deprived of their tar in the usual fashion, the gas itself being allowed to go to waste.

I shall refer to another process of carbonisation by means of combustion gases later on, in connection with the furnaces of Pintsch, in which preliminary carbonisation in a current of flue gases takes place.

(3) *Combined Apparatus*

It has long been known that in the gasification of bituminous coal in producers tar is obtained. This tar is, however, of very poor quality when taken hot from the generators, for instance, for the purpose of utilising all its sensible heat in a Siemens-Martin furnace. The lighter constituents of the tar do not separate and they are chemically changed by superheating; the higher boiling fractions separate on the cool parts of the pipes and conduits. By contrast with hot generators a considerable quantity of good tar is obtained in Mond gas producers (Fig. 40), which for the purpose of a high yield of ammonia are run at low temperature by means of a copious supply of steam. Mond gas tar is closely related to primary tar; indeed with a fuel-bed of sufficient height a product equal to primary tar can be obtained.

Thyssen & Co., of Mülheim-Ruhr, have for some time been producing Mond gas tar which they work up for lubricants. As long as the production of primary tar in producers was restricted to "cold" generators, it did not admit of expansion.

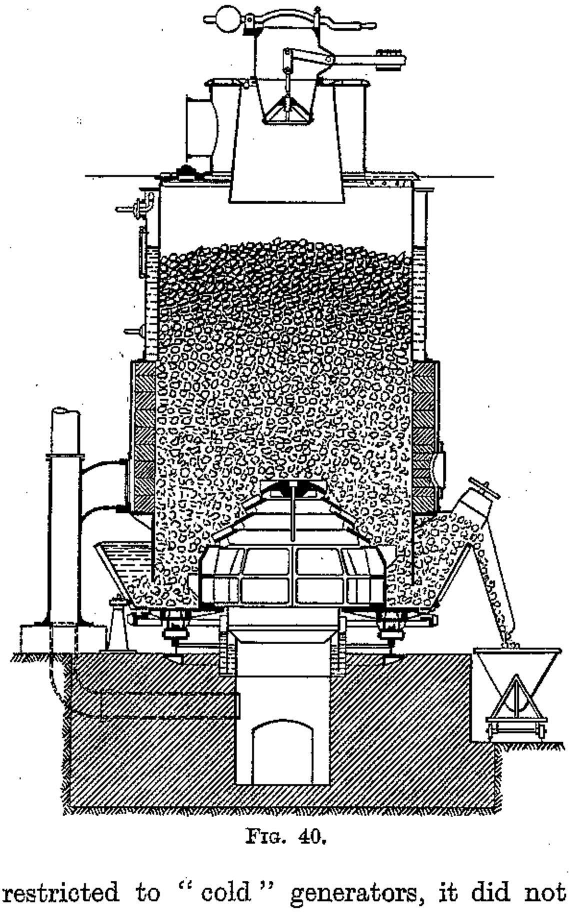

FIG. 40.

Hot-Run Generators Fitted with Carbonising Retorts.—The introduction of special carbonising retorts made it possible to obtain primary tar from "hot" generators. With generators provided with these special retorts, it is desirable to keep the distillation gases and tar vapours separate from the producer gas which is made in the lower zone of the generator by the gasification of the semi-coke. The producer gas can then be carried to the point of consumption, whilst the carbonisation gas passes to the tar separators. This separate collection of the carbonisation gases has the great advantage that condensation of the tar and recovery of benzine can be effected from a much smaller volume of gas, in smaller apparatus, and more completely, than if it were diluted with many times the volume of producer gas.

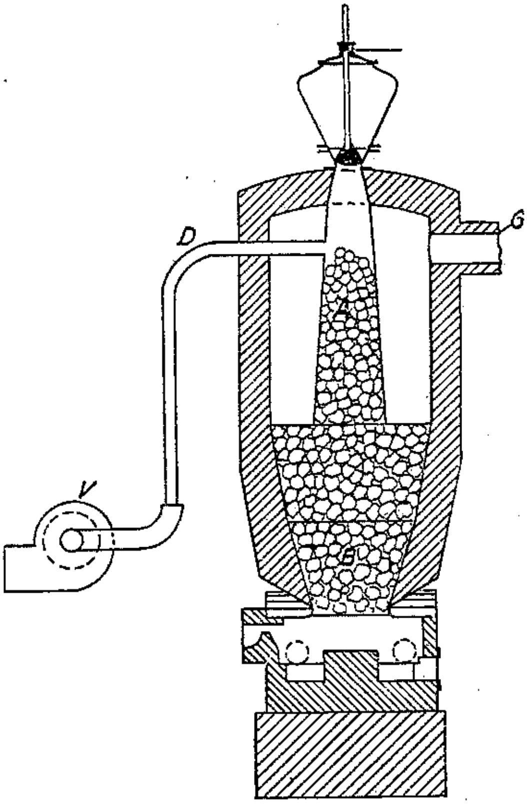

Fig. 41.

If gas flame coal is thus gasified in a producer so that it has first to pass through the retort *A* (Fig. 41), in which it is heated up to 500° before it enters the shaft *B* below, and if it is left in the retort long enough, a yield of 100 kg. of tar and 50 cb.m. of gas per ton will be obtained, the gas having a heating value of about 9000 calories. On the other hand, the coal, after removal of the tar and of the gas, will yield in the producer portion proper only about 5 cb.m. of a gas of approximately 1200 calories.

During the passage of the gas through the retort, a considerable amount of water is evolved, so that, in addition to the 100 grams of tar and the 50 litres of gas from every kg. of coal charged into the retort, 0·7 kg. of semi-coke is discharged from it. This semi-coke will then yield only ·70 per cent. of the 5 cb.m., that is, 3·5 cb.m. of producer gas of 1200 calories.

The producer gas is withdrawn from the generator at *G* and can be utilised hot; the small quantity of distillation gas, which only amounts to 1/70th of the producer gas, is taken off at *D*, and cooled for the recovery of tar oils and water. As the distillation gas has a calorific value of more than 9000 calories, even in the completely tar-free condition, it may be utilised not only for enriching the generator gas, but also for such special purposes as autogenous welding.

The enrichment of the producer gas cannot be pushed very far, as only

50 litres of this richer gas are available per 3·5 cb.m. of the poorer gas. In the most favourable circumstances the calorific value may rise from 1200 to 1300 calories.

The small yield of rich gas has certain advantages in the removal of the tar and the liquor. One kg. of a gas flame coal with 30 per cent. of volatiles at 500° yields 50 litres of gas and 100 grams of primary tar. From these 50 litres of gas, 100 grams of tar can be easily and quantitatively scrubbed out, including the more volatile hydrocarbons. The condensing apparatus required and the consumption of cooling water will be small. On remixing this gas, freed of tar and water (50 litres), with the large volume of hot producer gas (3500 litres), the temperature of the latter is hardly lowered. If, however, the distillation gas is diluted by drawing in producer gas, its volume is increased and its calorific value is correspondingly diminished. From this large volume of gas, the lighter tar oils and the benzine are not so easily recovered, and on remixing, the producer gas is cooled to a greater extent.

In ordinary Mond gas plants, in which the distillation products are not taken off separately, it would be necessary to extract 100 grams of tar oil per kg. of coal from 3500 litres instead of from 50 litres. The recovery of the light tar constituents becomes therefore much more difficult. The tar recovery plant must also be much larger. In practice it has been found, however, that the separate withdrawal of the gases is not conducive to the smooth working of the generator. If the heat required for the carbonisation of the coal is entirely transmitted through the wall of the retort, carbonisation does not proceed rapidly enough to keep step with the gasification of the semi-coke. Additional heat must consequently be supplied to the coal by other means. This is brought about by drawing a considerable portion of the hot producer gas through the retort, and thus utilising it for directly heating the coal. How large this portion should be can be easily ascertained by trial. The producer gas withdrawn directly must be free of tar, whilst the retort gas should contain a maximum of tar. The moisture percentage of the fuel used naturally exerts a considerable influence upon the amount of the producer gas required for complete carbonisation. The desire for a separate withdrawal of the retort and producer gas has led to a compromise, which, with respect to the recovery of the lower boiling constituents, still remains more advantageous than the combined take-off of the two gases. The first systematic experiments in this direction were made by Ehrhard and Sehmer, of Saarbrücken, and were continued by the off-shoot of this firm, the A.-G. für Brennstoffvergasung (Fuel Gasification Co.). Originally the generators were fitted with several carbonising retorts, which were replaced by an annular chamber designed by Linck, which is illustrated in Figs. 42 and 43. The annular chamber is sub-divided into compartments, which are fed from one common hopper, below which a revolving saucer, provided with an opening, uniformly distributes the semi-coke over the cross-section of the generator. The heating of the chamber is effected partly from outside by means of the hot generator gas which surrounds the chamber, and partly from the inside, a portion of the hot generator gas being drawn through the retort. The bulk of the producer gas is available for use in a hot state for any application. With bituminous coal, about 25 per cent. of the producer gas made must be drawn through the retort; with brown coal briquettes more is required. Trials on a works scale in Upper Silesia, in the Ruhr and in the Saar district, have proved that the tar yield was 80 per cent. of that obtainable in the laboratory.

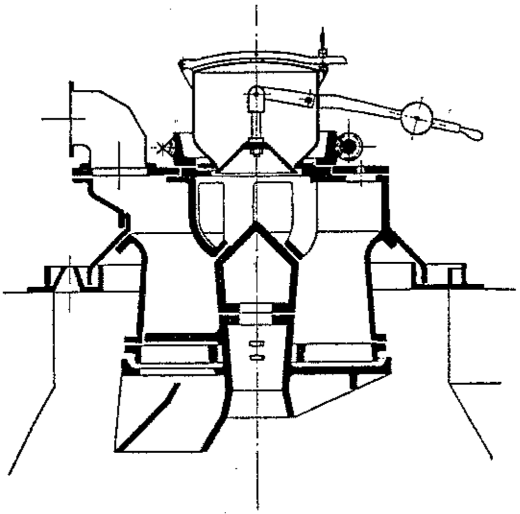

FIG. 42.

The primary tar produced in this way is quite satisfactory, and it is stated that the additional consumption of coal to make up for the tar extracted does not exceed 10 per cent. As tar fetches a higher price than coal, the process, if carefully operated, seems to be commercially sound.

If the whole of the producer gas is cooled for tar recovery and then applied in metallurgical processes, it is very doubtful whether the same temperatures can be reached in the furnace as with gas still containing its sensible heat. Against this objection it must be remembered that the gas loses during condensation not only tar, but also a large portion of its water vapour. Since the water vapour in the gas acts merely as ballast, its condensation raises the calorific value of a given volume of gas. The argument that the removal of tar would considerably lower the calorific value is not quite tenable, for the tar vapours in gas leaving the generator at about 800° are already partially decomposed to permanent gas, and the absence of these decomposition products of the tar will diminish the calorific value only to a slight extent. The further objection that in the Siemens-Martin furnace a flame free of tar and soot cannot be so easily controlled, is not very serious with proper supervision. Besides, less valuable tar products might be added to make the flame luminous.

Retorts Combined with Low-temperature Producers.—Thyssen & Co. do not fit their gas producers with retorts, but prefer to carbonise in large rotary drums, and to gasify the semi-coke subsequently in Mond gas producers.

The Deutsche Mondgas- und Nebenprodukten G.m.b.H., on the other hand, have developed carbonising retorts fitted into their generators. These retorts are provided with mechanical stirrers which, by loosening and by mixing the coal, make for a more rapid and more complete carbonisation (Fig. 44).

A number of other generator systems might be named, working with or without carbonising retorts, which have, however, not found any general adoption.

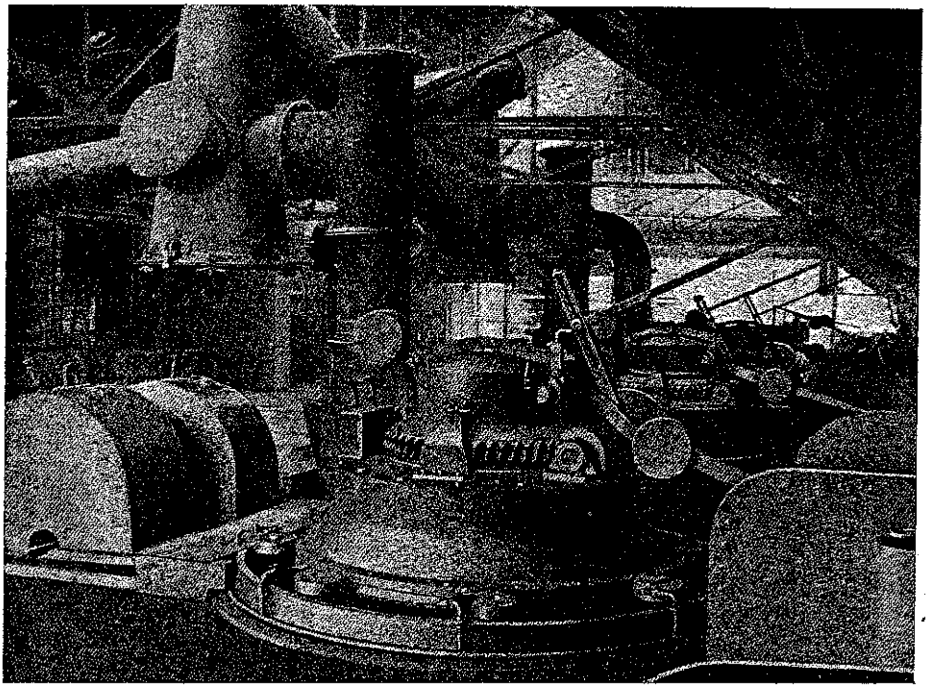

FIG. 43.

A very large plant for the recovery of tar and mineral oil from coal has been erected at Rositz, by the Deutsche Erdöl-A.-G., in which brown coal briquettes are gasified in producers. These are combined in groups of five with a common condensing plant, and they are provided with carbonising retorts, as shown in Fig. 45. The whole of the producer gas from each generator is drawn through this carbonising retort. The gas is stripped of tar, benzine and ammonia. Since their erection these producers have undergone various structural alterations. At the present time they have a rated throughput of 32 tons of briquettes per generator in 24 hours, with a yield of tar approaching that of the laboratory test. The tar production of this plant amounts at present to 35,000 tons per year, or nearly equal to that of the rest of the Central German brown coal distilleries. The tar is worked up

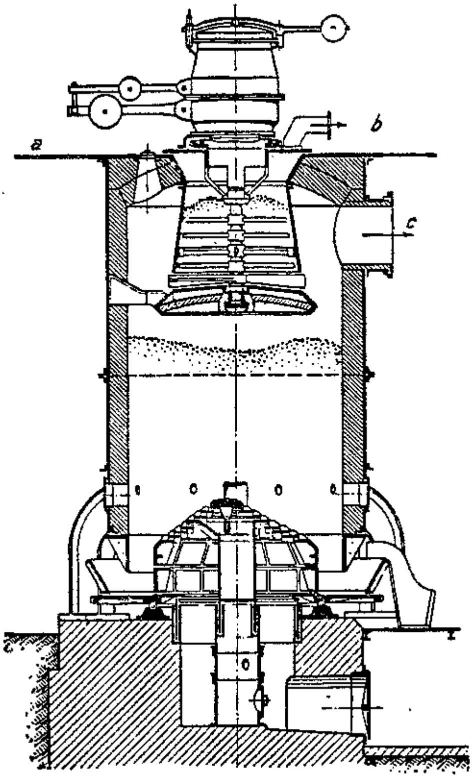
a
b
c

FIG. 44.

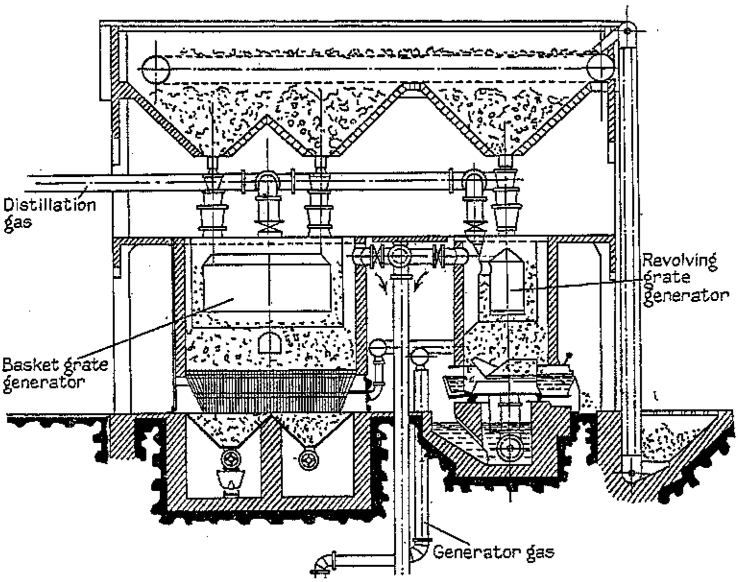
Distillation gas
Revolving grate generator
Basket grate generator
Generator gas

FIG. 45.

in a separate oil works into paraffin wax (candles), lubricants, fuel oils and benzine. In addition, sulphate of ammonia is produced.

A similar large plant in Lützkendorf, which has been completed, and was intended for the gasification of unbriquetted coal, has never got to the producing stage because it was found that the tar recovery from undried raw coal was not profitable.

The Dellwick-Fleischer-Wassergas-Gesellschaft, Frankfort-on-Main, has erected a water gas plant combined with tar recovery on the Tri-gas system at Mathias Stinnes Colliery. During the blow period, the blast enters at the level between the carbonising and producing zone, being directed both downwards and across the coke over an arch. The temperature in this intermediate zone is thereby raised so high that the water gas made during the run enters the layer of coal above sufficiently hot to cause an adequate degasification of the coal. The heat given by the blast must be very intense, because the amount of water gas available for the carbonisation is only a fraction of the gas made in an ordinary generator with steam and air. In the Tri-gas plant the coal gas is mixed with the water gas produced. Since the latter volume is much greater as compared with the amount of water gas simultaneously produced, the calorific enrichment of the water gas is not of a high order. 1·8 cb.m. of Tri-gas made per kg. of gas flame coal can contain not more than 50 litres of coal gas. The object of this plant was not so much the enrichment of water gas with retort gas as the direct production of water gas from bituminous coal instead of coke, together with the recovery of primary tar. The Mathias Stinnes Colliery has also begun to carbonise gas coal in a rotary retort so as to produce semi-coke and primary tar. The production of water gas from the semi-coke then proceeds much more smoothly and without caking troubles in the generator.

Preliminary Carbonisation of Furnace Fuel.—It has been repeatedly pointed out that the recovery of oil from bituminous coal cannot be expected to assume large dimensions as long as the daily throughput of the apparatus in use, vertical retort, rotary retort or producers, remains too small. If it were possible to carbonise the coal which is fired under the boilers of electric power stations, large quantities of tar would become available. The simplest plan seems to be to place a rotary retort in front of the fire-box, and to send the coal through this retort into the boiler furnace. The retort might be externally heated by means of the products of combustion. There is the further possibility, especially in the case of brown coal, to carbonise on Seidenschnur's principle, utilising the sensible heat of the waste gases.

To the best of my knowledge this procedure has not yet been applied. Julius Pintsch, A.-G., of Berlin, have put down a plant in the Lichtenberg Electricity Works for carbonising the fuel prior to firing it under the boilers. Gerdes [81] reported on this process in some detail. The fuel passes from the bunker into a carbonising shaft mounted above a chain-grate stoker; it slowly descends the shaft while the hot products of combustion travel upward from the grate through the fuel and are withdrawn through the gas outlet into the purifier plant (Figs. 46 and 47). It is stated that a good yield of primary tar of good quality was obtained. Unfortunately the experiment had to be discontinued, owing to the closing down of the works on incorporating the town supply in a district power distribution scheme.

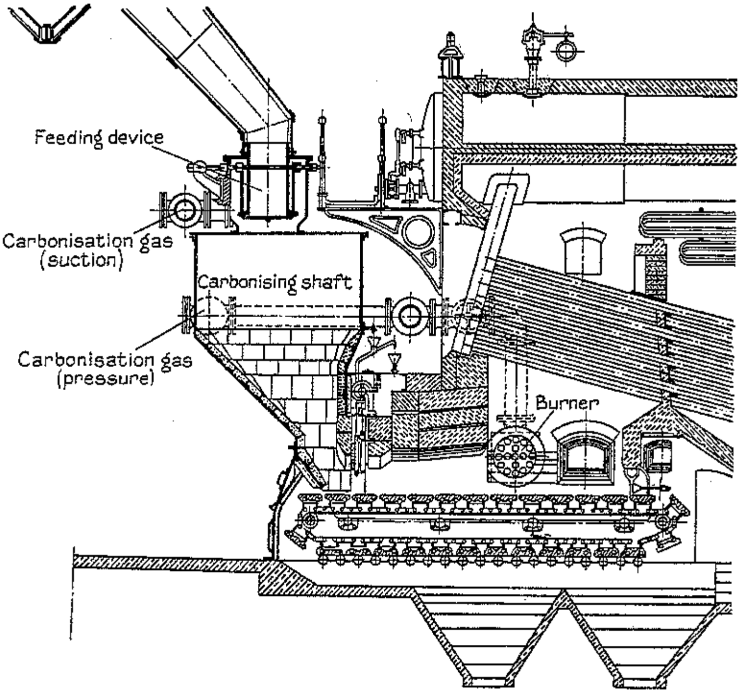

FIG. 46.

From the section on the production of primary tar, it will be recognised that the problem of its production from coal is being energetically and systematically attacked in all countries, and that the tar problem itself may be regarded as solved, but that there are still some difficulties in connection with the semi-coke and its utilisation. There can be little doubt that these outstanding questions will find a solution within the next few years.

The low-temperature carbonisation processes yield, as has been shown, chiefly heavy fuel oils, the benzine percentage being about 10 per cent. of the tar. In the Synthol process we shall find a process which can be combined with low-temperature tar production for the utilisation of semi-coke, and which yields essentially light motor spirits.

(*p*) THE INFLUENCE OF RETORT DESIGN UPON THE COMPOSITION OF PRIMARY TARS AND GAS BENZINES

Primary tars vary with the design of the plants, and particularly with the distribution of temperatures in the retort and the path which the tar vapours take. This question may therefore be discussed a little more in detail. Let us study the conditions of benzine evolution from coal at 300°. Fischer and Gluud [82] have shown that a large portion of the benzines preformed in the coal are liberated at 300° to 350°, and that these benzines, according to analysis, specific gravity, etc., are hydrocarbons definitely non-aromatic, but richer in hydrogen. We obtained such benzines by heating the coal in our rotating drum, raising the temperature in one to two hours from the ordinary to 500°. The vapours appearing, say, between 300° and 350°, were at once withdrawn and condensed, before the retort had reached an appreciably higher temperature (Fig. 48).

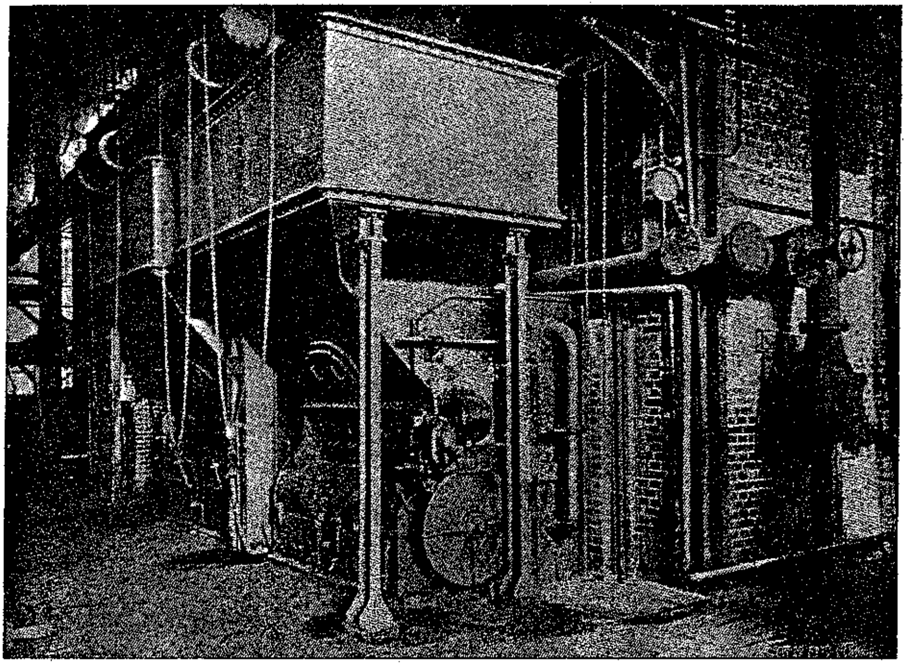

FIG. 47.

Similar conditions prevail in a gas producer (Fig. 49). Here the benzine evolved, say, at 300°, travels upwards through the cooler descending coal without passing through a zone of higher temperature, say, of 600°, at which it might be dehydrogenated or undergo some decomposition.

Our small laboratory rotating drum was obviously not designed for continuous working. The large rotary retorts of Thyssen & Co. are designed for continuous working; the tar vapours do not flow in counter-current with the coal, and they are taken off at the coke discharge end. If the retort were to work on the counter-current principle, the conditions would be as favourable for preserving the primarily-formed compounds rich in hydrogen, as in our intermittent process or in a well-conducted producer, and the material vaporised at 300° would not subsequently be exposed to higher temperatures. Where the gas and coal run in the same direction, the products liberated at 300° are bound to be heated to 500° or 600° at the coke discharge end of the drum.

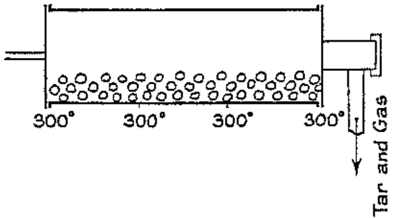

FIG. 48.

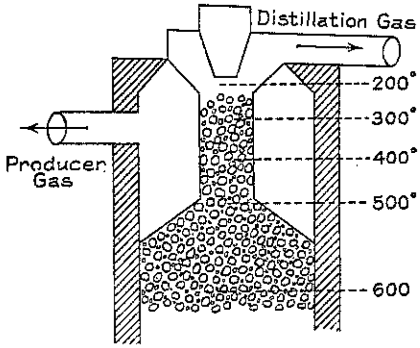

FIG. 49.

The benzines are not the only constituents susceptible to thermal change. Fischer and Gluud have shown that primary tar is modified to a considerable extent even by repeated distillation at ordinary pressure, and particularly when heated to 600°.

In the rotary retort of Fellner and Ziegler, as in those of Thyssen & Co., the tar vapours are withdrawn at the same end as the coke, as indicated in Fig. 50. I am informed that the reason for this practice lies in the difficulty of tar discharge at the cold end of the retort. It is conceivable that under these conditions the high-boiling constituents will be re-deposited upon the cold coal to be returned to the hot zone. Still, it should be possible to design a plant on the lines of Fig. 51, in which the products of distillation are withdrawn through a lagged pipe from a zone of about 300° towards the cold end of the retort. Whether or not this would be feasible in practice, I am not able to judge.

Published descriptions, now coming forward, of low-temperature tars produced in rotary retorts of the type shown in Fig. 50, prove that these products have passed through temperatures from 500° to 600°, and no longer accord in their composition with those Gluud and I obtained in the first instance. The discrepancies cannot be explained by the differences in coals carbonised, but must be sought in superheating phenomena in the retorts.

Naphthenes are known to undergo dehydrogenation at 500°, and Jones[83] has shown that *cyclo*-hexane, methyl-*cyclo*-hexane and tetrahydro-naphthalene begin to be decomposed at or about this temperature. *cyclo*-Hexane yielded benzene and higher olefines, the balance being gas, chiefly hydrogen. The other naphthenes examined behaved similarly. Since there are naphthenic and partly hydrogenated aromatic hydrocarbons contained in the middle and higher fractions of low-temperature tar, it is not surprising that tars produced in too hot a retort, though not containing naphthalene (which is not formed below 700°), yet contain considerable amounts of aromatic hydrocarbons produced by dehydrogenation. In order to avoid confusion it would be desirable to give in future descriptions of primary tars the results of their detailed examination on the lines set out on p. 31. It will then be found if they are appreciably aromatic that they also show signs of superheating, and that the hydrocarbon fraction 200° to 300° has a specific gravity of rather more than 0·95 at 20°. The lubricating oils being likewise of naphthenic character, such tars will not contain as much lubricating oil as a true primary tar. The lubricating oils change, slowly at 300° and very rapidly at 500°, into cracked products poorer in hydrogen.

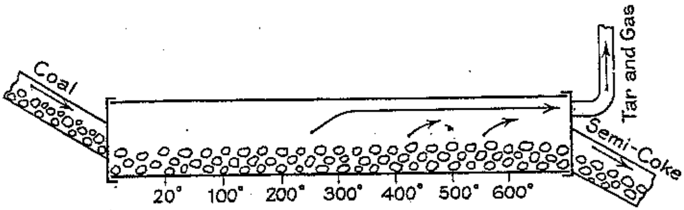

FIG. 50.

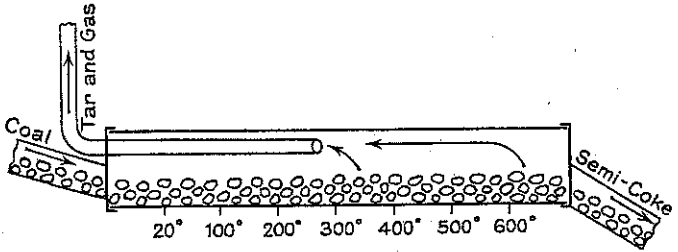

FIG. 51.

(*q*) THE INFLUENCE OF COAL DRYING ON THE OIL RECOVERY

With bituminous coal of low moisture content drying is not as important as with brown coal, which as mined frequently contains 50 to 60 per cent. of water. The following remarks relate therefore chiefly to brown coal and even more to peat which contains 90 per cent. of water; to coal, perhaps, in special cases. Although the drying of brown coal requires the same expenditure of energy, whether performed separately, or in the carbonising plant, there is a certain inclination to saddle the retort or generator with this extra duty. What are the disadvantages of such a procedure? Starting from *dried* brown coal, the distillation yields, in addition to tar and gas, a little liquor, which is evil smelling and objectionable owing to its phenol and sulphur compounds, of low ammonia content and therefore of no commercial value. It may cause difficulty by contaminating ground water and polluting rivers. If the coal is carbonised undried, and drying is effected in the carbonising apparatus, the liquor gets diluted by the original moisture of the coal, and the quantity of the malodorous and objectionable water to be disposed of is materially increased. These drawbacks are avoided if the coal is first passed through an independent dryer at a temperature considerably below that of carbonisation.

In a continuous rotary retort it is quite possible to dry brown coal, as mined, and to carbonise it in the hotter parts of the retort, supplying the heat required from outside sources; in producer working the conditions are different. The heat capacity of the hot producer gas rising from the gasification zone is mostly insufficient for the drying and carbonisation of the upper layers of coal, or for heating them sufficiently to carry the tar vapours from the generator. For this reason regular carbonisation of raw brown coal in a producer is not possible, and still less if the coal is of an earthy character and tends to clog the fuel-bed. This will particularly happen when steam is condensed in the upper layers by lack of heat in the gas. The tendency in future will therefore be to dry the raw brown coal in separate dryers, and thus avoid the dilution of the gas liquor with the natural moisture of the coal. With the rotary system this can be easily effected by heating a separate rotating drying tube with the products of combustion from the carbonising furnace. The steam liberated will be mostly allowed to escape, though it may be collected if desirable. Lump brown coal should be dried down to 15 per cent. moisture, and then charged still warm into the generator. Smaller earthy coal should be duly dried and briquetted. The cost of the treatment cannot be as high as is often asserted. The main expenditure of energy in briquetting goes towards the drying; the pressing takes a fraction only. The energy spent on drying is afterwards saved in the generator, the capacity of which becomes much higher.

It is sometimes stated that it is profitable to run a producer on moist coal, because water vapour is necessary for the gasification, and that it would protect the tar vapours from decomposition. Both these arguments are unsound. The temperature of the fuel-bed of a producer increases from the top downwards. Water is vaporised in the uppermost layer. By the time the coal gets into the carbonising zone the moisture has been expelled; this is certainly the case when the semi-coke has passed into the gasification zone. To charge the generator with moist coal therefore serves no useful purpose.

The suggestion that the tar is protected by the water vapour might have some foundation in the case of a continuous rotary retort not worked on the counter-current principle. In this case the water vapour actually travels through the whole tube and pushes the tar vapour in front of it. It condenses afterwards, together with the liquor from the carbonisation, and increases its volume in the undesirable manner described.

From the scientific standpoint only one objection can be raised against the preliminary drying of the coal. It has been shown [84] that moist coal gives the highest tar yield, and that all dry coals yield less tar, especially when dried by contact with the air.

It may be presumed that, so far as the tar yields are concerned, there is little difference between moist and air-dried brown coal as long as the action of atmospheric oxygen is limited to a few days' storage. Graefe has shown that *prolonged* storage in the air at ordinary temperature has a definite oxidising effect, and influences the tar yield. The same phenomena occur in a shorter time if coal is exposed to atmospheric oxygen at higher temperatures, for instance, when the coal is dried at 105°. In our experiments the samples of moist coal yielded, after drying in the air at 105°, in three cases from one-third to two-fifths less tar than the moist coal as mined; whilst air-dried coals, or those dried at 105° in a current of CO_2, gave a quarter to one-tenth less tar than the moist samples. Thus, for example, Kayna coal gave:

moist as mined	15% primary tar
air-dried	11% ,, ,,
dried in CO_2 at 105°	11% ,, ,,
dried in air at 105°	8·5% ,, ,,

These phenomena, observed in the laboratory, may become very important in the drying of brown coal for briquetting on a large scale. Here, moist coal is also heated to higher temperatures in contact with the air, and is therefore modified so as to diminish the tar yield. It is of importance particularly where briquetted coal is gasified in producers for the production of primary tar. Taking Kayna coal as an example, with a preliminary drying down to 10 or 15 per cent. moisture, the tar yield should not be as low as 8·5 per cent., though it might be depressed to 11 per cent. after ordinary air drying. How far a modification of the conditions of drying will prevent the loss of tar due to internal oxidation, *e.g.* by drying either for longer periods at lower temperature or for shorter periods at higher temperature, is an open question on which some data should be available in works practice. In any event, it would be of advantage to dry, not in air, but in a gas free of oxygen or at any rate poor in oxygen, such as flue gases.

The reduction of the tar yield caused by the preliminary drying is not as serious as it appears at first. It includes mainly the less valuable constituents of phenolic character, whilst the yield of hydrocarbons, per unit weight of coal carbonised, remains the same. If the tar produced is to be worked up for paraffin wax, lubricating oils and fuel oils, the reduction caused by preliminary drying of the coal is not a disadvantage. Graefe pointed out that tar obtained from coal after long storage makes up in quality what it has lost in quantity, and confirmed this assumption experimentally.

To summarise this section, moist coal should be dried in separate apparatus before being carbonised. It should be briquetted if of earthy character, as is done, for instance, in the large works of the Deutsche Erdöl A.-G., at Rositz, where a raw brown coal is used in generators.

(r) UTILISATION AND WORKING-UP OF PRIMARY TAR.

1. *Direct Utilisation of Primary Tar*

Crude primary tar from coal has a calorific value of about 9000 and could no doubt be used as liquid fuel. At the ordinary temperature it is fluid, but rather viscous, the viscosity being greater the less completely the low-boiling constituents were condensed. This tar will, however, hardly be applied as liquid fuel as it is too valuable for that. Containing the low-boiling constituents, its setting point will be near + 4°. The setting point might be lowered by allowing the paraffin wax to crystallise, and removing it in the usual way. Crude primary tar from coal may also be used in large internal combustion engines of the Diesel type. In its general applicability this tar stands mid-way between coke-oven tar and crude petroleum. Owing to its viscosity (50° Engler at 20°) crude primary coal tar may act as a lubricant to a very limited extent. In order to obtain good lubricating oils the tar must be worked up by the methods used in the petroleum industry.

Crude primary tar thickens by evaporation of the low-boiling constituents and hardens by oxygen absorption; it can therefore be used as a protective paint for wood. With an admixture of zinc dust the tar gives a grey-green coating, which on iron soon dries and forms a good protective. Such preparations are, however, inferior to linseed oil paints.

The crude primary tar from brown coal, owing to its high setting point of + 35° (ointment consistency at ordinary temperature), is not suitable for the above-mentioned purposes, and should be worked up into more valuable products.

2. *Working-up of Primary Tar by Distillation* [85]

Chemical Changes on Distillation.

The essential superficial difference between the primary tar from bituminous coal on the one hand, and from brown coal on the other, is that the former is fluid whilst the latter is semi-solid. Thus the primary tar which we prepared in the rotating drum [21] from gas flame coal of Lohberg Colliery had specific gravity 1·00 at 25°, 0·982 at 50°, and setting point + 3·5°.

The coal had the following composition :

Proximate Analysis.

	Per cent.
Moisture	1·8
Volatile matter	37·2
"Fixed Carbon"	59·3
Ash	1·7
	100·0

Ultimate Analysis.

	Per cent.
Carbon	80·8
Hydrogen	5·5
Nitrogen	1·9
Sulphur	0·7
Oxygen by diff.	9·4
Ash	1·7
	100·0

The primary tar from Saxon brown coal (coke yield 51 per cent.) was semi-solid at the ordinary temperature, its setting point was + 37°; in the liquid state its specific gravity was 0·853 at 50°.

That the primary tar from bituminous coal is liquid, whilst that from brown coal is semi-solid, is easily accounted for by the fact that the former is rich in liquid phenols, but poor in paraffin wax, and that the coal contains already liquid hydrocarbons which pass into the tar. From brown coal, on the other hand, only solid constituents can be extracted,[36] such as montan wax, etc.

TABLE XVI

Yields.	Primary tar from gas flame coal.	Primary tar from Saxon brown coal.
	Spec. grav.: At 50° 0·982 ,, 25° 1·000 Setting pt. + 3·5°	Spec. grav.: At 50° 0·883 Setting pt. + 37°.
1. *Distillation*		
Distillate	83%	91% (dry)
Coke	10%	4%
Spec. grav. of distillate	at 50° : 0·948 at 25° : 0·971	at 50° : 0·869
Setting point of distillate	— 19°	+ 28°
2. *Distillation*		
Distillate	94·4%	96·2% (dry)
Coke	3·0%	1·2%
Spec. grav. of distillate	at 50° : 0·942 at 25° : 0·946	at 50° : 0·864
Setting point of distillate	below — 19°	+ 15°
Cracking at 600°		
Distillate	70%	73·5% (dry)
Coke	very little	0·8%
Spec. grav. of distillate	at 25° : 0·990	at 50° : 0·878
Setting point of distillate	below — 19°	— 13·5°

On distilling brown coal a tar is produced which, in addition to paraffin wax, contains a fair amount of undecomposed montan wax and other solids which cause the higher setting point. For the further study of the behaviour of these two primary tars on distillation the following methods were used :—

Bituminous Coal.—The primary tar from a gas flame coal was first distilled at ordinary pressure from an iron still filled to half its capacity. It was to be expected that a large portion of the high-boiling constituents, comprising the highly viscous and unstable lubricating oil, would be destroyed. Table XVI shows that distillation left 10 per cent. of coke and gave 83 per cent. of an oily distillate. The loss of 7 per cent. includes water and gas formed. The specific gravity of the distillate was lowered, and the setting point dropped to − 19°.

A distillation, on identical lines, of the distillate obtained, yielded only 3 per cent. of coke and 94·4 per cent. of distillate, with a loss of only 2½ per cent.; the specific gravity of the second distillate was still lower, and the setting point was below − 19°.

Finally, the second distillate was once more distilled from an iron retort, and passed through an iron tube charged with broken brick heated to 600°. The yield of distillate was then only 70 per cent. owing to the strong decomposition, accompanied by evolution of gas; the specific gravity increased, but the setting point remained below − 19°.

These experiments indicate that the primary tar of gas flame coal becomes more and more fluid by distillation at atmospheric pressure, and that it assumes an extraordinarily low setting point which would render it very suitable as fuel oil and for other purposes.

Brown Coal.—Brown coal primary tar was then treated in the same way. It left in the first distillation only 4 per cent. of coke and yielded 91 per cent. of distillate, of a setting point + 28°.

The second distillation gave 1·2 per cent. of coke and 96·2 per cent. of distillate, of setting point + 15°. Primary tar from brown coal is therefore likewise converted into a liquid oil by repeated distillation at ordinary pressure. The setting point is more markedly affected by destructive distillation at 600°. 73·5 per cent. of distillate, of setting point − 13·5°, was obtained with strong evolution of gas. The specific gravity was slightly higher than originally, but not as much as in the case of the primary tar from bituminous coal.

These experiments demonstrate that, in the production of primary tar, both from bituminous and brown coal, the setting point may be lowered at will by subsequent distillation at atmospheric pressure.

In the commercial production of such tars in generators, it should also be possible to operate the plant so as to get tar of any viscosity and setting point, *i.e.*, of any desired degree of decomposition. The least decomposed tars are those of the highest setting point, provided that the temperature has not risen above 600°, and that all the constituents of the crude gas, which are liquid at the ordinary temperature, have been condensed. If the low-boiling constituents remain uncondensed, the consistency of the tar will, of course, change according to the temperature of the gas leaving the condenser. Instead of an oily tar, under these conditions one containing viscid clots may be obtained.

Only limited percentages of unaltered lubricating oils may be expected in a tar distilled at ordinary pressure and at 600°. Brown coal tar in particular, if by repeated distillation or a corresponding thermal treatment is produced as an oil, liquid at the ordinary temperature, is not likely to contain valuable lubricants. Unsaturated compounds in either tar can, by subsequent heating to the proper temperatures, with or without catalysts, be converted by condensation or polymerisation into products of lubricating oil character.

The deduction to be drawn from these experiments and considerations is that the recovery of viscous oils from the primary tars of bituminous and brown coal demands a cautious treatment, and the application of lower distillation temperatures such as are attained by the use of superheated steam or vacuum.

Working-up by Distillation at Ordinary Pressure

In the distillation at ordinary pressure, the constituents of tar chiefly affected by chemical change are those boiling above 200°. The products boiling below 200°, crude benzine, are obtained as readily by ordinary distillation as by steam or vacuum distillation. The oils which boil between 200° and 300° are obtained in much larger proportions by ordinary distillation than by steam distillation. The reasons are that the fractions boiling above 300°, and in particular the highly-viscous lubricating oils, are decomposed into constituents of lower boiling points, some of which boil between 200° and 300°. Two litres of primary tar from Lohberg gas flame coal, prepared in the laboratory rotating drum, were distilled from a fire-heated iron still.[87] A light oil fraction was collected below 225°, and a motor oil fraction from 225° to 300°; the temperature was then raised to 325°.

The products were: 120 grams of water, 448 grams of light oil, 517 grams of motor oil, 438 grams of fraction 300° to 325°, and 450 grams of pitch.

The motor oil produced had too low a flash point, being below 60°. In order to raise it the oil was redistilled up to 225°, and the distillate obtained was added to the light oil. The yield of light oil thereby rose to 524 grams, whilst that of the motor oil was diminished to 441 grams. At the same time the flash point of the motor oil rose to 71°. The further examination of these products gave the following results :—

Light oil: flash point below 35°; yield 26 per cent. of tar used; calorific value, gross 9734 cals., net 9194 cals.; analysis, carbon 80·52 per cent., hydrogen 9·99 per cent.

Motor oil: flash point 71°; yield 22 per cent. of the tar used; calorific value, gross 9132 cals., net 8630 cals.; analysis, carbon 83·06 per cent., hydrogen 9·29 per cent. Specific gravity at 20°, 0·988; viscosity at 20° = 5° Engler; setting point, below + 3°, with stirring, − 12°.

Fraction 300° to 325°: yield 22 per cent.; 9 grams of paraffin wax were filtered from this fraction; the filtrate had at 50° a viscosity of 5° Engler. The removal of the phenols lowered the viscosity of the residual hydrocarbons to 1·9° Engler at 50°. The hydrocarbons did not deposit any more paraffin wax at room temperature.

Pitch: yield 22·5 per cent. of the tar used. The pitch remained hard when heated in the water-bath; gradual softening without decomposition set in at 125°. In order to lower the softening point of this product, it was regenerated in the following way: 100 grams of pitch were melted, and gradually small portions of the fraction 300° to 325° (after removal of paraffin wax) were stirred into the fused mass. After adding 30 grams of oil the softening point had dropped to 80° (Krämer-Sarnow). The regenerated pitch had the following properties: Specific gravity at 20° = 1·190; calorific value, gross 8619 cals., net 8271 cals.

Distillation products obtained without further chemical treatment are tabulated below :—

TABLE XVII

	In tar.	Boiling range.
Light oil	26%	Below 220°
Motor oil	22%	220–300°
Phenolic lubricating oils	22%	300–325°
Paraffin wax	0·5%	—
Pitch	22·5%	Above 325°

This treatment yielded fairly crude products; the most valuable constituents, the viscous lubricating oils, were destroyed in this distillation.

Lubricating oils may be regenerated by treating the phenolic lubricating oil boiling from 300° to 325° in the following way :—

*Regeneration of Lubricating Oil.**—All distillates above 300° were freed of paraffin wax and then, by treatment with 10 per cent. caustic soda, of phenols. The hydrocarbons were separated from water by centrifuging and then heated in an iron pot to 265°. 627 grams of this material yielded some water and 32 c.c. of a light yellow oil. The residue showed at 50° a viscosity of 2°

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* According to Engler's method.