conclusion is supported by two other independent modes of reasoning.

In peat, the earliest recognized form of coal, alkali-soluble acids, called "humic" acids, have long been known to exist. During coalification these acids condense and lose their solubility in alkali but may be regenerated by mild oxidation (cf. citation 5). Humic acids may therefore be regarded as an important step in the formation of coal. Fuchs (4) has concluded that humic acids are condensation products of a compound having the aromatic structure shown in Figure 2.

Evidence is presented to show that coals from the peat stage through the higher ranks contain as an essential part of their structure six-membered carbon rings similar to those present in aromatic compounds and graphite. Coal is not a hydrocarbon and contains little, if any, free hydrocarbon. Consideration of the nature of the products obtained from coal with increasing severity of thermal treatment suggests that the aromatics found in coal tar result primarily from dehydrogenation and decarboxylation of depolymerized nuclei present in the original coal. Finally data are presented which support the idea that "free" carbon in pitch, coke, and charcoal may be regarded as highly polymerized aromatic hydrocarbons which gradually and regularly approach graphite as the temperature at which they are formed increases.

Evidence has been presented to show that coals from the peat stage through the higher ranks are aromatic in structure. Keeping this in mind and considering that the agencies effective in coalification are commonly regarded to be heat and pressure, the data presented in Figure 3 may help to make clear the changes

The free peripheral bonds may

hold hydrogen atoms, hydroxyl,

methoxy, or carboxyl groups

which provide the means for con-

The essential aromatic nature

of the coal substance is further

indicated by the work of Bone.

Horton, and Ward (3) who ob-

tained as high as 49 per cent

yields of benzene carboxylic

acids by oxidation of coals of

various ranks by alkaline per-

manganate at 100° C.

densation.

undergone in the metamorphosis of coal and in its thermal decomposition. On Figure 3, showing carbon, hydrogen, and oxygen as its three coördinates (cf. citation 15), there is plotted a coal band which includes a large majority of published analyses of coals. Furthermore, there are two lines: One, starting at the composition of water and ending at 100 per cent carbon, passes through a point representing the composition of cellulose (C₆H₁₀O₅)n, and may be characterized as the "carbohydrate line" or a "line of dehydration;" the other, starting at the composition of carbon dioxide, and only for purposes of illustration passing through C₆H₁₀O₅, may similarly be characterized as a "line of decarboxylation" (loss of carbon dioxide). The coals all lie between these lines, and it may be concluded that the first effect of heat and pressure on the vegetable matter, from which the coals are formed, is the elimination of carbon dioxide and water, primarily the latter. Only when we reach the higher rank coals does another agency appear; we may infer that to form these higher rank coals more heat and pressure are required than for the lower rank coals, and that

sufficient methane is lost during the later changes in rank to cause the observed change in curvature of the coal band. The loss in methane may be attributed directly to the higher temperatures involved. Figure 3 indicates the modification in these conclusions necessitated if one postulates the loss of carbon monoxide, ethane, or other hydrocarbons. It is of interest to note that, if decarboxylation of cellulose could be accomplished with relatively little de-

POLYNUCLEAR FIGURE 2. AROMATIC SKELETON OF A Unit of Structure of HUMIC ACID AS SUGGESTED BY FUCHS (4)

The indicated free bonds contain hydrogen, hydroxyl, methoxyl, and carboxyl groups.

hydration, the result would be a hydrocarbon, possibly similar to petroleum. Treatment of cellulose in water (which would retard dehydration) containing high concentrations of alkali, favoring decarboxylation, has actually been shown by Berl (2) to yield an "artificial petroleum;" in weaker alkali (less decarboxylation) the product resembled coal. Both facts would be expected from consideration of Figure 3.

Throughout the greater part of the slow process of coalification, the progressive change in composition yields coals approaching more nearly to hydrocarbons, as is also shown by

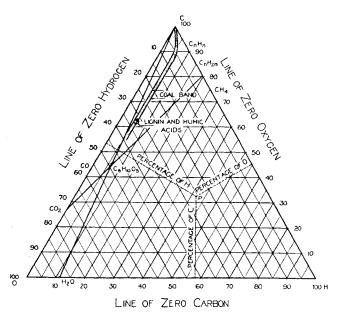


FIGURE 3. DIAGRAMMATIC REPRESENTATION OF COMPOUNDS OF CARBON, HYDROGEN, AND OXYGEN, INCLUDING THE "COAL BAND"

the data in Table I. It is to be expected that, in the more rapid processes represented by laboratory pyrolyses, the simple picture graphically shown in Figure 3 is not applicable. In addition to the changes taking place in coalification, under usual conditions of carbonization a more deep-seated breakdown of the condensed and polymerized aromatic coal substance occurs, and, since the distillate is free to escape and condense, coal tar results. The complexity of the units resulting from the pyrolysis depends entirely on the severity of the thermal treatment. Benzene and its homologs are to be found only in high-temperature tar and do not represent the initial products of thermal decomposition of coal. Solvent extraction, vacuum distillation, and low-temperature carbonization represent increasing severity of thermal treatment of coal and yield progressively simpler products, all, however, more complex than the "secondary" products in high-temperature tar. No one has yet treated a single coal by all these methods and compared the products quantitatively. This is one of the objectives of the Coal Research Laboratory and should yield information on the actual mechanism of thermal decomposition of coal.

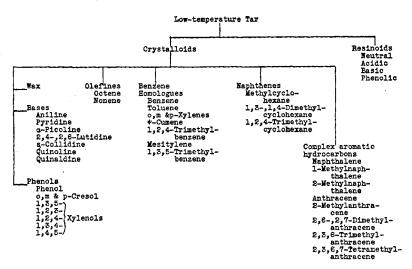
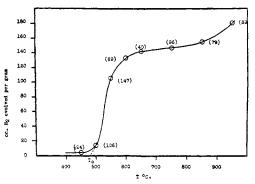


FIGURE 4. CONSTITUENTS OF LOW-TEMPERATURE TAR (12)

THERMAL DECOMPOSITION OF COAL

Data are, however, available in the literature that throw much interesting light on this question. Pictet (13) has made a very comprehensive study of the benzene extract obtained from coal at 80° C. and a vacuum tar obtained at 450° C. and 15 to 20 mm. pressure. The coal used in the solvent extraction was a high-volatile bituminous coal from the Saar, while that used in the distillation study was a coal of similar rank, Montrambert. 5200 kg. of coal, on extraction for 4.5 days in two portions, yielded a total of 13.30 kg. of extract. Vacuum distillation of 1500 kg. of coal provided 60 kg. of tar. Both the tar and the extract were subjected to detailed separation and many individual chemical compounds were identified. In Table II is given a partial list of hydrocarbons isolated together with compounds of the same series identified by Mabery (11) in crude petroleums. In many cases the structure of the hydrocarbons was identified, and the hydrocarbons containing more than



TOTAL CUBIC CENTIMETERS OF HY-FIGURE 5. DROGEN EVOLVED PER GRAM OF ANTHRACITE COAL, UP TO AND INCLUDING THE TEMPERATURE INDI-CATED

The figures in parentheses represent the number of hours the sample was maintained at each temperature.

eleven atoms of carbon did not all belong to the same series. In addition to the hydrocarbons both the vacuum tar and benzene extract contained small amounts of alcohols and bases, several of which were isolated and identified. No aromatic hydrocarbons were found by Pictet although the hydrocarbons did contain six-membered rings. This is to be contrasted with the occurrence of naphthalene reported by several investigators in petroleum (19). It seems reason-

able to assume that the aromatic hydrocarbons in coal tar have been formed by dehydrogenation of hydrocarbons similar to those listed in Table II as well as by further pyrolysis of the coal substance itself at higher temperatures. Sufficient evidence is not available to state definitely that aromatic hydrocarbons may not be formed by "primary" thermal decomposition of coal.

A comparison of the data reported by Pictet on the composition of vacuum tar obtained at a temperature not exceeding 450° C. with similar data on low-temperature tar indicates that aromatic hydrocarbons appear first between 450° and 600° C. The most thorough published work on identification of the chemical compounds in low-temperature tar has been presented by Morgan and collaborators working in cooperation with the Fuel Research Board in Great Britain. His results are conveniently summarized in part in Figure 4 (12). These data indicate clearly that temperatures of 1000° C., characteristic of usual methods of coal carbonization, are not essential to the formation of the aromatic compounds found in high-temperature tar. It is true that high-temperature tar does contain a larger propor-

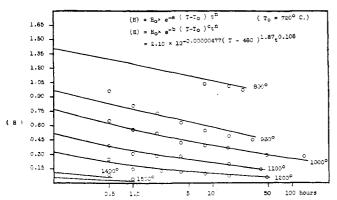


FIGURE 6. PERCENTAGE HYDROGEN BY WEIGHT IN THE SOLID RESIDUE OF AN ANTHRACITE COAL HEATED TO DIFFERENT TEMPERATURES AS A FUNCTION OF TIME

Circles represent observed points; curves are calculated from the equation given in Table III, footnote a.

tion of the simpler aromatics of the benzene series than does low-temperature tar, but this would be expected from cracking of the more complex aromatics.

TABLE II. HYDROCARBONS ISOLATED FROM VACUUM TAR AND BENZENE EXTRACT OF COAL (13) AND FROM Petroleum (11)

SATURATED	Unsatu- rated ^a	SATURATED	UNSATU-	SATURATED	UNSATU-
DATCHATED	C7H10b	CasHababacad	C12H16	DATUMATED	CiaHiab
CaH16(?)b	C ₈ H ₁₂ b,c	C12 H2401018	C12H14C	C16H32d	C16H 80 C
C9H18(?)b.d	C9H14b,c	C13H20b.c.d	C13H16b,c		C ₁₇ H ₂₀
	C10H16b C		$C_{13}H_{12}b$	C30 H 50 b, c , e	• • • •
	C9H14b,c	C13H2,6,c,d	C13H16b,c		C17 H20

- ^a Markovnikov and Ogloblin [Ber., 16, 1873 (1883)] have found in Russian petroleum, members of the series C_nH_{2n-s}, C_nH_{2n-10}, and C_nH_{2n-12}.
 ^b Benzene extract.
 ^c Yacuum tar.
- Petroleum.
- Galician petroleum (Pictet).

So much work has been reported on the composition of high-temperature tar that it does not appear necessary to present here any list of hydrocarbons that have been isolated.

It has been the purpose of the paper thus far to present evidence regarding the source of the aromatic hydrocarbons found in coal tar although coal itself is not a hydrocarbon and contains little, if any, free hydrocarbon. According to the point of view presented, this source is to be found in the chemical structure of the coal itself in which several investigations have clearly shown the existence of polynuclear aromatic structures.

Such data as have been cited on the composition of benzene extract, vacuum tar, and low-temperature tar are necessarily limited in their application to coals of ranks lower than anthracite. According to the figures presented in Table I, anthracite of all the coals approaches most nearly to a hydrocarbon in composition. In the progressive metamorphism of coal, with the elimination of water, carbon dioxide, and methane, it seems unreasonable to expect that the aromatic structure characteristic of coals of lower rank would disappear during the transition to anthracite, which therefore may be regarded as a highly condensed multinuclear aromatic hydrocarbon. This point of view is supported by x-ray evidence (8), which

has proved the existence in anthracite of carbon atoms arranged in six-membered rings as in both benzene and in graphite.

THERMAL DECOMPOSITION OF ANTHRACITE

A study of the thermal decomposition of anthracite yields information on the nature of the more condensed hydrocarbons, among which we may include the "free" carbon in pitch, coke, and charcoal (cf. citation 1). When anthracite is pyrolyzed, it behaves similarly to other complex molecules which contain the remarkably stable six-carbon ring structure, losing hydrogen and the simpler hydrocarbons (predominately methane) and leading to chemical condensation through carbon-to-carbon linkage. In this way larger and larger molecules are formed (cf. citation 7), richer in carbon and poorer in hydrogen until the end stage, graphite, is reached. For each anthracite coal there appears to be a critical temperature (To) which must be exceeded before evolution of hydrogen begins. Above this temperature the elimination of hydrogen progresses slowly and regularly as shown in Figure 5 (10). The composition of the residue is a function of both the maximum temperature to which the sample is heated and the time at this temperature, the former being more important as is evident from Figure 6. (The percentage figures shown are on a weight basis; to change to an atomic basis it is necessary to multiply the percentage hydrogen by 10-11.) With different coals, the results of analyses (for method of analysis, see original paper, 10), for carbon and hydrogen can be equally well expressed by suitable modifications of the values of the constants in the equation shown on Figure 6. Similar data are given for fourteen coals in Figure 7 (10), each point representing the average of multiplicate analyses of samples heated to the indicated temperature ±5° C. for 30 minutes in an atmosphere of hydrogen. For purposes of comparison, there are plotted also in Figure 7 two points, V, for wood charcoal (18), a point, R, for activated coconut charcoal (16), and a point, P. for sugar charcoal (14).

It is of particular interest that the activated charcoal falls within the band of the coal residues, since "active" carbons have been frequently regarded to be a special form of carbon. The process of activation consists essentially in heating the carbonaceous residue from thermal decomposition of anthra-

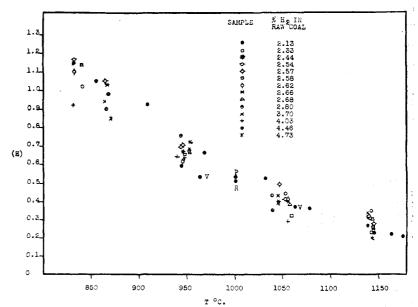


FIGURE 7. HYDROGEN CONTENT DATA OF FOURTEEN DIFFERENT COALS HEATED IN AN ATMOSPHERE OF HYDROGEN FOR 30 MINUTES AT VARIOUS Temperatures

cite, nutshells, wood, or sugar in an atmosphere of steam or carbon dioxide. The data in Table III (10) show that the carbon to hydrogen ratio is essentially independent of the atmosphere in which the samples are heated. This has elsewhere (9) been interpreted as support of a hypothesis that for each given carbon to hydrogen ratio there is a specific adsorptive capacity characteristic of a definite degree of unsaturation of the solid and that the activation process is primarily one of increasing the surface by chemical action. As the residues are heated to progressively higher temperatures, they approach more and more closely to graphite in properties, the crystal size as indicated by x-ray studies increases (8), and the last traces of hydrogen are not eliminated until the carbon atoms have sufficient mobility to take the most stable configuration of graphite. In accordance with this point of view, "amorphous" carbons are complex hydrocarbons of aromatic structure and represent a definite stage in the thermal decomposition of any organic complex which leaves a solid residue on pyrolysis.

TABLE III. HYDROGEN CONTENT OF SAMPLES OF ANTHRA-CITE COAL TREATED AT DIFFERENT TEMPERATURES IN HYDROGEN, AIR, AND CARBON DIOXIDE (10)

° C.			
900 0.76 (1000 0.49 (1050 0.33 (1100 0.29 (1150 0.22 ((4) b 0.67 (9) (4) 0.48 (5) 1) 0.36 (3) 2) 0.28 (3) (7) 0.22 (1) (2) 0.18 (3) (1) 0.13 (2) (2) 0.09 (1)	0.67 (6) 0.47 (3) 0.36 (3) 0.28 (6) 0.22 (8) 0.17 (15) 0.14 (1) 0.09 (2)	0.78 0.49 0.37 0.28 0.21 0.15 0.11

These samples were calculated using the following equation: $H = 2.10 \times 10^{-0.0000527} \, (T-490)^{1.37}$ assuming $t^{0.108} = \text{constant} = 1.105$, which is combined with the constant, a, giving the value 5.27×10^{-6} . The figures in parentheses are the number of samples prepared at the conditions indicated. The hydrogen content given is an average of this number.

SUMMARY

Coal is not a hydrocarbon and contains little, if any, "free" hydrocarbon. It contains, however, as an essential part of its chemical composition, the six-membered carbon ring structure characteristic of aromatic hydrocarbons. This nuclear structure becomes more and more condensed as coal passes through its various ranks, peat, lignite, bituminous coal, and anthracite. The condensation of aromatic nuclei appears to be the main reaction in the solid residue during pyrolysis of coal and other complex organic substances and does not end until graphite is formed.

The chemical nature of the condensed products obtained in the thermal decomposition of coal depends primarily on the severity of the thermal treatment. It is suggested that solvent extraction may be regarded as the mildest type of pyrolysis with vacuum distillation, low-temperature carbonization and high-temperature carbonization representing successively severer conditions. The similarity in chemical

composition of a benzene extract and a vacuum tar obtained by Pictet was pointed out. Neither the extract nor the vacuum tar contained simple aromatic hydrocarbons, though both contained saturated and unsaturated hydrocarbons having six-carbon ring structures and both contained hydrocarbons identical with those isolated from crude petroleums by Mabery and others. The simple aromatics appear first in low-temperature tar, and it is therefore suggested that they are first formed between 450° and 600° C., probably not as "primary" products but by dehydrogenation of compounds such as were isolated from the benzene extract and the vacuum tar. By further pyrolysis of the more complex hydrocarbons, represented by the "primary" products of coal distillation, high-temperature tar contains a still higher concentration of the simpler aromatics.

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GASOLINE FROM COAL TO BE PRODUCED BY STATE-OWNED PLANT IN SAXONY. A plant to produce gasoline from coal will be constructed and operated by the State Government of Saxony if present plans materialize, according to a report from the Consul at Dresden, submitted to, and made public by the Commerce Department.

The manufacture of gasoline, the report shows, is to be based to the resources of the State-owned mine in Bohlen. The new on the resources of the State-owned mine in Bohlen. enterprise is to be in line with a national motorization scheme developed by the present German régime. Its close coördination with the national scheme is supported by the fact that the central government has offered to furnish 5,000,000 reichsmarks, the initial capital required for plant construction.

It is proposed to convert approximately 600 tons of lignite into gasoline daily by a special process, it is stated. The annual output of gasoline from the plant is forecast at between 5000 and 20,000 metric tons. At present, Germany depends largely upon imported gasoline. It is anticipated that total consumption in 1933 will amount to 1,360,000 metric tons, of which 830,000 tons valued at 200 million reichsmarks will have to be imported. marked increase in gasoline consumption is predicted for 1934 as a result of recent developments in highway construction, reduction of taxes, and other government measures. In the in-terest of national economy, therefore, Germany has a strong desire to produce its own motor fuel.

Compared with the total national demand for gasoline, production of 20,000 tons annually from soft coal does not appear important. As a beginning, however, it is important, since sufficient raw material exists in Germany for the production of the

largest part of the country's gasoline requirements.