# OILS FROM CANNEL COAL.

A Thesis
in fulfilment of the requirements
presented for the degree of Ph.D. of

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by

DAVID M. WILSON B.Sc.,

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ProQuest LLC.
789 East Eisenhower Parkway
P.O. Box 1346
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# CONTENTS

# INTRODUCTION.

The greater part of the work described in this thesis was undertaken as part of a Survey of Cannel and similar oil-bearing minerals in Scotland, carried out by the Oil from Coal Committee of the Scottish Development Council. The material for chapters 2 and 3 was collected as a result of this Survey. It is included in a paper on the Survey presented to the recent I.P.T. Conference on Oil Shales and Cannel Coals, held in 1938, by Professor W. M. Cumming and Dr. H. B. Nisbet respectively Chairman and Secretary of the Cannel Inquiry Sub-committee. This work was carried out under a grant from the Commissioner for Special Areas in Scotland.

The work on cannel oil was continued by the author, under the direction of Professor Cumming, after the completion of the survey, and is described in chapters 4, 5 and 6.

The chief difficulty experienced during the work on cannel tar after the completion of the survey was the difficulty of obtaining adequate quantities of cannel tar which was representative of that likely to be obtained from a commercial plant. As a result, the cracking experiments, in particular, were limited in scope.

The work, therefore, could not be a planned research on one aspect of cannel, but was largely exploratory. It is, too, only part of a wide survey of cannel resources, which may bear fruit if cannel as an oil-bearing mineral is mined and retorted. At present the Edinburgh Corporation Gas Department are using a certain proportion of cannel in their retorts, but the primary object is gas production, and the tar is not of importance. Another sign of interest in cannel is a plant at present being erected at Afton Colliery, Ayrshire, for retorting cannel by the Freeman process. This process yields motor spirit as its only oil product, so that fuller examination of the tar will presumably not be possible.

Apart from the purely economic aspect, however, cannel is a mineral of interesting properties which is worthy of study on its own account. It is from this aspect it should be considered in such a thesis as this. The weight which must be given to the wider aspects in these times makes this difficult.

# ACKNOWLEDGEMENTS.

Grateful acknowledgement is made to the Scottish Development Council, under whom this work was started: and to the Commissioner for Special Areas in Scotland, for providing the necessary grant. Acknowledgement is also made to:-

The Directors of the Royal Technical College for the Research Assistantship which enabled the work to be continued.

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The Author expresses his thanks to Professor Cumming for his direction of the work, and for his helpful advice.

# CHAPTER I

## CANNEL COAL AND CANNEL RESOURCES.

Although cannel has been known for a very long time, the term "cannel" has never been strictly defined. Despite properties which distinguish it more fundamentally from bituminous coal, it is recognized in practice by simpler physical differences. Its most obvious property is its uniformity and lack of the banded structure of bituminous coals. It does not soil the hands, and breaks with a conchoidal fracture. Normally of a uniform dull black colour, it may vary from brownish black to grey black. In addition, although most Scottish cannels seem to be hard and brittle - a property which shows close relation to the "parrot" coals - many of them can be cut and polished and have on occasion been made into ornaments and even desks and tables. This seems to have been a local industry in Western Bohemia - see (Schulz, F. - Proc. I.P.T. Conference on Oil Shales and Cannel Coals, 1938, p. 283.)

The name "cannel" is applied because of the property of continuing to burn freely - in a "candle" like manner - when ignited. This empirical test, which is largely given by Scottish cannels, would seem to connect them more with the Torbanites than with the "typical cannel" described by (Hickling, H.G.A. - J. Inst. Fuel, 5, 1932, 318-332.)

The origin of cannel has been the subject of much speculation. Although it is found so frequently associated with bituminous coal, it is quite distinct from it, only on very rare occasions showing a gradation from coal to cannel (Skilling, W.J. - Proceedings I.P.T. Conference on Oil Shales and Cannel Coals, 1938, P.36 and Discussion.)

The most important evidence giving a clue to its mode of formation is based on the structure shown by examination under the microscope. The general mass of the cannel is seen to be composed of a dark, more or less homogenous material formed from macerated plant debris. Imbedded in this there are varying quantities of round or flattened yellowish bodies, together with micro-spores, and occasionally fish remains. In bituminous coal, plant structure is recognizable: in cannel it is not. The fact that cannel is found in more or less shallow beds with well defined boundaries, together with the finely divided nature of the vegetable matter and the presence of algae, gives rise to the present widely held theory of cannel formation. During the laying down of the coal measures flood water would bring down a considerable quantity of broken-up plant material, which would collect, along with drifting algae and spores, and suspended mud, in the stagnant water at the edges of the lagoons and swamps. The separation achieved by this natural flotation process would account quite logically for the sharp difference in properties between cannels and the bituminous coals among which the cannels are found.

The most interesting point of discussion in connection with the origin of cannels is the presence of the so-called "yellow bodies". It now seems generally accepted that the "yellow bodies" are the fossil remains of algal colonies. This view, first put forward by Edgeworth David in 1889, has since been confirmed by several workers, more especially in recent years.

A short summary of the literature on the origin of the "yellow bodies" is given by Skilling, W.J. - Proceedings I,P.T. Conference on Oil Shales and Cannel Coals, 1938, 32. He points out that the work of Bertrand and Renault (Bertrand, C.E. and Renault, B. - Bul. Soc. Nat. Hist. Autun, 1893, VI, 321.), Zalessky (Zalessky, M.D. - Bull. Soc. Geol. France, 1917, XVII, 373.), Thiessen (Thiessen, R. - U.S. Geol. Surv. Prof. Papers, 1925, 132 - I), Paul Bertrand (Bertrand, P. - Congres Internat. des Mines, Metallurgie et Geol. Appliquee, Geol. Sect., Session VI, pp. 159-168.), all confirm the view that the "yellow bodies" are algal remains. He concludes "Microscopic examination has shown that the material known in Scotland by the name of cannel contains algal remains in varying proportions, the other organic matter consisting of macerated plant debris, microspores, and frequently fish remains."

These "yellow bodies" make up a large part of the "Boghead" or "Torbanehill Mineral", the large oil yield given by this material being ascribed to the presence of the algal bodies. If this is so, the cannels having the highest content of fossil algae should be the richest from the oil-yielding point of view. This is certainly the conclusion reached by Skilling in the paper quoted, where he continues - "A study of the microstructure of these materials, in conjunction with the yield of oil obtained from them, indicates that the source of the large quantities of oil must be the algal bodies, as those deposits in which the algae are relatively few produce quantities of oil only slightly higher than the average from a bituminous coal."

Other suggestions have been made to account for the presence of the "yellow bodies". Jeffrey, for example, describes them as spores (Jeffrey, E.C. - Proc. Amer. Acad. Arts Sci., 1910, XLVI, 273.), while Conacher (Conacher, H.R.J. - Trans. Geol. Soc. Glasgow, 1917, XVI, 164.) considered them to be resin fragments. These Conclusions do not seem to be generally accepted, however, particularly since the papers of Blackburn and Temperley (Blackburn, K.B. - Trans. Roy. Soc. Edin, 1936, LVII, part 3, 841-868 - Part I., and Temperley, B. - Ibid. - Part II.), which give well founded evidence to support the algal theory, and indicate that the fossil algae are very similar to a known living alga.

This latter conclusion is of great interest in view of the fact that similar algae are believed to give rise to the substance "coorongite" found occasionally on the shores of lakes of fresh or brackish water in Australia and other places. (Conacher, - Proc. I.P.T. Conference on Oil Shales and Cannel Coals, 1938, p. 42., and Mawson - Proc. I.P.T. Conference on Oil Shales and Cannel Coals, 1938, p. 50.) Such localities show features which conform to those described as resulting in the formation of cannels and torbanites. Intermittent flooding and drying occur, together with the drifting and segregation of various types of vegetable matter on the edges as the water dries up. It might well be that the occurrence of coorongite is a modern example of the processes which formed the present seams of cannel.

Although from the oil-bearing point of view no sharp division between shales and cannels is possible, cannels differ from shales in their close association with coals. Macgregor, for example, in a paper on the oil shales and cannels of Scotland, says (Macgregor, M. - Proc. I.P.T. Conference on Oil Shales and Cannel Coals, 1938, 6.) " --- in the Oil-Shale Group of the Lothians, coals are few and far between." and again "Cannel coals are found in the Lower Limestone Coal Group, in the Upper Limestone Group and in the Productive Coal Measures, and in most cases they are closely associated with ordinary bituminous coals. They occur as one of the constituents of compound coal-cannel seams, in which the relative thickness of the two components varies considerably. Cannel may form the roof or base of a coal, or it may be present as a layer within the seam. In other cases it may constitute the entire coal."

This association of cannel with coal is found in many parts of the world. Cannel itself is present in America, in South Africa, Australia, Central Europe, China and Russia. The only deposits, however, which have been utilised to any extent are those in Britain and Esthonia. The British deposits have been exploited quite considerably in the past, and cannel mining and distilling was at one time a flourishing industry - (see, for example, Giffard, H.P.W. - Ibid. - p. 78.)

The main sequence of events in the utilisation of cannel seems to have depended largely on the influence of natural petroleum and of the gas industry on the market for cannel oil and cannel. Thus in the early days of the gas industry, when illuminating value was the quality most desired in a gas, cannel was soon found to be excellent for this purpose and became widely used. When the possibility became evident of retorting cannel for the production of oils, and, later, paraffin wax, several works were started in Scotland and Wales. A short period of activity followed, and quite a considerable industry grew up. The extent of this is well shown in the historical tables given by Cumming and Nisbet (Proc. I.P.T. Conference on Oil Shales and Cannels, 1938, p. 437) and in a paper by Giffard (Ibid., p. 76.) When Young's patent expired in 1864, the industry was taken up by a host of small concerns, most of which experienced a few years' prosperity, followed by collapse.

The reason for the decline of activity, so far as oil production is concerned, was the competition of the natural petroleum products from America. As

American production increased, prices decreased, until it was quite impossible for the British industries to compete. Even without the distillation of cannel, however, cannel mining might have continued as a prosperous branch of the mining industry, as cannel was very largely used for gas making. The amount used for this purpose in the latter half of the nineteenth century far exceeded the maximum production for the cannel oil industry. A very excellent history of the coal-oil industry is given by the late Professor Briggs, (Briggs - Trans. Inst. Min. Eng. 1930-31, 81, 489.) and among the figures he gives are those showing the production of cannel material in 1865, about the peak period for oil production. Of the 1,418,175 tons of cannel mined, 250,000 tons were retorted for oil, the remainder being used for gas making.

With the discovery of the incandescent mantle, the production of a luminous gas was no longer vital. For this reason the supremacy of cannel for gas making was challenged by the more abundant bituminous coal, which was being mined in ever increasing quantities as the use of steam power increased. In addition, the greater yield of oil from cannel became a disadvantage, as there was no market for it.

This period - about 1870-80 - marked the end of the activity in the cannel mining industry and thereafter cannel was neglected, and looked on as waste material. In most cases it was left underground when the associated coal was mined. When it was more convenient to bring it to the surface with the coal, it was separated sometimes by hand and found its way to the refuse heaps.

This long period of neglect lasted until the war years, when the revived interest in home production of oils led to several investigations of the oil-producing minerals, including cannels. These investigations were undertaken by the Institute of Petroleum Technology, the Petroleum Research Department, the Mineral Oil Production Department, and in 1918 by a committee appointed by the Ministry of Munitions. This work was summarised by the Institute of Petroleum Technology in 1918, and put on record by J. A. Greene in his "Treatise on British Mineral Oil", published in 1919.

The conclusions reached by the I.P.T. committee emphasised the extent of the resources of oil bearing minerals. They give a figure of 10,000 tons per day, of material yielding 15-80 gallons of crude oil per ton, as a justifiable estimate of the possible production. The cessation of hostilities eliminated the need for home produced oil, and the findings of the various committees were never published and were almost entirely forgotten.

Even the arrival of the many processes for the production of oil from coal, which followed the setting up of Parker's "Coalite" retort in 1906, did not revive the interest in the oil-bearing minerals. Attention was concentrated on bituminous coals, which were abundant and possessed well-known properties. Cannel, when it was considered as a possible material, was discarded on account of the lack of definite information as to its reserves. This lack of information should have been remedied by the investigations undertaken during the war years, but it seems very difficult to correct the impression that cannel is invariably found in very small patches. That this is frequently true is certain. But as the survey undertaken by the Scottish Development Council shows, there are such important exceptions to this that the widely held ideas as to the sporadic occurrence of cannel are entirely misleading.

A staunch believer in the widespread occurrence of oil-bearing minerals, Professor Briggs, writing in 1930, pointed out that the pioneers of the coal-oil industry found materials which were supremely favourable to their purpose. He continues "That they found them all is improbable: it is more likely that there exist in our coalfields considerable resources of bituminous shales, passing under such inauspicious names as parroty blaes, rhums, bastard cannel, rattlejack, hoo cannel, wild parrot, etc., which are members of the torbanite or oil shale families: yet no attempt is being made to ascertain their potentialities." (Briggs - Trans. Inst. Min. Eng. 1930-31, 81, 489.)

This is a point of view which is not sufficiently stressed. Many cannels, for example, appear on casual examination to be so worthless that they would be completely disregarded in ordinary colliery practice, and would possibly be quite unrecorded. When one has assayed such unpromising material, to find that it gives an oil yield of 40 or 50 gallons per ton, it is obvious that the Gray-King assay is the deciding test: not the appearance.

When, in 1934, the Oil from Coal Committee was appointed by the Scottish Development Council, the question was reopened, and in 1935 a geological investigation was started by Professor Briggs, the chairman of the Committee, on the authority of the Commissioner for the Special Areas in Scotland.

Following on this investigation, a revived interest in cannel for gas-making purposes was displayed in a paper by Jamieson and King. Addressing the Institute of Gas Engineers, (Communication No. 135, 1936.) they gave an account of experiments carried out at Edinburgh Corporation Gas Works, in which a large quantity of cannel was retorted in a bank of vertical retorts which had been isolated from the main system. They showed that the excellent gas-making qualities of cannel were retained under modern retorting conditions. A saleable coke was produced, which made the process an economic one: so much so, that, allowing only a small figure per gallon for the large yield of tar, it was definitely advantageous to use cannel, and the Authors recommended its use to the Gas Industry.

This work was, indeed, a very valuable contribution to the study of the utilisation of home mineral resources, and, given suitable treatment, such as hydrogenation, the tar would be a valuable source of home produced fuel. Even normal processing of the tar gave a satisfactory motor spirit and diesel oil, though not in great yield. A particularly valuable feature was the scale on which the experiments were carried out, which made it possible for authoritative estimates of the economic possibilities to be made.

## CANNEL SURVEY.

In order to give a more complete picture of the work on cannel undertaken by the Oil from Coal Committee, it might be of value to indicate in general the extent and results of the geological work which formed the necessary basis of the subsequent chemical investigation described in this thesis.

The geological work was carried out by Mr. D. G. Gow, under the supervision at first of Professor Briggs. When Professor Briggs died in 1936, the work of direction devolved on Professor Cumming and Dr. Nisbet. The results of the survey were published in the Second Report of the Oil from Coal Committee, and are given, together with more recent estimates of reserves, in a paper presented at the Institute of Petroleum Conference on Oil Shales and Cannels, by Professor Cumming and Dr. Nisbet. (Cumming, W.M. and Nisbet, H.B. - Proc. I.P.T. Conference on Oil Shales and Cannel Coals, 1938, 423.)

The work in connection with the Survey falls into two main parts:-

(1) A questionnaire was sent to every colliery company in Scotland, asking for details of all canneloid material present in their seams, whether worked or not, and for such necessary information as the thickness of seams, the possible output, and the possible price of the material.

(2) The committee's geologist investigated seams personally, and obtained and assayed samples of the various minerals likely to yield oil.

As a result of this work it was possible to give a reasonable estimate of the resources of cannel in certain areas of Scotland, together with the oil yield and ash content.

The areas investigated were Ayrshire, Fife, Mid and East Lothian, Douglas, Lanarkshire, and in addition, Campbeltown and Brora. Of these, Fife and the Lothians showed large deposits which represent possible sources of cannel. In these areas there are altogether 100,000,000 tons of cannel in reserve, all of sufficient quality to give over 40 gallons per ton. In particular there are seams of 75 million and 8 million tons of good quality cannel available in seams 30 inches thick, and which would thus be quite easily workable.

## TABLE I

| Area | Cannels yielding over 40 gals. crude oil/ton and containing:- | | Total |
|---|---|---|---|
| | Less than 8% ash | Over 8% ash | |
| Lothians | 21,124,600 | 6,443,000 | 27,567,600 |
| Fife | 467,000 | 74,073,000 | 74,540,000 |
| Ayrshire | 4,000,000 | - | 4,000,000 |
| Lanarkshire | 500,000 | 1,235,210 | 1,735,210 |
| Other Areas | 22,800 | 56,000 | 78,800 |
| | 26,114,400 | 81,807,210 | 107,921,610 |

Table I shows the disposition of the main reserves in Scotland, and gives an excellent idea of the widespread occurrence of cannels. While it is true that in many cases a seam which has attractive reserves might be difficult to work, many others are easily available in existing workings or in thickness sufficient to justify fresh workings. The figure of 40 gallons per ton was chosen as the possible economic limit since retorting on the large scale was liable to reduce this figure to $40 \times 0{\cdot}7 = 28$ gallons per ton. The average yield obtained from Lothian shale today varies between 18 and 22 gallons.

The division of coals according to ash content is important from the economic point of view. A residue with a high ash content would normally be unsuitable for use as a smokeless fuel, and the lack of a market for it, might adversely affect the balance sheet of the process. The decision as to which cannel might be most advantageously mined would of course depend on other qualities as well as ash content. A thick and easily worked seam of cannel even if it had a high ash content and a low oil yield would possibly be more attractive economically than a thin seam of high grade cannel which might be difficult and expensive to work. For this reason, cannels are included in the Survey of rather higher ash content than is usually considered desireable from the point of view of smokeless fuel production.

The conclusions reached as a result of this Survey are:-

(1) There are present in Scotland reserves of cannel and canneloid material totalling well over a hundred million tons, and yielding more than 40 gallons of crude oil per ton.

(2) The greater part of this material is available in two or three seams of large extent and of reasonable thickness.

# CHAPTER 2

## PRODUCTS OBTAINED
## by
## RETORTING A SERIES OF CANNELS.

As the Cannel Survey progressed it became apparent that there were large resources of cannel with a high oil yield. Now it seems reasonable that if a plant were to be erected for processing cannel, two very important considerations would be:-

(1) The cannel should be available in sufficient quantity to supply the plant during its economic life.

(2) The quality of the cannel should be sufficiently uniform to enable a variety of cannels to be treated successfully, without alteration in the design of the plant.

The first point is discussed in the preceding chapter where it is shown that supplies were found in reasonable quantity. This chapter is concerned with the investigation of the second one. During the course of the Cannel Survey it was the practice to send to the author samples of all cannels which yielded over 40 gallons per ton in the Gray-King assay, and which were available in reasonable quantity. These cannels were retorted in a small horizontal retort (see p. 21 ) and the products obtained were examined in greater detail than was possible with the Gray-King unit.

A sample of about 40 lbs. was usually available, and this was crushed in a ½ in. jaw crusher and a representative sample of about 4 lbs. retorted.

## CARBONISING CONDITIONS

The laboratory evaluation of any coal for the purpose of assessing the products of carbonisation is very difficult. This difficulty is a very real one even when an attempt is made to determine on a small scale the yields and character of the products likely to be obtained from one particular large scale plant. It is of course impossible to obtain in the laboratory a general figure which can be applied to any type of commercial plant.

This difficulty was experienced when the type of laboratory unit which should be used to evaluate cannels for the purpose of this Survey was considered. It was therefore assumed that the process likely to be used if the cannels were to be commercially exploited would be a Low Temperature Carbonisation. As the temperature of carbonisation in practice could not be determined beforehand, it was decided to carbonise at as low a temperature as possible.

This course has several advantages:-

(1) The tar would be subjected to the minimum of cracking.

(2) Partly as a result of (1) the yield of tar would be as large as possible. The yield of tar would then represent the "maximum practicable yield".

(3) The temperature necessary for carbonisation would be found.

That these conditions held in practice is shown by:-

(a) The small percentage of unsaturateds in the gas.

(b) The fact that in several cases the Gray-King yield was exceeded.

The retort used was a small steel retort of circular cross section, placed horizontally. This was considered as the simplest to construct and operate. To ensure that the temperature conditions were as low as possible, the retort was heated very slowly, starting from cold, and the rate of gas evolution was taken as a rough measure of the amount

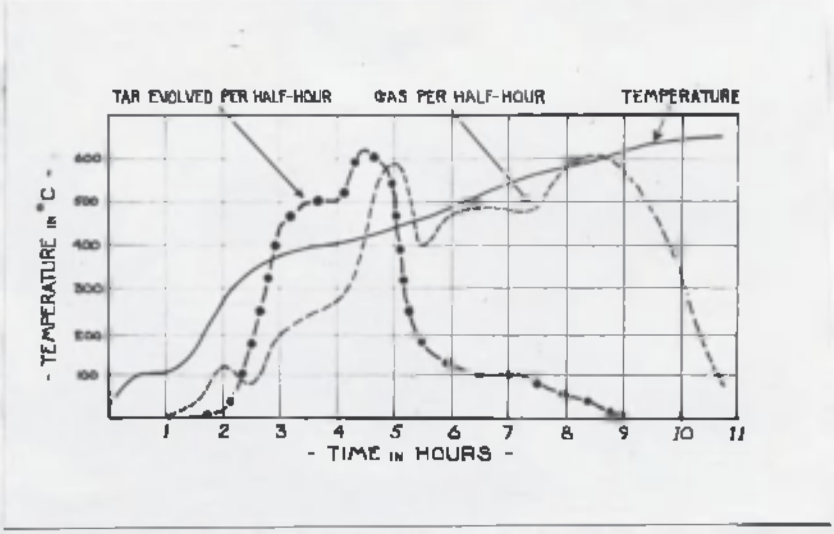


Fig. 1.

Conditions during a typical carbonisation.

To face p.20.

of cracking taking place in the retort. Thus if the gas evolution seemed unduly great it was assumed that undesireable cracking was taking place, and the heating was reduced. As this method, if carried to extreme, would result in a very slow carbonisation, the rate of gas evolution was standardised so that carbonisation was complete in about 11 hours.

This method of carbonisation, although rather tedious, standardises conditions for each coal, but allows for variations in the carbonising properties of different coals. For example, if the carbonising temperature had been kept rigorously at say 600°C, then obviously no distinction is made between a coal which shows maximum tar evolution at 350°C, and one which does not evolve the tar till 500°C. During a carbonisation by the method adopted, however, such a difference in carbonising properties would be easily noticed and allowed for.

In each case, heating was continued until gas evolution ceased, in order to ensure complete distillation. Measurements of the rate of tar and gas evolution during the course of a distillation showed that the time of carbonisation could be reduced considerably in practice. The tar evolution almost ceased after about six hours, by which time a temperature of about 500°C was reached.

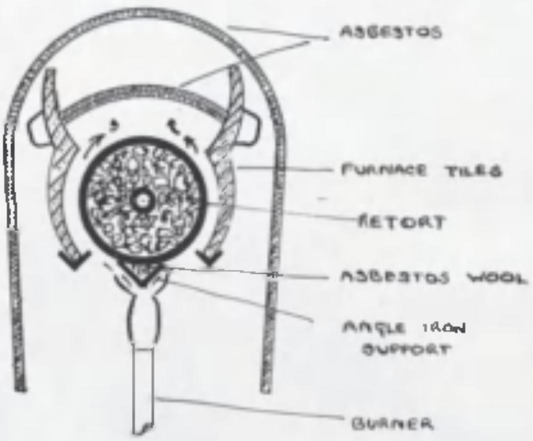


Fig. 3.

Diagrammatic section of furnace.

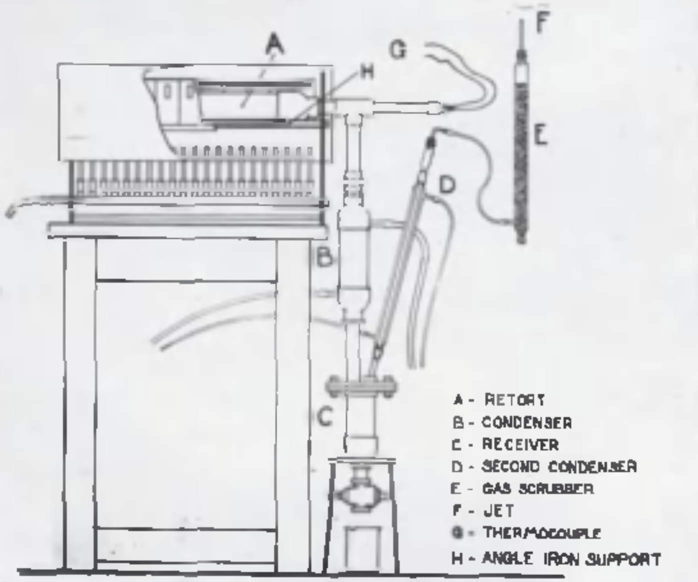


Fig. 4.

Retort Assembly.

To face p.21.

The gas continued to be evolved for a further 5 hours. The conditions during a typical carbonisation are shown graphically in Fig. 1

## THE RETORT

The retort used was a small mild steel horizontal retort of circular cross section, with a capacity of about four pounds. It was made up largely of standard pipe fittings, and gave no trouble in operation. The constructional details are shown in Figs 2 and 3, while Fig. 4 shows the complete assembly.

The component parts are:-

### (a) THE FURNACE.

The heating unit was a gas-fired combustion furnace of the normal type. The body of the retort rested on an angle iron support as shown in Fig. 3, the space between the two being filled with asbestos wool so that the flames did not impinge directly on the bottom. The hot gases were directed by means of fireclay tiles and asbestos sheet, round the walls of the retort, and were discharged at the top end.

This method of heating proved quite satisfactory, and a temperature of up to 700°C could be

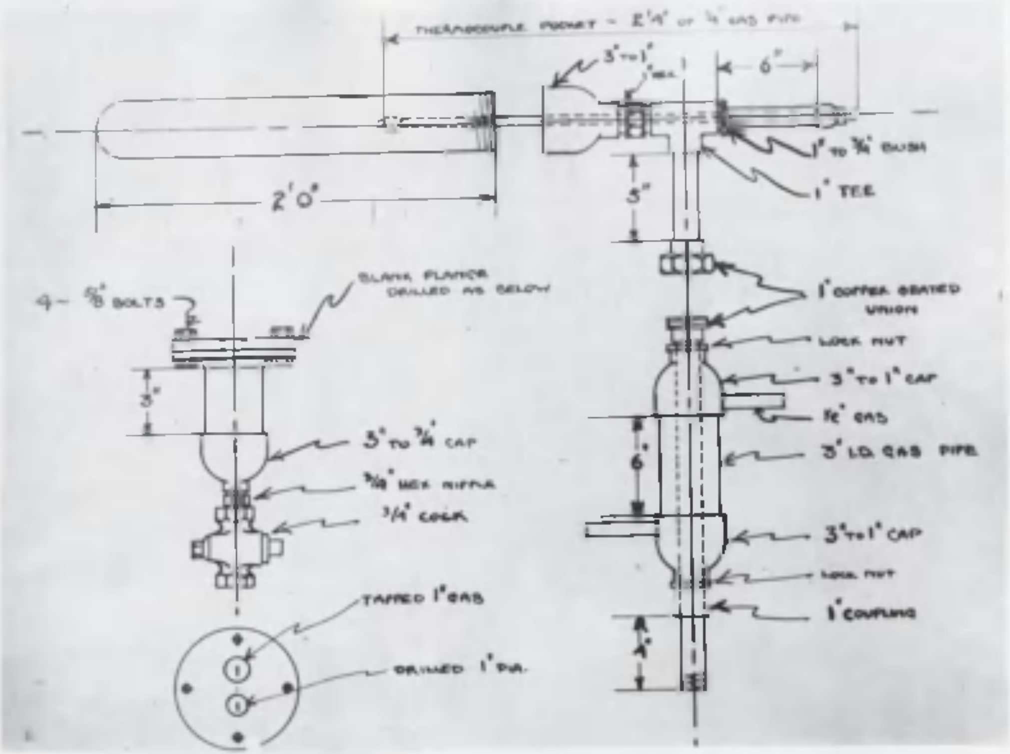


Fig. 2. Details of Retort Components.

To face p.22.

attained at the centre of the retort. As nearly all the tar is distilled by about 400°C (see page 20), this temperature was seldom reached in practice. A manometer fitted in the gas supply to the furnace enabled heating conditions to be duplicated successfully.

(b) THE RETORT.

The retort proper is a 2'6" length of 3" diameter mild steel tube, closed at one end. The open end screws into a 3" to 1" reducing cap, and the retort is charged by unscrewing the body at this point. The retort, thermocouple pocket, and vapour off-take, can be readily detached from the assembly for this purpose by uncoupling the copper seated union (Fig. 2).

The temperature is recorded by a thermocouple in the sheath, situated at the centre of the charge, as shown in the sketch (Fig. 2).

The body of the retort has so far shown very little sign of scaling: and although the joint (J) is made up each time with red lead and linseed oil, it is always possible to unscrew the body of the retort for discharging.

### (c) THE CONDENSER.

The hot vapours from the charge pass down the vertical limb of the T-piece into the condenser. This is made up as shown (Fig. 2) from standard parts, and proved serviceable. Except at the beginning of a distillation the water in this condenser was allowed to run hot. The condensed tar is collected in the receiver, and is run off periodically through the cock.

### (d) THE RECEIVER.

The receiver is made up of standard fittings as shown (Fig. 2), the top consisting of a flange which is blanked off. The condenser tube, is screwed into the blank, into which is also fitted, by means of a cork, a Liebig condenser.

### (e) SECOND CONDENSER.

This is an ordinary glass condenser, which serves to cool the gas, and also condenses any vapour passing the main condenser. The gas emerging was quite clear, no trouble being experienced with tar mist.

### (f) THE SCRUBBER.

From the second condenser the gas passes to the scrubber. This is made of glass as shown in figure 4 and contains active carbon. The carbon rests on a layer of glass beads which prevent choking of the gas entrance. The carbon is steamed by connecting a steam supply to the bottom side arm, a condenser being attached to the top by a right angle bend of wide-bore glass tubing.

### (g) GAS BURNER OR HOLDER.

The scrubbed gas was usually burned in a small jet. As the heating of the retort was regulated to give a slow gas evolution, the height of the flame from this burner was used to indicate the correct rate of heating. Occasionally the gas was not burned, but was passed into a gas holder for examination.

## CARBONISATION

### PROCEDURE

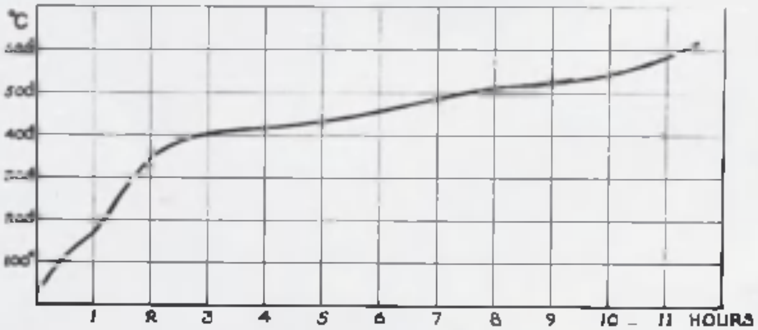
The procedure during the carbonisation was the same in each case, so that conditions varied as little as possible.

The retort was charged by unscrewing the body from the cap at the offtake end, and filling in a weighed amount of cannel. The end was then screwed on again, using red lead and linseed oil to seal the threads, and the apparatus assembled as shown (Fig. 4).

At the start of a distillation the water for both condensers was full on, and the retort was heated gently - a rise in temperature of about 100$^{o}$C per half hour was suitable. The heating was regulated to give a very gentle gas evolution. This was indicated by the height of the flame from the burning gas. A flame 2" - 3" high from a jet 1/16 in. in diameter was the normal rate, and in an attempt to cut down cracking as much as possible this was not exceeded. By noting the pressure of the heating has (on manometer M - Fig. 4), the rate of heating could be duplicated when the gas evolved was not burned.

Every half hour during the carbonisation readings were taken of the temperature of the charge, and the pressure of the heating gas. The tar collected was run off and measured. Where the gas was collected the volume was noted. Soon after the moisture had been driven off, the cooling water to the first condenser was cut off and the condenser allowed to run warm. This was necessary because of the wax present in the tar, which would otherwise block the condenser tube.

A sample log is given on the next page, and a heating curve is shown in Fig. 5.



TEMPERATURE CONDITIONS DURING CARBONISATION

Fig 5

# SAMPLE LOG.

| Hours | E.M.F. | Temp. | Tar Fraction | Total Tar | |
|---|---|---|---|---|---|
| 0 | - | - | - | - | Started up |
| ½ | 3.1 | 100 | 15cc. liquor | | |
| 1 | 5.8 | 170 | 19cc. liquor | | No gas evolution |
| 1½ | 9.5 | 260 | 5cc. liquor | | No tar |
| 2 | 12.0 | 315 | 3cc. liquor | | |
| 2½ | 13.2 | 345 | 3cc. liquor | | No gas evolution |
| 3.5 | 14.0 | 365 | 8cc. | 8cc. | Gas-¼in. flame, non-luminous. |
| 4 | 14.6 | 370 | - | - | Flame slightly luminous |
| 4½ | 15.0 | 375 | 18cc. | 26cc. | Flame luminous, 1½-2" |
| 5 | 15.6 | 400 | 74 | 100 | Flame luminous, 2" high |
| 5½ | 16.0 | 410 | 100 | 200 | " " " |
| 6 | 16.0 | 410 | 47 | 247 | Flame 2" |
| 6½ | 16.2 | 415 | 44 | 291 | |
| 7 | 16.9 | 430 | 40 | 331 | |
| 7½ | 17.0 | 430 | 26 | 357 | Flame 2" |
| 8 | 17.5 | 445 | 20 | 377 | Flame 2" - 3" |
| 8½ | 17.5 | 445 | 38 | 415 | Flame 1" - 2" |
| 9 | 18.8 | 475 | 20 | 435 | Flame 2" - 3" |
| 9½ | 19.1 | 480 | 10 | 445 | Flame 1" - 2" |
| 10 | 19.8 | 500 | 5 | 450 | |
| 10½ | 21.0 | 525 | - | 450 | Flame 3" |
| 11 | 22.0 | 535 | - | 450 | Flame dying out-<br>Shut down. |

At the rate of heating quoted above, the maximum temperature was reached in about eleven hours. The retort was allowed to cool overnight, and the residue removed and weighed.

## SCRUBBER NAPTHA.

The spirit scrubbed from the gas was recovered by steaming the active carbon. After separation, the volume and specific gravity of the spirit were noted.

## TAR.

The tar was run off from the receiver either at intervals during the distillation or at the end. In order to ensure that all the tar was off, it was usually necessary to play a bunsen flame on the receiver and run-off cock, to soften the waxy oil which tended to solidify. The tar obtained was separated from the liquor by allowing it to stand for some hours. Owing to the high setting point the oil has to be kept warm during the settling. The average quantity of tar obtained from one distillation was about 300 cc. so that the number of tests applied to the tar was necessarily limited. The tests applied were:-

### (a) SPECIFIC GRAVITY.

Owing to the great variation in setting point (from 8·7°C to 29·5°C) determination of specific gravity of the liquid oil would have necessitated temperature correction to bring them to a uniform basis. As the temperature coefficient of the tar was not known, and as the high temperature necessary in some cases would have made determinations more difficult, the gravity was determined at 15°C, at which temperature the tars were mostly solid.

(b) SETTING POINT.

This is an indication of the wax content, besides having a bearing on the ease of handling in practice. The method was the standard I.P.T. method L.O.11a.

(c) WAX CONTENT.

The I.P.T. method (I.P.T. - C.P.31) was again used.

(d) FRACTIONAL DISTILLATION.

A 200 cc. quantity of oil was fractionally distilled in order to give some indication of the yield of motor spirit, diesel oil, etc., likely to be obtained in practice.

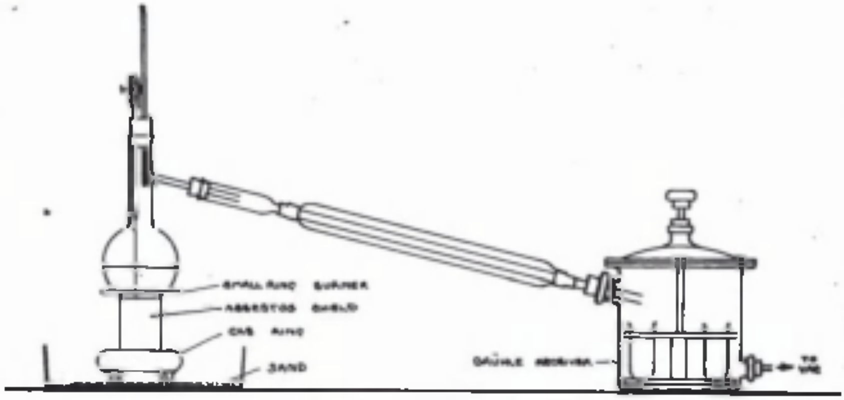


Fig. 6.

Apparatus for vacuum distillation.

To face p.30.

The fractionation was done in two stages -

1. The tar was topped to 180°C at atmospheric pressure.
2. The topped tar was distilled under a pressure of 3 - 6 mm. Fractions were collected at every 50° up to 300°C. The first fraction collected was 0° - 100°C.

The same apparatus was used in both cases, consisting of a Pyrex distilling flask, connected by a condenser to a Bruhl Vacuum receiver.

The apparatus is shown in Fig. 6. The heating arrangement adopted was an attempt to minimise the "bumping" which was a troublesome feature of the vacuum distillation. The asbestos tube, by directing the heat away from the bottom of the flask confined the boiling more to the surface of the liquid, and led to a smoother distillation. With the flask placed as shown, the flames from the burner did not impinge directly on the glass. Near the end of a distillation it was sometimes necessary to use the small ring burner in order to complete the distillation.

When the heavier fractions were distilling, it was necessary to fill the condenser jacket with hot water, to prevent the distillate from solidifying in the tube.

## TOPPING.

The vacuum pump was disconnected and the tar distilled in the usual way. The volume of spirit distilling up to 180°C was noted and expressed as "Percentage Crude Distilling to 180°C".

## VACUUM DISTILLATION.

After topping, the tar was allowed to cool and the apparatus sealed and connected to a vacuum pump. By rotating the carrier the receivers are charged at 100°, 150°, 200°, 250°, and 300°C. From the volumes of the fractions expressed as percentages, a vacuum-distillation curve was drawn. For tables see pp 41 to 58.

## (e) RESIDUE.

The residue was weighed when taken out of the retort. Its appearance was noted, and if it showed any signs of coking its ash content was determined.

# DESCRIPTIVE.

The oil fractions were in general of good appearance when freshly distilled, but all darkened very much on standing. The spirit especially developed a characteristic reddish colour in a few days. All the products, also possessed a peculiar garlic-like odour, even after refining. The refining methods, however, have not been investigated exhaustively, and the colour and odour could be improved by suitable treatment. The characteristics of the various fractions were as follows:-

## SCRUBBER NAPHTHA.

This light spirit, probably owing to its method of recovery, was water white and had a pleasant odour. The colour was quite stable, and after more than a year's exposure to daylight the spirit remained water white. Recently (after between two and three years' standing) samples of this spirit showed a small deposit of a gummy substance, probably a polymerisation product.

Owing to its volatility, it is unlikely that this spirit could be used alone as a motor fuel, but it would form an excellent blending material. The scrubber naphtha from the

Dysart Cannel (see p. 70 ) had a specific gravity of 0.787 as compared with 0.73 for the naphtha obtained from the same coal by the method just described. Even the heavier Dysart naphtha, however, showed a 77% distillate up to 100°C, as compared with the 30 - 33% for a commercial motor spirit.

## CRUDE TAR.

The crude freshly distilled tar is of a dark green or greenish brown colour, which quickly changes to black. Owing to the high content of wax usually met with, it is semi-solid at ordinary temperatures. As is usual with cannel tar it is unlike ordinary tar and tends more towards shale oil. It certainly shows a higher paraffin content than coal tar, and differs markedly in the percentage of tar acids and tar bases: it usually contains about 4% of each of these.

## LIQUOR.

The liquor separated from the tar is evil smelling, and rapidly turns dark red in colour. Its specific gravity over the range of cannels tested was fairly constant at about 1.02, with a yield of about 1.5 gallons per ton.

## LIGHT SPIRIT.

This spirit as it distils has an amber colour and a rather unpleasant odour. It darkens after a day or two, developing a characteristic bright red colour which is also noticeable in the succeeding fractions. The source of this colouration is evidently all distilled by the time the wax begins to come over, as the wax-bearing fractions are much more stable, and darken only slowly through brown to black. The percentage of unsaturateds and aromatics in a sample of spirit were determined by extraction with cold sulphuric acid, (86% and 100%) giving 11% and 43% respectively.

## HEAVIER FRACTIONS.

The tar fractions showed some variation in yield, but the appearance in general was the same for fractions from different cannels.

## FRACTION UP TO 100°C UNDER PRESSURE of 3 - 6 mm.

This is a light mobile liquid and when freshly distilled has a clear amber colour. It darkens to the same red colour shown by the light spirits.

## FRACTION 100°C to 150°C. (3 - 6 mm.)

A heavier and darker liquid than the previous fraction it frequently has a green fluorescence, and tends to deposit crystals of wax. It darkens more slowly than the lighter fractions.

## FRACTION 150° to 200°C. (3 - 6 mm.)

The main part of the wax comes over in this fraction which solidifies on cooling.

## FRACTION 200° to 250°C. (3 - 6 mm.)

This fraction solidifies very readily in the condenser which requires to be kept warm. While liquid, it is a deep golden-brown oil, solidifying to a waxy brown mass. It does not darken quickly.

## FRACTION 250° to 300°C. (3 - 6 mm.)

Care is required when distilling this fraction, as it is a heavy waxy solid, melting at about 40 - 50°C, which solidifies readily in the condenser. The liquid is dark brown or reddish-brown, and viscous; it solifies to an opaque brown solid.

## RESIDUE.

Although some of the cannels carbonised showed excellent coking properties, the majority did not. *Indeed, some of the residues were difficult* to distinguish from the original cannel. During the carbonisation the cannel had not reached the plastic stage: the fragments had retained their shape and size. Larger material would be mechanically strong enough to use as a fuel. For this purpose, its ash content would need to be below certain limits. A similar residue has been stated (Jamieson and King. Inst. Gos. Eng. Communication No. 135, 1936.) to be saleable as a domestic fuel.

In several cases the carbonisation caused swelling and fusion of the charge to a homogeneous mass of coke. This coke had good strength, a bright metallic appearance, and a fairly good ring when struck.

The greatest obstacle to its use as a fuel (certainly as a domestic fuel) is its high ash content, up to about 40% in some cases. The lowest figure, however, viz. 3.6%, fulfils the requirements laid down in the first Report of the Oil from Coal Committee of the Scottish Development Council, issued in 1935.

For industrial use the difficulty might be the *character rather than the quantity of the ash.* Possible outlets for the residue could only be determined by combustion tests on the coke. with particular reference to the ash formed. The possibility of using the residue for the production of water gas for synthetic conversion to liquid hydrocarbons is dealt with in the Second Report of the S.D.C. Oil from Coal Committee. (1936)

## GAS.

The gas evolved by this method of carbonisation was a rich gas, as can be seen from the analysis given in Table.

### TABLE II

### Analysis of Gas Evolved During Distillation.

| | |
|---|---|
| $CO_2$ | 3.7% |
| $O_2$ | 0.5% |
| Unsats. | 5.0% |
| CO | 2.1% |
| $H_2$ | 12.0% |
| Methane | 68.6% |
| Ethane | 7.8% |
| $N_2$ | 0.9% |
| | 100.6 |

This analysis shows that under the conditions holding in the small retort cracking does not take place to any great extent. This is particularly noticeable when this gas is compared with the gas evolved during the carbonisation of the Dysart cannel at the Fuel Research Station. (see p. 70 )

On the following pages ( 41 - 58 ) are the results for individual cannels.

Yields are expressed as gallons (or cwts.) per ton of the original cannel.

Specific Gravities are given at 15°C relative to water at 15°C.

Percentages are given on a basis of Crude Tar (after separation of water) equal to 100%.

Sampling. Samples of about 40 lbs. were secured either direct from the colliery or from [illegible] Development Company's Geologist. The coal was crushed to size and the retort charged with a representative sample of about 4 lbs.

## RESULTS

On the following pages ( 41 – 58 ) are given the results for individual cannels.

Yields are expressed as gallons (or cwts.) per ton of the original cannel.

Specific Gravities are given at 15°C relative to water at 15°C.

Percentages are given on a basis of Crude Tar (after separation of water) equal to 100%.

Sampling. Samples of about 40 lbs. were received either direct from the colliery or from the Development Council's Geologist. The coal was crushed to size and the retort charged with a representative sample of about 4 lbs.

## SAMPLE REFERENCES.

References are made to the pages of a confidential Report presented by Cumming and Nisbet to the Commissioner of Special Areas in Scotland, in October 1937. Unfortunately, owing to the fact that several coal companies took the view that information given by them as to sampling sites, etc, should be regarded as confidential, it was considered impossible to make this Report public. This also holds in the case of the samples carbonised as just described.

The same difficulty explains the letter and figure references in Table III of the Second Report of the Oil from Coal Committee (already referred to).

After the details of the carbonisations given in the pages that follow, a Table is given showing the areas from which samples were obtained, and such details as to site and seam as are allowable. In some cases reference letters are given. These correspond to the reference letters in Table II of the Second Report of the Oil from Coal Committee, in which some further details of the samples may be obtained.

Reference. Report - page 332.

Time of Carbonisation 10 hrs. Final Temperature - 575°C

**Scrubber Naphtha**

| | |
|---|---|
| Yield | 1.24 gals/ton. |
| S.G. | 0.68 |

**Crude Oil**

| | |
|---|---|
| Yield | 74.5 gals/ton. |
| S.G. | 0.88 |
| Setting Point | 29.5°C |
| Wax Content | 23% |

**Light Spirits**

| | |
|---|---|
| Yield | 10.3 gals/ton. |
| S.G. | 0.805 |

Percentage Crude distilling to 180°C.
29.5%

**Vacuum Distillation.** - Pressure - 6 mm.

| Fraction | Percent Distillate | Total Percent Distillate |
|---|---|---|
| 0 - 100°C | 9.0 | 23.5 |
| 100 - 150° | 14 | 37.5 |
| 150 - 200° | 20 | 57.5 |
| 200 - 250° | 12 | 79.5 |
| 250 - 300° | Not distilled | |

Residue + Loss 20.5%

**Residue.**

| | |
|---|---|
| Yield | 10.5 cwts/ton. |
| Ash Content | 64.5% |

No coking properties.

Reference. Report - page 333.

Time of Carbonisation 11 hrs. Final Temperature - 600°C

**Scrubber Naphtha**

| | |
|---|---|
| Yield | 0.98 gals/ton. |
| S.G. | 0.695 |

**Crude Oil**

| | |
|---|---|
| Yield | 40.5 gals/ton. |
| S.G. | 0.903 |
| Setting Point | 11.1°C |
| Wax Content | 9.0% |

**Light Spirits**

| | |
|---|---|
| Yield | 6.7 gals/ton. |
| S.G. | 0.788 |

Percentage Crude distilling to 180°C

16.5%

**Vacuum Distillation** - Pressure - 5 mm.

| Fraction | Percent Distillate | Total Percent Distillate |
|---|---|---|
| 0 - 100°C | 17 | 33.5 |
| 100 - 150° | 10.5 | 44 |
| 150 - 200° | 20.0 | 64 |
| 200 - 250° | 24.5 | 88.5 |
| 250 - 300° | Not distilled | |
| Residue + Loss | 11.5 | |

**Residue**

| | |
|---|---|
| Yield | 14.3 cwts/ton. |
| Ash Content | 16.9% |

This cannel gave a fairly good coke.

Reference. Report - page 334.

Time of Carbonisation 9¼ hrs. Final Temperature - 670°C

**Scrubber Naphtha.**

| | |
|---|---|
| Yield | 0.91 gals/ton. |
| S.G. | 0.73 |

**Crude Oil**

| | |
|---|---|
| Yield | 55 gals/ton. |
| S.G. | 0.93 |
| Setting Point | 22.2°C |
| Wax Content | 14.6% |

**Light Spirits**

| | |
|---|---|
| Yield | 8.5 gals/ton. |
| S.G. | 0.796 |

Percentage Crude distilling to 180°C 15.5%

**Vacuum Distillation** Pressure 3mm.

| Fraction | Percent Distillate | Total Percent Distillate |
|---|---|---|
| 0-100 C | 14 | 29.5 |
| 100-150 | 15 | 44.5 |
| 150-200 | 12.5 | 57 |
| 200-250 | 29 | 86 |
| 250-300 | 9 | 95 |
| Residue + Loss | 5 | |

**Residue**

| | |
|---|---|
| Yield | 12.0 cwts/ton. |
| Ash Content | 8.1% |

The charge had been partially plastic, giving a coke of moderate strength.

Reference. Report - page 335.

Time of Carbonisation 10 hrs. Final Temperature - 650°C

**Scrubber Naphtha**

| | |
|---|---|
| Yield | 1.0 gals/ton. |
| S.G. | 0.74 |

**Crude Oil**

| | |
|---|---|
| Yield | 65 gals/ton. |
| S.G. | 0.93 |
| Setting Point | 27.8°C |
| Wax Content | 13.5% |

**Light Spirits**

| | |
|---|---|
| Yield | 7.15 gals/ton. |
| S.G. | 0.77 |

Percentage Crude distilling to 180°C 11%

**Vacuum Distillation.** Pressure 6mm.

| Fraction | Percent Distillate | Total Percent Distillate |
|---|---|---|
| 0-100°C | 16.5 | 27.5 |
| 100-150° | 17.3 | 44.8 |
| 150-200° | 19.5 | 64.3 |
| 200-250° | 18.5 | 82.8 |
| 250-300° | 9.3 | 92 |
| Residue + Loss | 8 | |

**Residue.**

| | |
|---|---|
| Yield | 11.2 cwts/ton. |
| Ash Content | 3.5% |

Slight binding but the charge was not plastic at any time during the carbonisation.

Reference. Report - page 336

Time of Carbonisation 11 hrs. Final Temperature - 100°C

**Scrubber Naphtha**

| | |
|---|---|
| Yield | 1.10 gals/ton. |
| S.G. | 0.73 |

**Crude Oil**

| | |
|---|---|
| Yield | 66.2 gals/ton. |
| S.G. | 0.88 |
| Setting Point | 16.4°C |
| Wax Content | 15.9% |

**Light Spirits**

| | |
|---|---|
| Yield | 10.9 gals/ton. |
| S.G. | 0.80 |

Percentage Crude distilling to 180°C 17.3%

**Vacuum Distillation.** Pressure 3mm.

| Fraction | Percent Distillate | Total Percent Distillate |
|---|---|---|
| 0-100°C | 8.0 | 25.3 |
| 100-150° | 12.5 | 37.8 |
| 150-200° | 15.5 | 53.3 |
| 200-250° | 20.0 | 73.3 |
| 250-300° | 14.5 | 87.8 |
| Residue + Loss | 12.2 | |

**Residue**

| | |
|---|---|
| Yield | 11.1 cwts/ton. |
| Ash.Content | - |

No coking.

Reference. Report - page 337

Time of Carbonisation 11 hrs. Final Temperature 700°C

## Scrubber Naphtha.

| | |
|---|---|
| Yield | 1.84 gals/ton. |
| S.G. | 0.66 |

## Crude Oil

| | |
|---|---|
| Yield | 86.2 gals/ton. |
| S.G. | 0.87 |
| Setting Point | 9.4°C |
| Wax Content | 8.5% |

## Light Spirits.

| | |
|---|---|
| Yield | 14.2 gals/ton. |
| S.G. | 0.76 |

Percentage Crude distilling to 180°C 16.8%

## Vacuum Distillation

Pressure 4.3mm.

| Fraction | Percent Distillate | TOtal Percent Distillate |
|---|---|---|
| 0-100°C | 10.2 | 27.0 |
| 100-150° | 13.8 | 40.8 |
| 150-200° | 19.5 | 60.3 |
| 200-250° | 21.5 | 81.8 |
| 250-300° | 10.3 | 92.1 |
| Residue + Loss | 7.9 | |

## Residue.

| | |
|---|---|
| Yield | 12.8 cwts/ton. |
| Ash Content | - |

No coking properties shown.

Reference. Report - page 338

Time of Carbonisation 11 hrs. Final Temperature - 70 °C

**Scrubber Naphtha**

| | |
|---|---|
| Yield | 1.33 gals/ton. |
| S.G. | 0.73 |

**Crude Oil**

| | |
|---|---|
| Yield | 79.8 gals/ton. |
| S.G. | 0.87 |
| Setting Point | 8.3°C |
| Wax Content | 10.0% |

**Light Spirits**

| | |
|---|---|
| Yield | 13.9 gals/ton |
| S.G. | 0.77 |

Percentage Crude distilling to 180°C
17.5%

**Vacuum Distillation** Pressure 4mm.

| Fraction | Percent Distillate | Total Percent Distillate |
|---|---|---|
| 0-100°C | 8.1 | 25.6 |
| 100-150° | 17.4 | 43.0 |
| 150-200° | 17.1 | 60.1 |
| 200-250° | 19.3 | 79.4 |
| 250-300° | 10.0 | 89.4 |

Residue + Loss 10.6

**Residue**

| | |
|---|---|
| Yield | 11.9 cwts/ton. |
| Ash Content | — |

No coking.

Reference. Report - page 339

Time of Carbonisation 11hrs. Final Temperature - 500°C

### Scrubber Naphtha

| | |
|---|---|
| Yield | 1.37 gals/ton |
| S.G. | 0.685 |

### Crude Oil

| | |
|---|---|
| Yield | 46.5 gals/ton |
| S.G. | 0.911 |
| Setting Point | 17.8°C |
| Wax Content | 17.8% |

### Light Spirits

| | |
|---|---|
| Yield | 6.27 gals/ton. |
| S.G. | 0.776 |

Percentage Crude Distilling to 180°C 13.5%

### Vacuum Distillation. Pressure 3 mn.

| Fraction | Percent Distillate | Total Percent Distillate |
|---|---|---|
| 0 - 100°C | 1 | 14.5 |
| 100 - 150° | 7.3 | 21.8 |
| 150 - 200° | 13.5 | 35.3 |
| 200 - 250° | 16.5 | 51.8 |
| 250 - 300° | 21.5 | 73.3 |
| Residue + Loss | 26.7 | |

### Residue.

| | |
|---|---|
| Yield | 12.4 cwts./ton. |
| Ash Content | 5.7 |

No coking was shown.

Reference. Report - page 340

Time of Carbonisation 8hrs. Final Temperature - 580°C

**Scrubber Naphtha**

| | |
|---|---|
| Yield | 0.74 gals/ton. |
| S.G. | 0.697 |

**Crude Oil**

| | |
|---|---|
| Yield | 55 gals/ton. |
| S.G. | o.906 |
| Setting Point | 18°C |
| Wax Content | 23.1% |

**Light Spirits**

| | |
|---|---|
| Yield | 9.1 gals/ton. |
| S.G. | 0.778 |

Percentage Crude Distilling to 180°C

16.5%

**Vacuum Distillation.** Pressure 6 mm.

| Fraction | Percent Distillate | Total Percent Distillate |
|---|---|---|
| 0 - 100°C | 8.5 | 25 |
| 100 - 150° | 13.0 | 38 |
| 150 - 200° | 19.0 | 57 |
| 200 - 250° | 21.5 | 78 |
| 250 - 300° | 14.5 | 93 |
| Residue + Loss | 7 | |

**Residue.**

| | |
|---|---|
| Yield | 12.1 cwts/ton |
| Ash Content | 11.5% |

No signs of coking.

Reference. Report - page 341

Time of Carbonisation 10hrs. Final Temperature - 600°C

**Scrubber Naphtha**

| | |
|---|---|
| Yield | 1.3 gals/ton |
| S.G. | 0.766 |

**Crude Oil**

| | |
|---|---|
| Yield | 48.0 gals/ton |
| S.G. | 0.92 |
| Setting Point | 26.7°C |
| Wax Content | 8.4% |

**Light Spirits**

| | |
|---|---|
| Yield | 6.7 gals/ton |
| S.G. | 0.78 |

Percentage Crude Distilling to 180°C

14%

**Vacuum Distillation.** Pressure 3 mn.

| Fraction | Percent Distillate | Total Percent Distillate |
|---|---|---|
| 0 - 100°C | 17.0 | 31.0 |
| 100 - 150° | 15. | 46 |
| 150 - 200° | 19.0 | 66 |
| 200 - 250° | 17.5 | 82.5 |
| 250 - 300° | 9.5 | 92 |
| Residue + Loss | 8.0 | |

**Residue.**

| | |
|---|---|
| Yield | 9.4 cwts/ton |
| Ash Content | -- |

Not a true coke.

Reference. Report - page 342

Time of Carbonisation 10hrs. Final Temperature -

**Scrubber Naphtha**

| | |
|---|---|
| Yield | 1.0 gals/ton. |
| S.G. | 0.68 |

**Crude Oil**

| | |
|---|---|
| Yield | 41 gals/ton. |
| S.G. | 0.937 |
| Setting Point | 16.1°C |
| Wax Content | - - |

**Light Spirits**

| | |
|---|---|
| Yield | 5.33 gals/ton. |
| S.G. | 0.771 |

Percentage Crude Distilling to 180°C

13%

**Vacuum Distillation.** Pressure 3 mn.

| Fraction | Percent Distillate | Total Percent Distillate |
|---|---|---|
| 0 - 100°C | 17.5 | 30.5 |
| 100 - 150° | 15 | 45.5 |
| 150 - 200° | 20 | 65.5 |
| 200 - 250° | 16 | 81.5 |
| 250,- 300° | 9 | 90.5 |
| Residue + Loss | 9.5 | |

**Residue.**

| | |
|---|---|
| Yield | 13.6 cwts/ton. |
| Ash Content | 26.2% |

Residue dull. No binding.

Reference. Report - page 343

Time of Carbonisation 10hrs. Final Temperature - 700°C

**Scrubber Naphtha**

| | |
|---|---|
| Yield | 0.98 |
| S.G. | 0.77 |

**Crude Oil**

| | |
|---|---|
| Yield | 40 gals/ton. |
| S.G. | 0.96 |
| Setting Point | 14.5°C |
| Wax Content | 3% |

**Light Spirits**

| | |
|---|---|
| Yield | 8.4 gals/ton. |
| S.G. | 0.82 |

Percentage Crude Distilling to 180°C

21%

**Vacuum Distillation.** Pressure 3.5 mn.

| Fraction | Percent Distillate | Total Percent Distillate |
|---|---|---|
| 0 - 100°C | 9 | 30 |
| 100 - 150° | 17.5 | 47. |
| 150 - 200° | 15 | 60 |
| 200 - 250° | 21 | 81 |
| 250 - 300° | 7.5 | 88.5 |
| Residue + Loss | 11.5 | |

**Residue.**

| | |
|---|---|
| Yield | 12.7 cwts/ton |
| Ash Content | 6.5% |

An excellent coke. The charge had been plastic during the carbonisation, and the result was a hard strong coke with a good lustre.

Reference. Report - page 344

Time of Carbonisation 10hrs Final Temperature - 550°C

**Scrubber Naphtha**

| | |
|---|---|
| Yield | 1.27 gals/ton. |
| S.G. | 0.69 |

**Crude Oil**

| | |
|---|---|
| Yield | 38.8 gals/ton. |
| S.G. | 0.95 |
| Setting Point - C | |
| Wax Content | - |

**Light Spirits**

| | |
|---|---|
| Yield | 4.8 gals/ton |
| S.G. | 0.81 |

Percentage Crude Distilling to 180°C 16.1%

**Vacuum Distillation.** Pressure 5 mn.

| Fraction | Percent Distillate | Total Percent Distillate |
|---|---|---|
| 0 - 100°C | 11.8 | 27.9 |
| 100 - 150° | 16.4 | 44.3 |
| 150 - 200° | 11.4 | 55.7 |
| 200 - 250° | | |
| 250 - 300° | Distillation Stopped. | |

Residue + Loss ---

**Residue.**

| | |
|---|---|
| Yield | 13.3 cwts/ton. |
| Ash Content | 11% |

Some coking took place, although the charge had not been completely plastic.

Reference. Report - page 345

Time of Carbonisation 9 hrs. Final Temperature - 610°C

**Scrubber Nanhtha**

| | |
|---|---|
| Yield | 0.2 gals/ton. |
| S.G. | 0.737 |

**Crude Oil**

| | |
|---|---|
| Yield | 32.7 gals/ton. |
| S.G. | 0.945 |
| Setting Point | 12.8°C |
| Wax Content | --- |

**Light Spirits**

| | |
|---|---|
| Yield | 4.9 gals/ton. |
| S.G. | 0.789 |

Percentage Crude Distilling to 180°C

16%

**Vacuum Distillation.** Pressure 6 mn.

| Fraction | Percent Distillate | Total Percent Distillate |
|---|---|---|
| 0 - 100°C | 13 | 29 |
| 100 - 150° | 13.5 | 42.5 |
| 150 - 200° | 21.5 | 64 |
| 200 - 250° | 16.5 | 80.5 |
| 250 - 300° | Not distilled | |
| Residue + Loss | 19.5 | |

**Residue.**

| | |
|---|---|
| Yield | 15.1 cwts/ton |
| Ash Content | 10.0% |

The residue was not a coke

Reference. Report - page 346

Time of Carbonisation 10½ hrs. Final Temperature - 725°C

**Scrubber Naphtha**

| | | |
|---|---|---|
| | Yield | 1.0 gals/ton. |
| | S.G. | 0.73 |

**Crude Oil**

| | | |
|---|---|---|
| | Yield | 41 gals/ton. |
| | S.G. | 0.94 |
| | Setting Point | 13.3°C |
| | Wax Content | 13.7% |

**Light Spirits**

| | | |
|---|---|---|
| | Yield | 7.3 gals/ton. |
| | S.G. | 0.78 |

Percentage Crude Distilling to 180°C

17.5%

**Vacuum Distillation.** Pressure 3 mm.

| Fraction | Percent Distillate | Total Percent Distillate |
|---|---|---|
| 0 - 100°C | 17.6 | 35 |
| 100 - 150° | 18.5 | 53.5 |
| 150 - 200° | 21 | 74.5 |
| 200 - 250° | 15.5 | 90 |
| 250 - 300° | 7 | 97 |
| Residue + Loss | 3 | |

**Residue.**

| | | |
|---|---|---|
| | Yield | 12.5 cwts/ton. |
| | Ash Content | ---- |

Dull Residue. Little Binding.

Reference. Report - page 347

Time of Carbonisation 10 hrs. Final Temperature - 690°C

**Scrubber Naphtha**

| | |
|---|---|
| Yield | 1.2 gals/ton. |
| S.G. | 0.69 |

**Crude Oil**

| | |
|---|---|
| Yield | 67.2 gals/ton. |
| S.G. | 0.91 |
| Setting Point | 15°C |
| Wax Content | --- |

**Light Spirits**

| | |
|---|---|
| Yield | 10.8 gals/ton. |
| S.G. | 0.77 |

Percentage Crude Distilling to 180°C
16%

**Vacuum Distillation.** Pressure 3 mn.

| Fraction | Percent Distillate | Total Percent Distillate |
|---|---|---|
| 0 - 100°C | 9.0 | 25.0 |
| 100 - 150° | 15.5 | 40.5 |
| 150 - 200° | 20.0 | 60.5 |
| 200 - 250° | 21.5 | 82.0 |
| 250 - 300° | 11.0 | 93.0 |
| Residue + Loss | 7.0 | |

**Residue.**

| | |
|---|---|
| Yield | 9.75 cwts/ton. |
| Ash Content | |

Coking properties - negligible

Reference. Report - page 348

Time of Carbonisation 10 hrs. Final Temperature - 620°C

**Scrubber Naphtha**

| | |
|---|---|
| Yield | 0.77 gals/ton |
| S.G. | 0. 73 |

**Crude Oil**

| | |
|---|---|
| Yield | 23.6 gals/ton. |
| S.G. | 0.90 |
| Setting Point | 19°C |
| Wax Content | 5.96% |

**Light Spirits**

| | |
|---|---|
| Yield | 3.3 gals/ton. |
| S.G. | 0.79 |

Percentage Crude Distilling to 180°C
14%

**Vacuum Distillation.** Pressure 3 mn.

| Fraction | Percent Distillate | Total Percent Distillate |
|---|---|---|
| 0 - 100°C | 1 .3 | 27.3 |
| 100 - 150° | 17.8 | 45.1 |
| 150 - 200° | 17.0 | 62.1 |
| 200 - 250° | 22.0 | 84.1 |
| 250 -.300° | 12.0 | 96.1 |

Residue + Loss 4

**Residue.**

| | |
|---|---|
| Yield | 15.8 cwts/ton. |
| Ash Content | -- |

Binding nil. No coking.

Reference. Report - page 349

Time of Carbonisation 8 hrs Final Temperature - 600°C

<u>Scrubber Naphtha</u>

| | |
|---|---|
| Yield | 0.95 gals/ton. |
| S.G. | 0.706 |

<u>Crude Oil</u>

| | |
|---|---|
| Yield | 47 gals/ton |
| S.G. | 0.919 |
| Setting Point | 21.7°C |
| Wax Content | 12.1% |

<u>Light Spirits</u>

| | |
|---|---|
| Yield | 7.66 gals/ton. |
| S.G. | 0.800 |

Percentage Crude Distilling to 180°C

16.3%

<u>Vacuum Distillation.</u> Pressure 7 mn.

| Fraction | Percent Distillate | Total Percent Distillate |
|---|---|---|
| 0 - 100°C | 8 | 24.3 |
| 100 - 150° | 12.5 | 36.8 |
| 150 - 200° | 17.5 | 54.3 |
| 200 - 250° | 15 | 69.3 |
| 250 - 300° | 24 | 93.3 |

Residue + Loss 6.7

<u>Residue.</u>

| | |
|---|---|
| Yield | 13.3 cwts/ton. |
| Ash Content | 36.4% |

No coking.

## TABLE GIVING POSITIONS OF SAMPLES.

| AREA | PAGE IN THESIS. | SEAM | OTHER DETAILS | REFERENCE NUMBER. * |
|---|---|---|---|---|
| Fife | 41 | Capeldrae Cannel | Lochore Basin | 30 |
| | 42 | Upper Five Feet | - | 12 |
| | 43 | Top Diamond | Same seam at different points | - |
| | 44 | Jersey | | - |
| | 45 | Dysart Lower | Sample "A" - 6" cannel | |
| | 46 | " " | Sample "B" - 14" cannel | 22 |
| | 47 | " " | Sample "C" - 9" cannel | |
| Lothians | 48 | Great Seam | Newbattle Cannel | - |
| Douglas | 49 | Nine Foot | - | - |
| | 50 | " " | - | - |
| | 51 | Gaberschit | Assoc. Big Drum | - |
| | 52 | Big Drum | Gas Coal | 2 |
| | 53 | Nine Foot | 5" cannel | - |
| | 54 | Kirkroad | Gas Coal | 3 |
| | 55 | Robb | Gas Coal | 4 |
| | 56 | Auchenheath Wee | Gas Coal | 33 |
| Lanarkshire | 57 | Virtuenell | Assoc. Shale | 37 |
| | 58 | Kiltongue Mussel Band | Gas Coal | 42 |

* Further details are given opposite these numbers in the Second Report of the Oil from Coal Committee, in Tables III and IV.

## CONCLUSIONS.

The principle variation shown over the range of cannels tested is in yield of tar: the tars resemble each other in most respects. The most obvious difference between the tars is in the amount of wax they contain. Thus while some tars appeared thin and comparatively mobile, others set almost solid at room temperatures. The principle figures for the crude tar are :-

| | Minimum | Maximum |
|---|---|---|
| Specific Gravity | 0.87 | 0.96 |
| Yield (gals/ton) | 40 | 82 |
| Percent distillate to 180°C | 11% | 21% |
| Residue over 300°C at 3-6 mm. press | 3% | 10% |
| Wax Content | 3% | 23% |

It is easily seen that, along with the yield, the wax content shows remarkable variation. The ability to handle, and especially to dispose of this wax might be a deciding factor in the choice between the various cannels which might be available.

The variation in spirit yield is not so marked, and is probably within the range of a normal distillation unit.

## LIGHT SPIRIT.

This fraction, a yellowish, mobile liquid, turning deep red on standing, varies somewhat in yield, but as can be seen from the percentage of the crude oil distilling to 180°C (above), it forms a more constant proportion of the tar.

| | Minumum | Maximum |
|---|---|---|
| Specific Gravity | 0.76 | 0.82 |
| Yield (gals/ton) | 5.3 | 14.2 |

## SCRUBBER NAPHTHA.

This light spirit scrubbed from the gas was stable and water white. In practice it would certainly form an excellent motor fuel, although the small amount available made testing impossible.

| | Minimum | Maximum |
|---|---|---|
| Specific Gravity | 0.66 | 0.787 |
| Yield (gals/ton) | 0.74 | 1.84 |

## RESIDUE.

| | Minimum | Maximum |
|---|---|---|
| Yield (cwts/ton) | 9.4 | 15.8 |

The residue in most cases was non-coking, although certain cannels (see pp. 43,52 ) gave strong cokes even at the low temperature of carbonisation. Only a few of the cokes examined had an ash content of less than 5%, which the Oil from Coal Committee of the Scottish Development Council recommend as the maximum permissable in a smokeless fuel. When attempts were made to burn these residues in an open fire, they soon became covered with a white ash which gave them a very unattractive appearance, besides slowing down combustion. This is probably due as much to the manner in which the mineral matter is dispersed throughout the coal as to the actual quantity of ash.

## GAS.

The gas has a high calorific value - about 1000 B.Th.U. and a yield of about 1500-2000 cu.ft. per ton.

In an independent report, it was described as suitable for synthetic conversion to liquid hydrocarbons.

It can be seen that over the range of cannels tested, the properties, so far as oil production is concerned, are uniformly good. It can thus be assumed that any cannels giving a satisfactory oil yield on Gray-King assay, will give an oil of properties similar to those described in this chapter.

# CHAPTER 3

## THE EXAMINATION OF A DYSART CANNEL TAR.

While the results given in the preceding chapter are interesting from the survey point of view, the small scale on which the tests were carried out made it impossible to evaluate the products effectively as motor spirit, diesel oil, lubricating oil and other products. For this reason it was felt that a larger quantity of tar should be obtained and examined in greater detail. Also sufficient spirit was required to enable an engine test to be done and its Octane Rating determined.

Accordingly, a half ton sample of Dysart Cannel was sent to the Fuel Research Station, and carbonised there in the Fuel Research mild steel horizontal retorts. (Fuel Research Technical Paper No. 32, 30 (D.S.I.R.) )

## CARBONISATION.

The following are the details of the carbonisation, as supplied by the Director of Fuel Research.

"At the request of the Oil from Coal Committee of the Scottish National Development Council, the Director of Fuel Research agreed to carbonise a half ton sample of cannel at low temperatures for the purpose of preparing a quantity of tar. Further, in order to assist the Committee in its examination of this tar, the Director agreed to carry out a short investigation of the behaviour of the tar on hydrogenation.

The sample of cannel was supplied from the Lower Dysart Seam, Francis Pit, Dysart Colliery, Fife. The coal was carbonised in 100 lb. quantities in a horizontal, mild-steel retort heated to an external carbonising temperature of 550°C.

The yields obtained were as follows:-

### CARBONISATION OF CANNEL

Yields per ton at 2.1% moisture

| | |
|---|---|
| Coke | 10.4 cwt. |
| Gas | 5010 c.ft. |
| Gas | 67.9 therms. |
| Gas, S.G. (air=1) | 0.936 |
| Tar (dry) | 52.0 gal. |
| Liquor | 11.0 gal. |
| Liquor, S.G. | 1.005 |

Analysis of Gas.

| | |
|---|---|
| $CO_2$ | 6.4% |
| $C_nH_m$ | 25.8 |
| $CO$ | 3.1 |
| $H_2$ | 11.4 |
| $C_nH_{2n+2}$ | 46.0 |
| $N_2$ | 7.3 |
| $n$ | 1.64 |
| C.V. | 1355 B.Th.U. |

The tar obtained from this carbonisation was subjected to a hydrogenation-cracking process at the Fuel Research Station, and the Report continues:-

"The composition of the tar made in the present examination was as follows:-

| | |
|---|---|
| C | 85.5% |
| $H_2$ | 10.8 |
| S | 0.5 |
| $N_2$ | 0.6 |
| $O_2$ | 2.6 |
| | 100.0 |

It may be calculated from this analysis that the disposable hydrogen is 10.3%, and the H/C atomic ratio is thus 1.45. The H/C ratio of Fuel Research Brick-Retort Tar is 1.0.

Consideration of the H/C ratio of cannel and of the tar produced shows that cannel tar should be readily amenable to hydrogenation, and experiment has shown that even cannel tar made during high-temperature carbonisation is as readily treated as low-temperature bituminous coal tar. It follows that low-temperature cannel tar will certainly make an excellent raw material for the hydrogenation-cracking process.

A short test was accordingly conducted in order to compare the results of hydrogenation of low-temperature cannel tar and Fuel Research Brick-Retort tar.

## APPARATUS AND METHOD OF OPERATION.

An experimental plant having a normal throughput of 12 ml. of tar per hour was used for the investigation. Tar and hydrogen at 200 atmospheres pressure passed downwards through an electrically-heated reaction vessel containing catalyst. The products are withdrawn at the bottom and pass to a separator. The half-hourly discharge of the liquid product and the rate of residual gas are controlled by needle valves at the bottom and top respectively of the separator.

The conditions of operation used were:-

(a) A reaction pressure of 200 atmospheres.

(b) A reaction temperature of 480°C.

(c) A rate of residual gas of 0.24 c.ft. per hour; the throughput of the plant was controlled by the rate of residual gas, the rate of hydrogen being adjusted to maintain the pressure constant.

(d) A throughput of tar of 12 ml. per hour; the corresponding throughput of hydrogen was approximately o.48 c.ft. per hour.

## MATERIALS AND METHODS OF ANALYSIS.

### Tar.

The tar and hydrogenation products were extracted with 20% solium hydroxide solution to determine tar acids; the neutral oils (130 gm.) from the products were then fractionated from a 250 ml. flask fitted with an eight-pear fractionating column. The cannel tar was dry and contained approximately 4% of tar acids and its specific gravity at 15°C was 0.910.

### Catalyst.

The catalyst used was granular alumina gel. (1/8 - 1/16 inch) impregnated with molybdic oxide. During the early stages of operation, the molybdic oxide is converted to molybdenum sulphide by the action of the hydrogen sulphide produced by the hydrogenation of sulphur compounds in the tar.

### Hydrogen.

The hydrogen contained 98% of $H_2$.

### Results of Hydrogenation-Cracking.

The tar was treated at a temperature of 480°C. Two test-periods were carried out, one of 12 hours and the second of 24 hours duration. The yields in the two periods were respectively (1) oil, 89.7 and 90.0%; (2) water, 3.4 and 3.9%; and (3) gas, approximately 9%. The analysis of the liquid products are given in Table 1.

## TABLE 1

### Analysis of Liquid Products.

| Basis of Calculation | On Crude Product. | | On Original Dry Tar. | |
|---|---|---|---|---|
| Period | 1 | 2 | 1 | 2 |
| Water | - | - | 3.4 | 3.9 |
| Oil | 100.0 | 100.0 | 89.7 | 90.0 |
| Total Product | 100.0 | 100.0 | 93.1 | 93.9 |
| S,G$_{15}$ of Oil Product | 0.801 | 0.801 | 0.801 | 0.801 |
| Analysis of Oil Product | | | | |
| Tar Acids | 0.4 | 0.5 | 0.4 | 0.4 |
| Neutral Oil | 99.6 | 99.5 | 89.3 | 89.6 |
| Total | 100.0 | 100.0 | 89.7 | 90.0 |
| Fractionation of Neutral Oil | | | | |
| Up to 100°C | 8.5 | 8.1 | 7.6 | 7.3 |
| 100 - 170 | 27.7 | 27.4 | 24.9 | 24.7 |
| 170 - 200 | 12.5 | 12.7 | 11.3 | 11.4 |
| 200 - 230 | 13.2 | 13.3 | 11.8 | 12.0 |
| 230 - 270 | 15.2 | 15.6 | 13.6 | 14.0 |
| Residue | 21.3 | 21.3 | 19.1 | 19.2 |
| Loss | 1.2 | 1.1 | 1.0 | 1.0 |
| Total | 99.6 | 99.5 | 89.3 | 89.6 |

To face page 66

These were colourless liquids almost entirely free from tar acids. The percentage yield of spirit, calculated on the basis of original tar, was 43.8 in the first period and 43.4 in the second period. The spirit yield from Fuel Research Brick Retort bituminous coal tar treated under similar conditions is 40.5% in the first period and 39.7% in the second. During the short test carried out, the catalyst showed very little deterioration.

This tar is therefore an excellent raw material for hydrogenation-cracking in the vapour phase."

The work described below is an attempt to evaluate the fractions obtained by distilling the crude tar, and to investigate their suitability for commercial use as motor spirit, diesel oil, and lubricating oil.

When the tar is compared with that obtained from the same cannel carbonised in the small retort described previously, it is obvious that the conditions of distillation are greatly different from these described in Chapter 2.

The greatest variation is to be seen in the gas evolved during carbonisation. This is shown in Table III, which gives the analysis of the gas obtained at the Fuel Research Station from the Dysart Cannel, along with the analysis of a typical gas obtained from the small retort. (Unfortunately particulars of the gas from the Dysart Cannel in the small retort are not available.)

## TABLE III

| | Gas from: Fuel Research Station Horizontal Retort | Small Retort (Chap. 2) |
|---|---|---|
| $CO_2$ | 6.4% | 3.7% |
| $O_2$ | - | 0.5% |
| $C_nH_m$ | 25.8% | 5.0% |
| CO | 3.1% | 2.1% |
| $H_2$ | 11.4% | 12.0% |
| $C_nH_{2n+2}$ | 46.0% | 76.4% |
| $N_2$ | 7.3% | 0.9% |

It will be seen that the gas from the Fuel Research Board retort is very high in unsaturated hydrocarbons. On the other hand, the gas from the small retort shows only 5% unsaturateds. This is explained by a comparison of the methods of carbonisation.

In the case of the Fuel Research Retort (Fuel Research Technical Paper No. 32/30), the coal is contained in shallow metal trays which are charged into the heated retort, which was at 550°C in the case of the Dysart cannel. The walls of the trays prevent the tar vapours from passing through the centre of the charge. The products of carbonisation are thus forced into contact with the hot walls of the retort, with consequent cracking. While this will hold for any material charged into such a retort, it is particularly important in the case of cannel, which is very sensitive to excessive heat.

The small retort on the other hand is heated slowly up to its maximum temperature, so that the walls are not much hotter than the temperature of distillation of the vapour at any particular time during the run. The conditions in this retort are such, therefore, as will reduce cracking to a minimum.

On the following page the routine tests on the crude Dysart tar are given, along with the tests on the tar obtained from the same cannel carbonised in the small retort. Here again the differences between the tars all indicate the greater cracking taking place in the Fuel Research Board retort. The Fuel Research Board tar had a Setting Point of - 2°C and gave a high yield of light oil and heavy residue. The tar from the small retort had a Setting Point of 10°C, and the oil showed less trace of cracking. This would indicate that the middle fractions (possibly valuable as a lubricating oil) have been cracked, giving two types of oil, one lighter and one heavier than the original. This process, of course, if carried further, would give a large yield of cracked spirit, together with pitch and coke.

[illegible]

| | | |
|---|---|---|
| Yield, gals/ton | [illegible] | [illegible] |

Crude Wax.

| | | |
|---|---|---|
| Yield, gals/ton | 52 | 55.5 |
| S.G. | 0.91 | 0.87 |
| Setting Point, °C | 1.7° | 9.4° |
| Wax Content | – | 8.6 |

Light Spirits.

| | | |
|---|---|---|
| Yield, gals/ton | 9.56 | 14.5 |
| S.G. | 0.80 | 0.7 |
| Per cent distilling to 150°C | 13.4% | 16.5 |

Vacuum Distillation. (at 6mm.) (at 4

| Fraction | % Distillate | Total % * Distillate | % Distillate |
|---|---|---|---|
| Up to 100°C | 13.2 | 31.6 | 10.2 |
| 100° – 150° | 17.0 | 48.6 | [illegible] |
| 150° – 200° | 17.5 | 66.1 | [illegible] |
| 200° – 250° | 16.2 | 82.3 | [illegible] |
| 250° – 300° | 12.2 | 94.5 | 10.5 |
| Residue + Loss | 5.5 | – | 7.8 |

* including light spirit.

Residue.

## Carbonisation of Dysart Cannel.

| | F.R.B. Retort. I | Small Retort. II |
|---|---|---|
| **Scrubber Naphtha.** | | |
| Yield, gals/ton | 1.4 | 1.84 |
| S.G. | 0.787 | 0.66 |
| **Crude Tar.** | | |
| Yield, gals/ton | 52 | 85.2 |
| S.G. | 0.91 | 0.87 |
| Setting Point, °C | 1.7° | 9.4° |
| Wax Content | - | 8.5% |
| **Light Spirits.** | | |
| Yield, gals/ton | 9.56 | 14.2 |
| S.G. | 0.80 | 0.76 |
| Per cent distillate to 180°C | 18.4% | 16.8% |
| **Vacuum Distillation.** | (at 6mm.) | (at 4mm.) |

| Fraction | % Distillate | Total % * Distillate | % Distillate | Total % * Distillate |
|---|---|---|---|---|
| Up to 100°C | 13.2 | 31.6 | 10.2 | 27.0 |
| 100° - 150° | 17.0 | 48.6 | 13.8 | 40.8 |
| 150° - 200° | 17.5 | 66.1 | 19.5 | 60.3 |
| 200° - 250° | 16.2 | 82.3 | 21.5 | 81.8 |
| 250° - 300° | 12.2 | 94.5 | 10.3 | 92.1 |
| Residue + Loss | 5.5 | - | 7.9 | - |

* Including light spirit.

**Residue.**

| | | |
|---|---|---|
| Yield, cwts/ton | 10.4 | 12.8 |

In spite of the difference in the nature of the products obtained by the two methods of carbonisation, the Fuel Research Board tar, the only one available in large quantities, was fractionated, and the fractions examined as possible fuels and lubricating oils. The results given here serve as a useful indication of the character of the oils given by the low temperature carbonisation of the cannel coal.

The tar fractionated into three main products:-

1. Motor spirit - up to 190°C, approx. 22% of the tar.
2. Diesel oil - 190° to 345°, 39% of the tar.
3. Lubricating oil. - 345° to 450°, approx. 28% of the tar.

These fractions will now be treated separately.

## MOTOR SPIRIT.

### Preliminary Tests.

Preliminary testing was done on samples topped from litre quantities of crude tar. The topping was first done to 180°C, as was usual with these tars (Chap. 2). It can be seen in Table IV that the end point of the spirit is below 200°C. As an end point of 200°C is permissible in a commercial spirit, a further quantity was topped to 200°. This brought the end point slightly above 200°, but gave a great increase in yield. It will be seen that in both cases the distillate to 100° is low. According to modern requirements for motor spirit it should be between 30% and 33%. Thus as blending would have to be resorted to in any case, it would probably be more profitable to distil off to the higher temperature and so obtain the benefit of the increased yield.

## TABLE IV.

| Tar topped to | 180°C. | 200°C. |
|---|---|---|
| Up to 100°C | 11% | 4% |
| 125° | 42% | 29% |
| 150° | 69% | 54% |
| 175° | 87% | 76% |
| End Point 196°C | 94% | |
| Up to 200°C | - | 91% |
| End Point 210°C | - | 94% |
| Percentage Distillate from tar | 18.4% | 27.3% |
| Yield in gals/ton coal | 9.6gals. | 14.2gals. |

The spirit to 200°C was then subjected to various tests, and the effect of refining on its properties was briefly investigated. This small sample of refined spirit was then compared with a commercial specification to find if any modification would be necessary during the large scale topping and refining of a sample for an engine test.

The tests applied are described below.

## Specific Gravity.

This was determined by the pyknometer at 15°C relative to water at 15°C.

## Bromine Number.

The Francis method (Ind. and Eng. Chem. 1926. XVIII - 821) was used to determine the Bromine Number of the spirit, and by using an approximate molecular weight a figure for the percentage unsaturateds was obtained. The molecular weight used is estimated as 54 + $\frac{1}{2}$ mean boiling point.

## Total Sulphur.

The apparatus used was a slightly modified form of that described in the standard I.P.T. method (I.P.T. G4). The method used was the standard one.

## Refining.

The spirit was given a thorough preliminary refining in order to investigate the properties of the refined oil. The results of the tests carried out on the refined and unrefined spirit are given in Table V.

The refining process consisted of:-

(a) Washing with successive 2% quantities of the sulphuric acid, S.G. 1.84 (six washes).

(b) Washing with water (once or twice).

(c) Washing with 5% quantities of caustic soda solution.

The solution was of 30% strength (two washes).

(d) Washing with very dilute sulphuric acid.

(e) Washing with water.

(f) Distilling to 200°C.

This last distillation invariably caused decomposition of various sulphur compounds, and a large amount of $SO_2$ was liberated. This was therefore followed by

(g) Washing lightly with alkali, followed by water.

The spirit refined in this way was a stable water white liquid of good odour.

## TABLE V.

### Properties of the Spirit to 200°C before and after Refining.

| | Refined. | Unrefined. |
|---|---|---|
| Specific Gravity | 0.83 | 0.79 |
| Bromine Number | 44.3 | 89.4 |
| Approx. percentage unsaturateds | 36% | 72% |
| Total Sulphur | 0.12 | 0.27 |
| Distillation Range. | | |
| Drop Point | 60° C | 60° C |
| Up to 100°C | 2.0% | 3.5% |
| 125° | 18.5 | 26.0 |
| 150° | 59.6 | 54.5 |
| 175° | 89.2 | 86.0 |
| 200° | 96.5 | 93.5 |
| Residue | 3.3 | 5.6 |
| Loss on Distillation | 0.2 | 0.9 |
| Total Loss on Refining | 25% | |
| Colour | water white<br>stable | Dark red<br>unstable |
| Odour | Fairly good | bad - smelling of sulphur compounds |

As can be seen from Table VI the refined spirit compares favourably with commercial specifications as regards colour, sulphur, etc. The distillate to 100°C and the 70% distillate, however, show that blending is necessary (blending with the scrubber naphtha is considered later - p 70 ).

TABLE VI.

| | Commercial Spirit | Cannel Spirit |
|---|---|---|
| Colour | Water white | Water white |
| Odour | Merchantable | Merchantable |
| Distillate to 100°C | 30/33% | 2% |
| Final boiling point | Not above 200°C | Over 200°C |
| 70% distillate | Not higher than 145°C | 159°C |
| Total Sulphur | Not over 0.1% | 0.12% |
| Doctor Test | Negative | Negative |

From the figures for distillation given on p. 75 , it can be seen that the final boiling point is over 200°C. It was decided therefore to top to 190°C when distilling the large sample.

## Large Scale Topping and Refining.

The tar was topped in the 25 gallon still and fractionating column recently presented to the Technical College. A 13 gallon batch was topped, and a distillate of 22.2% was collected. This represented topping to 190°C. This sample was then refined.

## Refining.

As facilities were not available for large scale refining, the spirit (about 3 gallons) was refined in small batches. Quantities of 1½ litres were shaken with the refining agent in Winchester quart bottles, and the sludges were syphoned off. The washed spirit was then steam distilled, the final products being combined.

The refining consisted of eight washes:-

One wash with 20% caustic sode (10% quantity).

One wash with water.

Two washes with 60% sulphuric acid (10% quantities).

One wash with water.

A light wash with caustic as before.

Two washes with water.

Followed by steam distillation.

The total loss on refining was 23%.

## Testing.

The distillation range of the refined spirit obtained in this way is given in Table VII.

## TABLE VII

### Distillation Range of Refined Spirit to 190°C.

| | Percent Distillate | Fraction |
|---|---|---|
| Drop Point | 60°C. | |
| Up to 100°C | 1% | 1% |
| 125°C | 26% | 25% |
| 150°C | 59% | 34% |
| 175°C | 89% | 55% |
| End Point | 189°C. | |
| Loss on Distillation | 1% | |

The principal object in obtaining this large sample was to enable an engine test to be done. This was carried out by Scottish Oils, to whom grateful acknowledgement is made. They gave a figure of 58 for the Octane Number, determined on the C.F.R. engine, Research Method. This is disappointingly low, but the absence of the gas spirit fraction may account in part for this.

With the object of improving the Octane rating of the spirit, the effect of blending and doping the spirit was investigated, Scottish Oils again kindly undertaking the engine tests.

Three blends were made up.

(a) Blending with Benzol.

This was a simple blend of 2 parts spirits to one part 90's benzol. The Octane Number (C.F.R.) was raised from 58 to 68.5.

(b) Doping with Tetra Ethyl Lead.

The spirit was treated with tetra ethyl lead in the proportions of 2 c.c. T.E.L. per gallon of spirit. The Octane Number after this treatment was 66. This is lower than that obtained by blending with benzol, although the concentration of T.E.L. is relatively high.

(c) Doping with Tetra Ethyl Lead in Presence of Aniline.

It has been shown (Tonberg, Quiggle, Fry and Fenske. Ind. Eng. Chem. 28, 792) that the anti-knock effect of aniline is additive to that of tetra ethyl lead. A blend was accordingly made up containing 2 c.c. of T.E.L. per gallon of spirit, in addition to 3% of aniline. This blend gave the very satisfactory Octane Number of 74.5. If the presence of the aniline does not adversely affect other properties (gum formation, stability, etc.) this might be a possible method of improving the spirit.

Scrubber Naphtha.

The gas from the carbonisation of the Dysart Cannel was scrubbed, and the gas spirit recovered. The yield was 1.4 gals/ton, and as this naphtha is a light volatile spirit, it should be very suitable for blending to give a motor spirit.

TABLE IX

## Comparison of Typical Motor Spirits with Cannel Spirits.

| | Scrubber Naphtha | Refined Light Spirit | Gas Spirit (J. and K.ref.1) | Shell * B.P. (Commercial) | R.O.P. * |
|---|---|---|---|---|---|
| Specific Gravity | 0.787 | 0.830 | 0.745 | 0.745 | 0.741 |
| Initial Boiling Pt. | 39°C | 60°C | - | 35°C | 45°C |
| Dist. up to 75°C | 41% | 1% | 0.6% | 15% | 10% |
| 100° | 77% | 1% | 38% | 29% | 30% |
| 125° | 89% | 26% | 76% | 49% | 58% |
| 150° | 93% | 59% | 94% | 71% | 86.5% |
| 175° | - | 89% | - | 90% | 95% |
| End Point | - | 189° | - | 192° | 190° |
| Residue | 2.5% | 10% | 6% | 2% | 1.5% |
| Loss | 4.5% | 1% | | 3% | 1.5% |

* From the Automobile Engineer October 1936.

To face page 80

## TABLE VIII.

## Distillation Range of Scrubber Naphtha (unrefined)

| | Percent Distillate | Fraction |
|---|---|---|
| Drop Point | 39°C | |
| Up to 75°C | 41% | 41% |
| 100° | 77% | 36% |
| 125° | 89% | 12% |
| 150° | 93% | 4% |
| Residue | 2.5% | |
| Loss | 4.5% | |

Table VIII shows that this naphtha is too light to be used alone as a motor spirit (see Table IX), but it could be blended with light spirit to give a product which would almost certainly have a satisfactory Octane Number. A blend of equal parts of naphtha and light spirits would have a convenient distillation range.

# TABLE X

| Fraction | Temp. at 28 mm. °C | Temp. at 760 mm. °C | % Dist. | Total % Dist. | S. G. | Viscosity in C. stokes at | | Viscosity Index |
|---|---|---|---|---|---|---|---|---|
| | | | | | | 100 F | 200 F | |
| Charge | - | - | - | - | 0.976 | - | - | - |
| 1. | 148 | 253 | 10.1 | 10.1 | 0.867 | - | - | - |
| 2. | 166 | 279 | 9.9 | 20.0 | 0.883 | - | - | - |
| 3. | 198 | 315 | 9.6 | 29.6 | 0.898 | - | - | - |
| 4. | 218 | 343 | 9.3 | 38.9 | 0.915 | 6.48 | 2.38 | 286 |
| 5. | 252 | 377 | 9.5 | 48.4 | 0.932 | 11.2 | 2.76 | 93.1 |
| 6. | 280 | 413 | 9.4 | 57.8 | 0.955 | 23.5 | 4.32 | 68.4 |
| 7. | 318 | 454 | 9.6 | 67.4 | 0.978 | 51.4 | 6.56 | 47.1 |
| Residue | - | - | 32.6 | - | - | - | - | - |

To face page 81

# BREAK-UP OF TOPPED TAR.

In order to break up the residue into fractions suitable for examination for diesel and lubricating oils, the residue after topping to 190°C was distilled under reduced pressure. It was not possible to do this in bulk, as only a small tar still was available for this purpose, the maximum charge being about 6 litres.

The obtain a true boiling point curve, fractions of about 10% quantities were taken and collected in weighed flasks. The specific gravity of each fraction was determined, and thus an accurate estimate of the volume distilled in each fraction was obtained. This is given in Table X which also gives figures for the viscosity and viscosity index of the more viscous fractions.

These results are shown graphically in Fig. 7, which represents the true boiling point distillation curve, the gravity mid percent curve and the viscosity mid percent curves at 100°and 200°F.

## TABLE XI

| | I<br>Dist. from * Naphthenic Aromatic Crude. | II<br>Scotch * Shale Gas Oil. | III<br>Dist. from Cannel Tar. | IV<br>Oil III 2% Ethyl Nitrate. |
|---|---|---|---|---|
| S.G. at 60°F | 0.904 | 0.833 | 0.896 | 0.986 |
| Flash Point (P.M.) | 206°F | 182°F | 212°F | |
| Cold Test°F | 20°F | 12°F | below 0°F | below 0°F |
| | 4.12 cs. | 2.95 cs. | 3.46 cs. | |
| Hard Asphalt | Trace | Nil | Nil | Nil |
| Spontaneous Ignition Temp. | 272°C | 268°C | 431°C | 285°C |
| Initial Boiling Point | 236°C | 205°C | 208°C | 208°C |
| Vol. to 200°C | - | - | - | - |
| 225° | - | 3% | 1.5% | |
| 250° | 18% | 20.5% | 19.0% | |
| 275° | 51% | 53 % | 47.0% | |
| 300° | 75.5% | 77 % | 69.0% | |
| 325° | 87% | 90 % | 86.0% | |
| 350° | 95% | 96.5% | 96.0% | |
| Residue | 5% | 3.5% | 4.0% | |

* Nash and Howe's Principles of Motor Fuel Preparation and Application, Vol.2, p.449.

To face page 82

## FRACTION TRUE BOILING POINT 250° - 370°C.

It was found that the oil distilling up to 370°C on the True Boiling Point curve had a suitable distillation range for a diesel oil, as is seen in the table opposite.

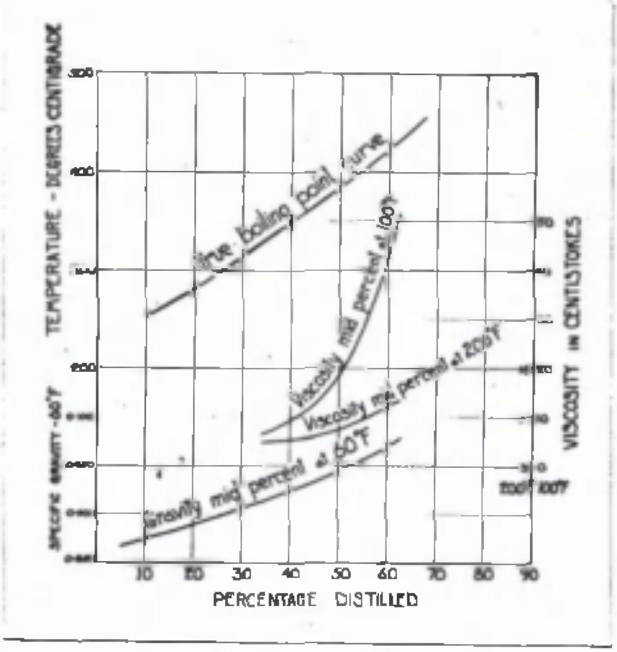


Fig. 7.

True Boiling Point data for Topped Dysart Cannel Tar.

This oil represents 50% of the residue after topping or 39% of the original crude. The yield per ton is thus 20 gals/ton.

The cannel diesel is very similar to the Gas Oil from Scotch Shale, except for the high Spontaneous Ignition Temperature. Accordingly tests were carried out with various dopes, only one of which, ethyl nitrate, reduced the ignition temperature appreciably.

### TABLE XII

Effect of Dopes on Ignition Temperature.

| | S.I.T. | Delay. |
|---|---|---|
| Oil alone | 420°C | 2 secs. |
| Oil 2% ethyl nitrate | 285° | 1 sec. |
| Oil 2% nitrobenzene | 406° | 2 secs. |
| Oil 2% amyl nitrate | 412° | 2 secs. |
| Oil extracted with caustic soda | 395° | 2 secs. |

Other properties examined were:-

Cetene Number.

This was determined for us, and was given as 40. This is very low for the modern high speed diesel engine (Av. 70), and indicates that the oil requires fairly thorough treatment before it could become marketable.

Aniline Point.

(I.P.T. method - F.O.23) 38.8°C.
(see page 88 )

## FRACTION TRUE BOILING POINT 370° - 454°C.

### Tests on Unrefined Oil.

The oils distilling after the diesel fraction were more viscous and contained much wax. These fractions were examined particularly as regards viscosity and stability to oxidation.

The viscosities of the oils compared favourably with light commercial lubricating oils, but their stability towards oxidation was much less satisfactory. This can be seen in Table XIII, which also gives the properties of the oils obtained by Jamieson and King (Inst. Gds. Eng. Communication No. 135, 1936) by high temperature carbonisation.

Table XIII

| | Commercial Lub. Oils | | Cannel Oils | | High Temperature Cannel Oils. | |
|---|---|---|---|---|---|---|
| | 1 | 2 | 3 | 4 | 5 | 6 |
| Gravity | | | | | | |
| Visc. at 100°F | 100.2 | - | 89.6 | - | 35.05 | - |
| " 200° | 11.5 | 10.3 | 7.34 | 7.98 | - | 12.22 |
| Viscosity Index | 83.4 | 46.3 | 73.9 | 58.2 | 104.1 | 343.9 |
| Volatility | - | - | - | 7.5% | 36.7% | 4.27% |
| Asphalt after Oxidation | Negligible | | 17.25% | 21% | 14.42% | 18.52% |

The oils included in the Table are:-

Column 1, a Russian Pale Oil.
2, a Californian Red Oil.
3, fraction 200° - 250°C at 16 mm. dewaxed but not refined.
4, fraction over 250° at 16 mm. dewaxed but not refined.
5, fraction 200° - 250° at 14.2 mm. dewaxed but not refined. (Jamieson and King - above)
6, fraction 250° - 300° at 14.2 mm. dewaxed but not refined. (Ibid)

It is evident that both types of cannel oil, although unrefined, show very low resistance to oxidation, and low viscosity indices. This means that thorough refining with its consequent losses is necessary.

Preliminary Refining.

The effect of refining on the properties of the Dysart cannel oil is shown in Table XIV. Two refining processes are shown, in both cases applied to the oil without dewaxing. These processes are:-

(a) Normal refining with concentrated sulphuric acid and alkali.

(b) Solvent extraction with furfural. The oils were extracted once only with an equal volume of furfural.

## TABLE XIV

### Fractions of True Boiling Point.

|  | 270° - 330°C. |  |  | 330° - 380°C. |  |  | 380° - 395°C. |  |  |
|---|---|---|---|---|---|---|---|---|---|
|  | 1 | 2 | 3 | 1 | 2 | 3 | 1 | 2 | 3 |
| Volatility at 140° for 24 hrs. | 13.7 | 7.9 | 7.7 | 8.1 | 6.8 | 4.9 | 6.5 | 7.2 | 15.5 * |
| Asphalt after Oxidation | 9.5 | 1.22 | 0.3 | 14.7 | 5.9 | 8.4 | 19.0 | 3.4 | 9.0 |

* High figure probably due to inefficient separation of furfural.

To face page 86

Column 1, unrefined oil.

2, oil refined with sulphuric acid.

3, oil extracted with furfural.

The refining shows that furfural is a suitable refining agent for these oils. Owing to the small quantities used for the experiments, the refining losses are much higher than they would be on a large scale. A continuous extraction process would give a better product than the single extraction.

The refining losses are high in both cases, of the order of 40%. In the case of the solvent extraction, of course, the extract could be worked up to give a low grade oil, or could be cracked. The furfural would be recovered, and in practice solvent losses are small.

After these preliminary tests it was decided to refine a large sample which Mr. Hunter of Birmingham University had kindly offered to examine.

## Test on Refined Oil.

### Dewaxing.

As it was intended to remove as little wax as possible, the dewaxing was carried out at 0°C. The cooled oil was enclosed in canvas and placed in a small hand press. The wax cake remaining was fairly hard, and amounted to 34.5% of the oil.

The dewaxed oil had a pour point of 35°F.

Refining.

The oil was extracted with four batches of furfural 50% by volume. The furfural was recovered from the raffinate layer, which was then distilled under vacuum, giving the refined lubricating oil.

It is difficult to obtain from the refining a reliable estimate of the losses on dewaxing and refining, as on the small scale the losses, particularly during dewaxing, are very heavy.

The refined oil so obtained was tested at the Department of Oil Engineering, Birmingham University. The results of the tests applied there are given in the form of reports on the suitability of the oil for use as (a) a diesel oil, and (b) a spindle oil.

The following is a summary of reports submitted by Mr. T. G. Hunter of the Department of Oil Engineering and Refining, Birmingham University.

The oil was a sample dewaxed and refined by solvent extraction with furfural, as described on pp. 85 It is referred to below as "Oil 8c/Lub."

# ABSTRACT OF REPORTS

# on

# OIL SAMPLE FROM ROYAL TECHNICAL COLLEGE.

## Boiling Range.

True boiling range of the oil was given as 360°C. to 450°C. These figures indicate that the oil could be classed as a heavy diesel oil or a light spindle oil.

## Viscosity.

| | |
|---|---|
| Viscosity at 100°F. | 13.3 centistokes.<br>62 Redwood secs. |

On this figure the oil could again be classed as a heavy diesel oil or spindle oil. The B.E.S.A. 'A' No. 1 diesel oil specification, for example specifies a maximum viscosity of 75 Redwood secs. at 100°F. while light spindle oils for ring spinning frames for cotton, worsted, silk or rayon vary in viscosity from 52 to 93 Redwood secs. at 100°F.

Owing to the fact that this oil might be considered as either a diesel or spindle oil, it was decided to test the oil first as a diesel oil and secondly as a spindle oil.

## SUITABILITY AS A DIESEL FUEL.

## Ignition Quality.

As the sample was not large enough to enable an engine test to be carried out, the Ignition Quality was estimated by measuring various properties which have shown satisfactory correlation with engine performance. Of these, the most reliable are Aniline Point and Diesel Index.

## Aniline Point.

The ignition quality of a diesel oil increases with increasing aniline point.

| | |
|---|---|
| Range for normal fuels, | 30°C - 85°C. |
| Sample tested, | 64.5°C. |

## Diesel Index.

$$\text{Diesel Index} = \frac{(\text{Aniline Pt. in }^{\circ}\text{F}) \times (\text{Gravity }^{\circ}\text{A.P.T.})}{100}$$

The ignition quality increases with increasing diesel index.

Range for normal fuels, 30 - 70. Sample tested , 40.

## Specific Gravity at $60^{\circ}$F. 0.893.

## Viscosity Gravity Constant.

Ignition quality increases with decreasing V.G.C.

Range for normal fuels, 0.9 - 0.805.
Sample tested, 0.860.

## Spontaneous Ignition Temperature (Moore Bomb).

High quality market diesel oils show a S.I.T. of $245^{\circ}$ - $260^{\circ}$C. The S.I.T. of the sample is $270^{\circ}$C, and suggests it to be an oil of fair quality.

## Carbon Residue.

Conradson Carbon Residue, 0.04%.

## Conclusions.

Technical Committee C on Fuels Oils of Committee D-2 on petroleum products and lubricants of the American Society of Testing Materials have tentatively classified diesel oils into 5 grades. Specifications of the first three grades, together with the specification of "Oil 8c/Lub." are shown below.

| Oil | Viscosity CS. at 100 F. Maxm. | Carbon Residue % Maxm. | Ignition Diesel Index Minm. | Quality V.G.C. Maxm. |
|---|---|---|---|---|
| Grade 1 - D | 7.5 | 0.2 | 45 | 0.86 |
| Grade 3 - D | 13 | 0.5 | 30 | 0.89 |
| Grade 4 - D | 55 | 3.0 | 20 | 0.91 |
| "Oil 8c/Lub." | 13.3 | 0.04 | 40 | 0.86 |

It will be seen from this table that the oil tested can be classed as Grade 3 - D oil for use in diesel engines for which this grade is suitable.

The above A.S.T.M. classification specifies also a minimum flash point, maximum water and sediment content, and maximum pour point. The sample was not examined for these properties because they are tests of suitability rather than quality, and any oil of the requisite ignition quality can be treated by ordinary refining methods to bring it within the limits required. Testing emphasis has been laid on ignition characteristics in which most oils produced from coal are deficient. The oil tested shows definite promise from the point of view of its utilisation as a diesel fuel.

## SUITABILITY AS A SPINDLE OIL.

Spindle oil specifications normally embrace specific gravity, open flash point, pour point, and viscosity. These properties of the sample are given in the table below, together with similar properties of average spindle oils.

| Property | Oil 8C/Lub. | Spindle Oil for ring spinning frames | Spindle oil for ring doubling frames | Shell Spindle Oil |
|---|---|---|---|---|
| Specific Gravity | 0.893 | 0.865 | 0.885 | - |
| Flash Pt. open °F. | * | 320 | 330 | - |
| Pour Pt. °F | 60 | 15 | 15 | 15 |
| Kinematic Viscosity at 70°F. in C.S. | 26.00 | 17.2 | 43.0 | 39.98 |
| 100°F. | 13.28 | 10.0 | 19.35 | 18.44 |
| 140°F. | 6.85 | 5.6 | 9.3 | 8.60 |
| 200°F. | 3.42 | - | - | 4.04 |
| Viscosity Index | 113 | - | - | 100 |

* Insufficient oil available for tests.

The above data show that the sample is satisfactory from the point of view of viscosity, while its viscosity index is excellent. The pour point of the sample is very high and renders it unsuitable as a spindle oil. This is not serious, however, as the pour point could probably be brought down to the usual figure of 15°F. by further dewaxing.

## Colour.

Spindle oils should be pale in colour and generally clean. The sample oil was unsatisfactory in this respect, being dark green. The colour could probably be made satisfactory, however, by treatment with decolourising clay.

## Oxidation Stability.

A good spindle oil must show an entire absence of tendency towards thickening or gumming under conditions of use. The oxidation stability of the sample was compared with that of Shell spindle oil by the following accelerated oxidation test.

60 ccs. of oil maintained at 200°C. was blown with air at the rate of 15 litres/hour for twelve hours. The oils were then examined.

| Test | Oil 8c/Lub. | Shell Spindle Oil |
|---|---|---|
| Appearance | Very black, a large amount of black solid sludge deposited. | Black, no sludge deposits. |
| Percent sludge | 32.5% | Nil. |
| Kinematic viscosity at 100°F. after removal of sludge, C.S. | 18.33 | 22.37 |
| Ratio $\frac{\text{Viscosity after oxidation}}{\text{Viscosity before oxidation}}$ | 1.38 | 1.21 |

The sample oil has a very poor resistance to oxidation, and sludge deposits are excessive. The sample is quite unsuitable from this point of view. The results from this test must inevitably condemn the sample from the point of view of its utilisation as a lubricating oil of any description, where oxidation stability is essential

## Refractivity.

The Manchester Committee on Cancer have specified standards on the basis of refractivity for non-carcenogenic spindle oils, as follows:- Oil of Sp. Gr. above .895 should have a refractivity below .5520; with a Sp. Gr. below .895, refractivity should be below .555 .

Sp. Gr. of sample was .893, and refractivity was .558.

These specifications are of very doubtful value indeed. Nevertheless since the oil fails to come within the limits specified, and in addition is derived from the distillation of coal, it would be looked upon with suspicion as being probably carcenogenic.

In my opinion this oil is quite unsuitable to use as a spindle oil, and it is doubtful if it would be made suitable by further refining treatment.

It will be seen that, so far as its use as a lubricating oil is concerned, the great instability towards oxidation of this oil at once ruled it out. It is almost certain that by proper fractionation a heavier oil could be obtained, but this would probably be less resistant to oxidation. It can be seen from Table XIV that the oxidation resistence decreases as the higher boiling oils are reached. It may be that suitable treatment during refining might increase the stability, but time did not permit an investigation, of this.

## Effect of Paraflow.

Removal of the wax to lower the Pour Point of the oil means a considerable loss in yield (40%). The wax itself is not injurious to the lubricating properties of the oil, so that if the pour point could be depressed artificially without removal of the wax, this loss in yield would be avoided: indeed, it is claimed (Davis and Blackwood. Ind. Eng. Chem. 23, 1452) that the presence of wax improves the viscosity characteristics of oil above the Pour Point. The effect of Paraflow as a pour point depressor was therefore investigated.

After dewaxing at 0°C, the pour point of a sample of lubricating oil fraction was 35°F. The addition of Paraflow in 1%, 3%, and 5% quantities lowered the pour point to 30°F, 30°F, and 25°F respectively. It means then, that this method is neither economical nor effective in lowering the Pour Point.

## CONCLUSIONS.

The cannel oil described in this chapter, gave on distillation to 190°C, a light spirit, which on refining had the following properties:-

| | |
|---|---|
| Yield | 12 gals/ton |
| Specific Gravity | 0.830 |

### Distillation Range

| | Total Percent Dist. | Percent Fraction |
|---|---|---|
| Initial Boiling Point | 60°C | |
| Up to 100°C | 1% | 1% |
| 125°C | 26% | 25% |
| 150°C | 59% | 34% |
| 175°C | 89% | 55% |
| End Point | 189°C | |
| Loss | 1% | |

### Octane Number

| | |
|---|---|
| Spirit | 58 |
| Blend A | 68.5 |
| Blend B | 66 |
| Blend C | 74.5 |
| Percentage Unsaturateds | 11% |
| Percentage Aromatics | 43% |

Although the Octane rating is low, this may be accounted for partly by the absence of light fractions, as the distillation range shows that blending is necessary.

The spirit scrubbed from the gas was not available in sufficient quantity for refining and engine tests, but its distillation range shows it to be very light and suitable for blending with a heavier spirit, such as the spirit topped from the tar. Thus the combination of scrubbed spirit and tar spirit should give a satisfactory motor fuel, particularly as the spirit scrubbed from the gas by Jamieson and King (quoted on Table IX ) showed excellent anti-knock qualities, having an Octane Number of 68.

Diesel Oil.

A 48% cut from the topped crude, which was tested as a diesel oil, had properties very similar to those of a Scottish Shale diesel oil, as can be seen from Table XI. Although the Spontaneous Ignition Temperature is high, it must be remembered that the cannel oil tested was quite untreated, while the shale diesel oil is normally refined. It is almost certain, therefore that a light refining treatment would give a product of satisfactory ignition quality.

This fraction corresponds to a yield of 20 gals/ton.

A heavier fraction, originally intended as a light lubricating oil, was tested, after refining, as a diesel oil at Birmingham University. It was shown to be comparable to modern high speed diesel oils in every way. While a fairly thorough refining is necessary before this oil meets these specifications, it could be added to that previously described, beinging the total yield of diesel oil up to 28 gallons per ton.

Lubricating Oil.

A heavy fraction was dewaxed and refined with furfural. Tests carried out at Birmingham University showed:-

(a) it corresponded in viscosity to a spindle oil,

(b) it would be unsuitable for use as a lubricant where stability to oxidation is essential,

(c) if this instability could be overcome, it would be a satisfactory lubricant, as its viscosity characteristics are good.

Before concluding that it is not possible to isolate a lubricating oil from this tar, it would be advisable, in view of the modern advances in solvent refining, to investigate the effect of these processes on the heavier fractions of the tar. To do this, a fair quantity of a representative tar would be required. Unfortunately, to obtain this is a matter of some difficulty.

The possibilities of improving the oxidation resistance by hydrogenation of the heavy fractions could also be considered.

# CHAPTER 4

## VAPOUR-PHASE CRACKING OF CANNEL TARS.

### I - APPARATUS.

As a considerable part of the normal processing of petroleum products resolves itself into an endeavour to obtain the maximum yield of motor spirit, it was felt that an examination of the cracking properties of cannel tar was a desirable extension of the work described in the preceding chapters. Hydrogenation-cracking of the Dysart Cannel Tar described in Chapter 3 was carried out at the Fuel Research Station, and this work is described in a Report presented by the Director of Fuel Research to the Oil from Coal Committee. This report, which is quoted in full in the previous chapter, concludes:

> "This tar is therefore an excellent raw material for hydrogenation-cracking in the vapourphase."

The process described, while giving good yields of motor spirit and diesel oil, is a relatively expensive one. How far, then, could normal cracking processes increase the yield of motor spirit?

As the plant for normal pressure cracking was not available, it was decided to attempt cracking in

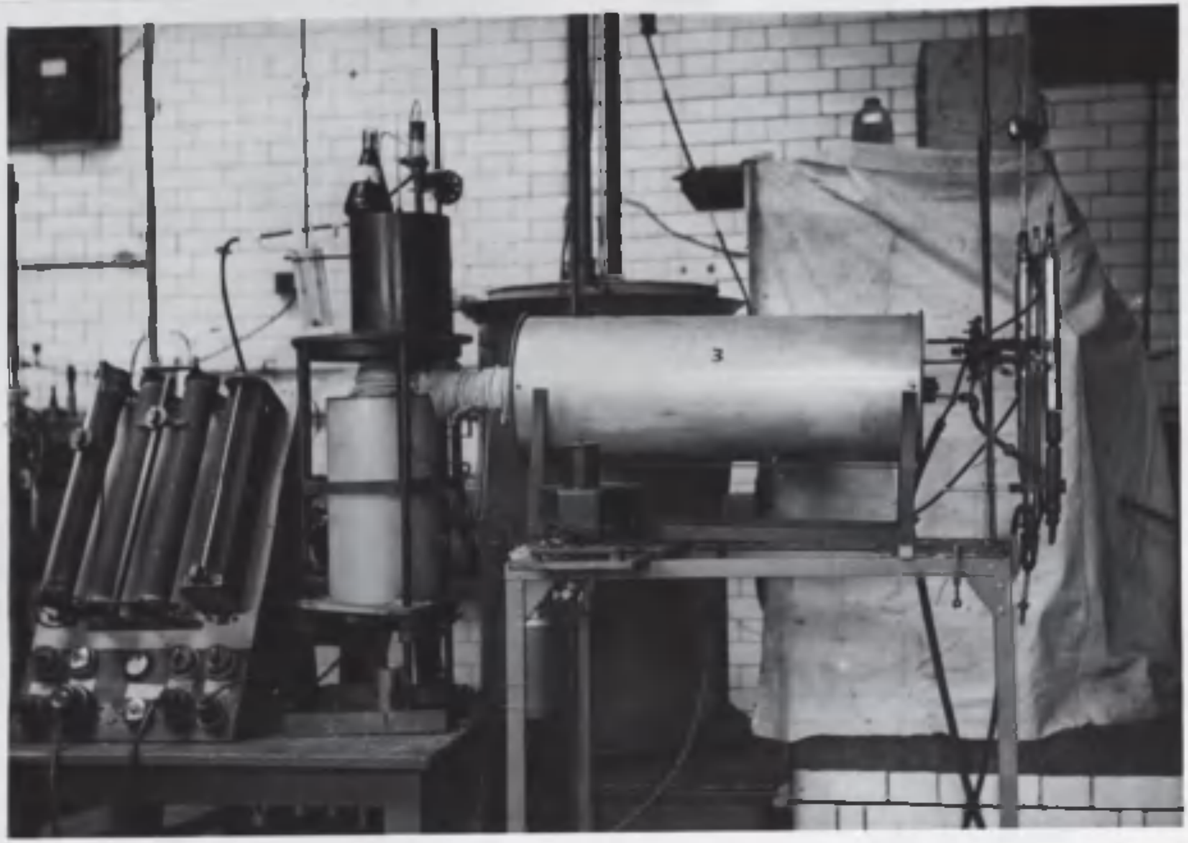


Fig. 8. Vapour Phase Cracking Unit.

To face page 98

the vapour phase at pressure not far removed from atmospheric. This would enable a simple type of apparatus to be used. Although this method of cracking is open to question on the grounds that the low pressure vapour phase conditions favour gas formation, it was felt that it conformed well enough to modern practice as exemplified in such a process as the True Vapour Phase.

The apparatus used is shown in Fig. 8, and is illustrated diagramatically in Fig. 9.

## General Description.

The cracking unit used in the tests consisted essentially of:

(a) a device for vaporising the tar feed,
(b) the cracking tube or tubes,
(c) a condensing system,
(d) a holder for cracked gas.

and the complete layout is shown diagramatically in Fig. 9

The tar is fed by means of the small reciprocating feed pump A, into a heated coil B where its temperature is raised to about 300°C and it is partially vaporised. Along with the feed a current of cracked gas from the holder enters. On theoretical grounds, the presence of this gas should tend to inhibit gas formation. The function of the

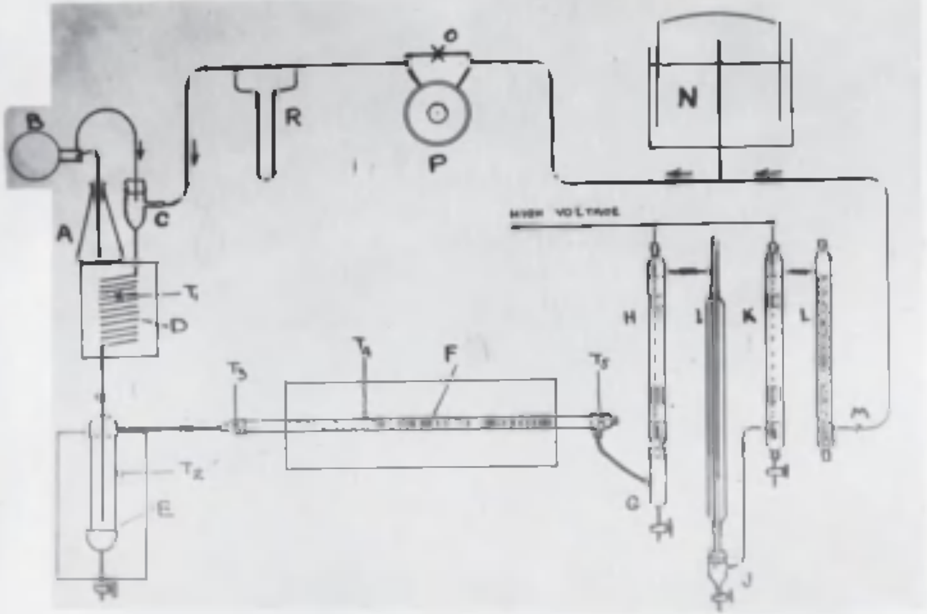


Fig. 9. Layout of Cracking Unit.

To face page 99

gas stream, however, was to sweep the tar vapours through the apparatus and to assist vaporisation. Control of the rate of gas flow is obviously an easy means of controlling the time taken by the vapour to pass through the cracking furnace.

The partially vaporised feed then flows on to a vaporising chamber at 400°C, where the unvaporised tar is run off as pitch, the tar vapours passing into the cracking tube.

The cracking furnace used was an electric one 30" long. In this furnace two types of cracking tube were tried:-

(a) a straight piece of 1" I.D. gas pipe, packed with porcelain chips.

(b) a series of stainless steel tubes arranged to give three complete passes through the furnace.

Originally, it was intended to use a straight steel tube, but it was felt that coking difficulties might be experienced with a narrow tube, whereas a wider tube would not give sufficient turbulence to ensure uniform temperature through the tube. Packing a wide tube with an inert material seemed a possible method of creating turbulence without coking. In practice the porcelain packing proved to have some remarkable properties. (see later p. 103 )

The vaporised tar, having passed through the cracking tube, cools on emerging from the heated

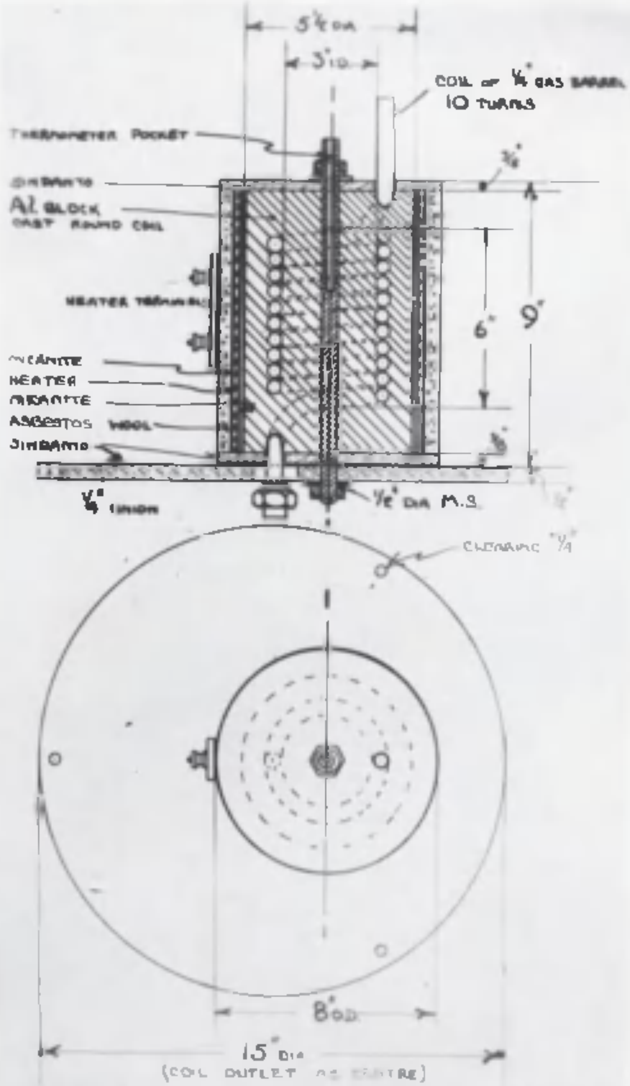


Fig. 10.

Detail of Preheating Coil.

To face page 100

zone giving a tar mist.

The tar mist was dealt with effectively by means of a condensing system consisting of:-

(a) a water cooled condenser,

(b) an electrostatic precipitator,

(c) a second condenser.

The cracked product was run off from these periodically.

The gas passing through the condensing system could be scrubbed if desired in an active carbon scrubber and then passed to a holder. From the holder, gas was taken off and fed by a pump into the apparatus at the point where the feed entered.

Components.

Details of the components are shown in the diagrams.

## (1) Preheater.

The preheating coil is shown in Fig. 10. It consists of a coil of seven turns of ¼" gas barrel. Round the coil is cast an aluminium block, which is heated by an electric heater wound on the outside. This gives uniform heating of the coil, preventing especially local superheating which would, particularly with cannel tars, cause difficulty with coking. The heating block is bolted to its "syndanyo" baseplate by a central bolt which is drilled for 5

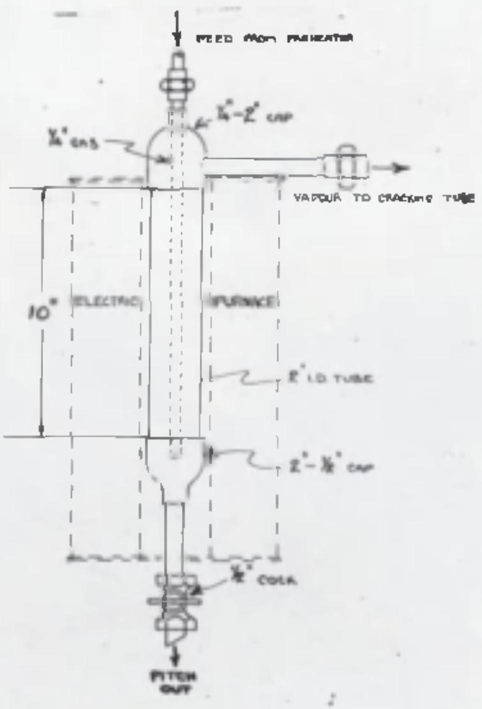


Fig. 11.

Details of Vaporiser.

To face page 101

inches of its length to form a thermometer pocket.

The tar is forced in at the top end of the coil and flows down to the vaporiser, aided by the current of gas. Instead of feeding the tar directly into the metal tube the feed pipe enters a wide mouthed glass tube which is attached to the end of the coil. Thus any stoppage of the feed can be seen at once. The gas enters through a side arm in the glass tube.

This method of preheating the tar proved quite satisfactory, but of course suffered from the disadvantage of all coils - the difficulty of clearing any blockage. To minimise this, the feed tar was all filtered through wire gauze.

### (2) Vaporiser.

The lower end of the preheating coil was connected by means of a union to the central tube of the vaporiser. The latter is shown in Fig. 11. It is built up from 2" mild steel pipe and standard fittings.

In the vaporiser the tar feed is separated into:-

(a) The vapour feed, which passes into the cracking tube, and

(b) Pitch, which is left unvaporised, and is run off by the cock shown in the diagram.

It follows that the Vapour Feed (a) is the true cracking stock. When cracking an oil, which has already been distilled, such as a gas oil, the amount unvaporised is of course small, and the vapour feed then approximated to the charging stock. In the case of the crude topped tars used, about 15 - 20% of the tar was run off as pitch, the remainder passing through the cracking tube. It is simpler to separate the pitch during vaporisation than by a preliminary distillation.

### (3) Cracking Tubes.

It is necessary in any vapour phase cracking, to ensure that only vapour is passed into the cracking tube. Particles of liquid, if allowed to enter, are liable to coke and cause choking. This is well exemplified in the True Vapour Phase process, in which a hot inert gas is added to the vaporised feed. This ensures complete vaporisation before the mixture enters the cracking zone, and thus reduces coking.

Although provision was made - in the form of a side arm on the reducing cap (Fig. 12) - for the addition of hot gases to the tar vapour before it entered the cracking tube, this has not so far been tried on the unit being described. The connection between the vaporiser and the cracking tube, however, was well lagged with asbestos to prevent any condensation between the vaporiser and the furnace.

(a) Straight Packed Tube.

The greater part of the cracking was carried out in this tube. It is shown in Fig. 12, and was packed with pieces of broken porcelain evaporating basin about ⅛" to ¼" in size. This packing proved very successful and the tube showed no tendency to choke under normal working conditions.

This freedom from coke seemed to be due largely to the packing which on use became covered with a thin film of carbon. Deposition then seemed to stop, and continued use did not alter the film appreciably. This property of porcelain packing has also been noted by Tropsch and others (Ind. Eng. Chem. 1938. 169).

That freedom from coking was definitely a property of the porcelain was shown in the early running of the plant. On the first occasion, the tube was filled with the porcelain chips, and to keep them in position a plug of asbestos was inserted at each end of the column of packing. When cracking was attempted, the tube choked almost immediately and on dismantling, it was seen that both asbestos plugs were solid with coke, while the porcelain had only the thin film of

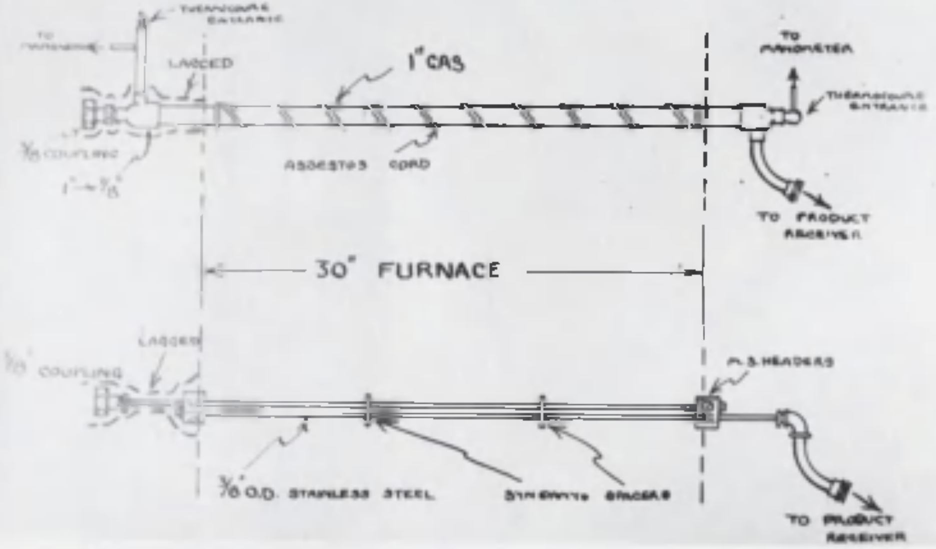


Fig. 12. Cracking Tubes.

To face page 104

carbon deposit. In a second attempt to keep the porcelain packing in position, a piece of monel gauze was inserted at each end. On trial, the gauzes coked up almost at once. Thereafter the packing was placed in the tube alone, and after being pushed into position was disturbed as little as possible during assembly. Packed in this way, the tube gave no trouble.

The tube itself was a 3 feet length of 1" I.D. gas pipe, passing through a 2" furnace tube. At each end there was a 1" to ⅜" reducing cap with a ¼" branch. At the inlet end, the straight connection was coupled to the vaporiser offtake pipe by means of a ⅜" union. Into the branch was screwed a short piece of ¼" pipe which served as thermocouple entrance and also carried a small manometer. At the exit end the branch was pointed down, and led via a ½" copper tube to a union which in turn was connected to the main product receiver. The straight connection served as thermocouple entrance and also led to a manometer.

The tube was wound on the outside with 15 - 20 widely spaced turns of asbestos cord, to prevent possible sagging and sticking to the silica furnace tube. In use, the tube showed some scale but it was never serious.

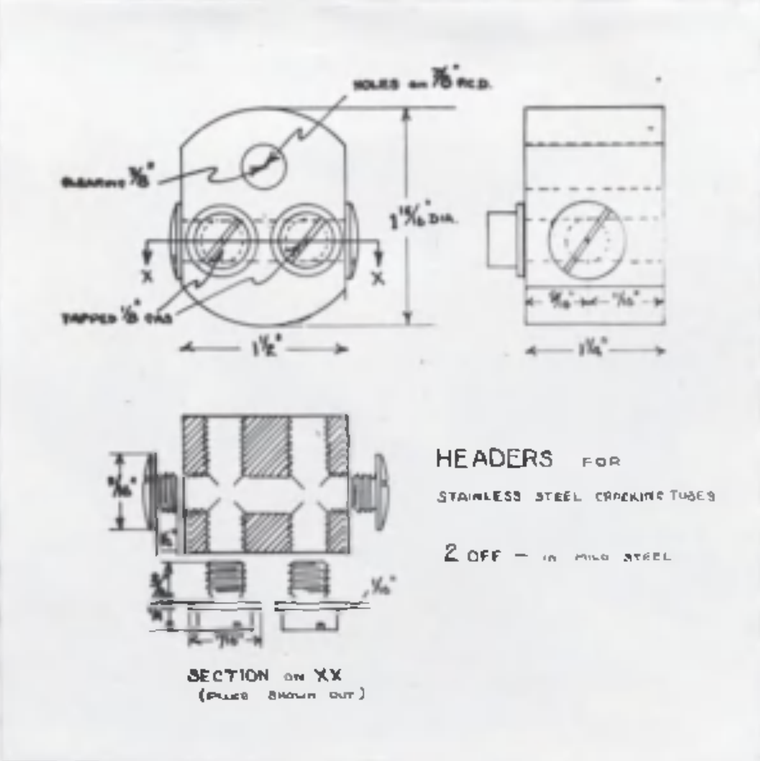


Fig. 13.

Details of Headers.

To face page 105

### (b) Stainless Steel Tubes.

The layout of these tubes is shown in Fig. 12. The three tubes are connected by two headers as shown, (Fig. 13) and give three passes through the furnace. It will be seen that the vapour passes in a series of straight paths, all of which can be cleaned out. Two "syndanyo" spacers along the tubes guard against possible sagging in the furnace when hot.

### (4) Condensing System.

The products of cracking pass from the cracking tube into the main product receiver G. The heavier material condenses here, and in the condenser leading from it, while the tar mist passes into the precipitator H, where the mist is dispersed. The gases and any uncondensed vapours then pass through a second condenser, the condensate from the precipitator and the second condenser being collected in a common receiver. In most cases the gas then passed to the holder, but in the experiments described in Chapter 6, an active carbon scrubber was included. In the latter case, also, two precipitators were used, with a condenser between them, the tar mist being precipitated hot. This modification is discussed in the next chapter, and is that shown in Fig. 9.

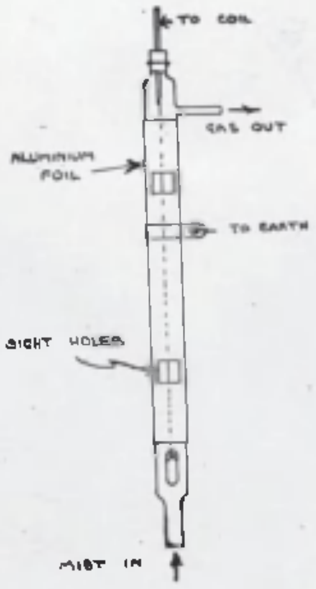


Fig. 14.

Electrostatic Precipitator.

To face page 106

## Condenser.

The condenser was of the normal Liebig type, the vapour from the product receiver passing upwards, the condensate draining into the receiver. A side arm at the top led to the precipitator.

## Electrostatic Precipitator.

Once the proper working conditions had been found, these proved very effective. Methods of mist dispersal involving baffles of any kind, e.g. glass wool, suffer from the disadvantage that the trapped material can not be easily collected, and represent undesirable hold-up. In a continuous plant, where samples are being run off at intervals, this is a very big disadvantage, as it is very difficult to obtain a representative sample over a given time. No information was available as to the action of such small precipitators, so that the design was purely experimental. The simple design adopted, however, proved satisfactory, and was undoubtedly successful in disposing of the tar mist.

The precipitators consisted of a glass Liebig condenser outer jacket, coated on the outside with Aluminium foil cemented on with shellac varnish, as is shown in Fig. 14. This foil was earthed via the metal stand. The central electrode enters at the top through a rubber bung, which gives satisfactory insulation if kept clean. This electrode was connected to the H.T. side of a 6V ignition coil of a type formerly used on motor cars. This proved more satisfactory than the normal type of laboratory coil, being of very robust construction well suited to continuous running, and giving a short, heavy spark. The other H.T. terminal was earthed.

The central electrode consisted of a single strand of 36 S.W.G. wire, hanging from a short length of 12 gauge brass wire. A small lead weight served to keep the central wire taut and vertical. So long as the coil gave a spark greater than about $\frac{1}{4}$" in length, precipitation was very satisfactory, the gas coming from the precipitator quite free from haze.

Initial attempts to run the precipitator showed the importance of the central electrode. This was at first made of a length of brass wire about $\frac{1}{8}$" in diameter. With this electrode, no precipitation could be obtained. When this thick wire was replaced by the 36 S.W.G. wire, precipitation was complete in the first four inches or so of the precipitator.

Second Condenser.

This led from a small glass receiver, into which the precipitator drained. The gases from the bottom of the precipitator entered this receiver, and passed up the second condenser to the gasholder.

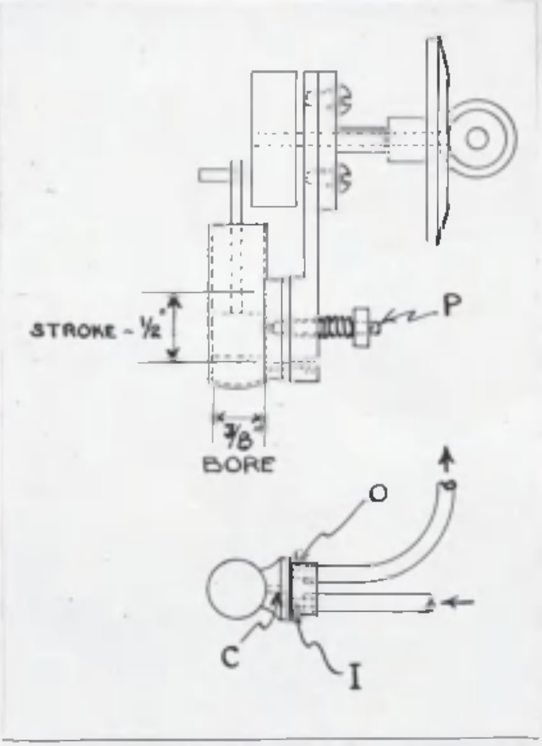
Scrubber.

In the later experiments the gas before entering the holder passed through an active carbon scrubber. This was similar to that described in Chapter 2 (page 24). It consisted of a Liebig condenser outer jacket filled with Benzorbon active carbon. It proved a desirable addition, preventing the lighter liquid hydrocarbons from entering the gasholder and being recycled, with consequent further cracking to gas.

## (5) Feed.

For these cracking experiments a feed rate of about 200 - 300 cc. per hour was desired. Initial attempts to run in the tar from a large separating funnel proved very unsatisfactory, as the rate of flow varied greatly, due to heating up, with consequent loss in viscosity, of the tar during a run. In addition, the rate of feed could not be set at a predetermined value.

The small reciprocating pump shown in Fig. 15a was therefore fitted. This pump was mounted as shown in Fig. 15b, and was driven by a belt from the gas circulating pump through reduction gearing. The inlet side is connected to a copper pipe which



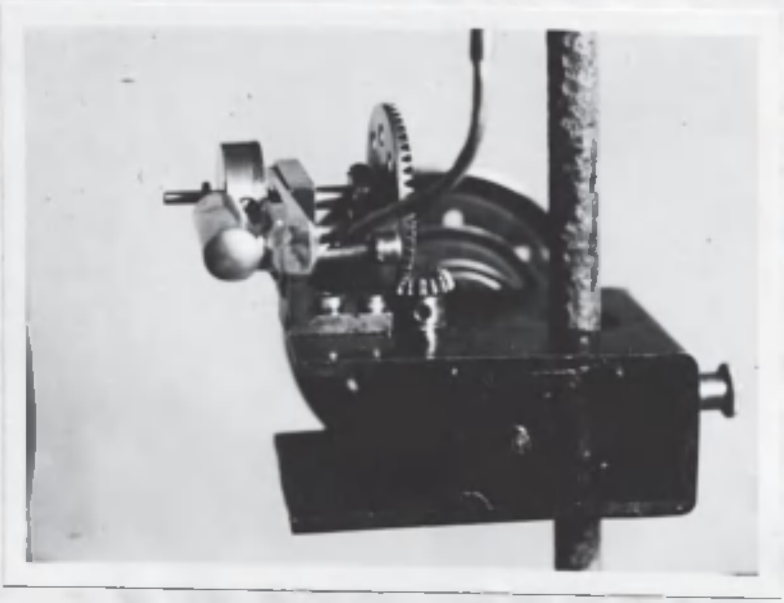
(a)

Fig. 15.

(a) Left – Detail of feed pump.

(b) Below – Photograph showing method of mounting.

(b)



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dips almost to the bottom of a flask containing the feed. Thus any small particles of solid remaining in the filtered tar settle to the bottom and do not enter the pump. During a run, also, this copper pipe can be raised slightly, the flask removed, and another weighed flask quickly substituted. In this way, the quantity of tar supplied during a given period can be determined accurately, and without interrupting the feed.

This pump, which is of a type common in model steam engines, is very suitable for use in feeding small quantities of a viscous liquid. The valve action is very simple and positive. The crank, besides reciprocating the piston, rocks the cylinder about the pin P. (Fig. 14) This rocking action brings the port C alternately opposite the inlet port I and the outlet port O, both of which are fixed. It is thus impossible for the valves to stick. If any of the ports become blocked, the cylinder and piston are detached by removing a nut on P, when all the parts in contact with tar are free for cleaning. This blockage can be easily avoided by filtering the tar before use through a Buchner funnel having a tightly fitting circle of 30 mesh wire gauze in place of filter paper. The pump and gearing are in one unit which can be mounted on a retort stand.

When adjusted to give a feed of about 300 cc. per hour, the pump speed is about five strokes per minute. This intermittent action is no apparent disadvantage, as the flow is largely evened out in the seven turns of the preheating coil.

### (6) Gas Circulation.

The current of gas was supplied by a pump. As the pump available was of much larger capacity than was necessary, a by-pass was fitted. By throttling the by-pass slightly, the small pressure required to force the gas through the system was easily obtained, and could be varied easily without varying the speed of the pump. The arrangement is shown in the diagram of the apparatus - Fig. 9. The gas from the pump P passed through a flowmeter R and entered the system at the point of entrance of the feed.

The rate of the gas flow was determined arbitrarily. In the first runs, the flow was varied, and the rate chosen which gave a positive sweeping action through the apparatus, but which remained within the capacity of the condensing system. Measurements of the rate then showed it to be about 0.03 cu.ft. per minute.

## III OPERATION.

The procedure followed during each run of the plant was substantially the same, the only variables being:-

(a) the temperature of cracking.

and (b) in certain cases, the type of cracking tube.

When the furnaces had reached the working temperatures the precipitators were started and the gas circulation begun. The feed pump was then started and tar fed in for about an hour, to allow cracking conditions to become steady. During this period, the product receivers were drained at intervals; the pitch, also, was run off from the vaporiser.

At a definite time, the product receivers were drained, pitch run off, and gas volume noted, all in as short a time as possible. The feed flask was then immediately changed for a full weighed one, and all product and pitch run off after these were collected in weighed vessels. When this feed flask became empty, the product and pitch were drained off, the gas volume noted. and the flask replaced with another. During a run, two or three such test periods were taken at the same cracking conditions.

Thus, in each test period, usually about one hour in duration, the following were obtained:-

(a) Weight of tar supplied.

(b) Weight of cracked product formed.

(c) Weight of pitch - i.e. tar unvaporised.

(d) Volume of gas formed.

After determining the density of the gas, it is then possible to add up the weights (b), (c), and (d), which should give weight (a) - the weight of the tar feed. This mass balance, which could not generally be achieved with accuracy, can be seen from the sample log given below.

At the end of the run, when the feed was stopped, the gas was circulated for half an hour or so, while the furnaces cooled somewhat. It was important to do this, in order to sweep out tar vapour, as any left in the cracking tube condensed on the packing, and on heating up for the next run it invariably coked, causing choking of the tube. This point again emphasises the desirability of avoiding liquid in the cracking tube at the temperatures used in vapour phase cracking.

# SAMPLE LOG. RUN 9.

FEED DYSART CANNEL TAR – TOPPED TO 198°C
PRESS IN HOLDER – 14 MM. WATER

RATES FEED – 200 CC/HR. APPROX.
BAROM. PRESSURE – 748 MM.

GAS – 0.03 CU.FT./MIN.
TEMPERATURE – 16.5°C

| TIME | TEMP. (°C) PREHEATER | TEMP. IN °C AT – | | | | GAUGES MM. WATER | | GAS HOLDER CU.FT. | FEED | | PRODUCT | | PITCH WT. | REMARKS |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | VAPOURISER | VAPOUR IN | VAPOUR OUT | FURNACE | IN | OUT | | No. | WT. | No. | WT. | | |
| | $T_1$ | $T_2$ | $T_3$ | $T_4$ | $T_5$ | $G_1$ | $G_2$ | | | | | | | |
| 11.30 | 346 | 385 | 180 | 370 | 800 | 4.0 | 1.5 | 4.693 | 1 | - | 1 | - | - | FEED STARTED |
| 12.00 | 302 | 400 | 180 | 382 | 800 | 4.0 | 3.0 | 3.530 | 1 | - | 1 | - | - | |
| 1.00 | 295 | 425 | 180 | 342 | 815 | 4.0 | 3.0 | 1.960 | 1 | - | 1 | - | - | |
| 2.00 | - | - | - | - | - | - | - | EMPTIED | | | | | | GASHOLDER FULL. GAS BURNED. |
| 2.15 | 303 | 425 | 180 | 398 | 830 | 5.0 | 4.0 | 4.48 | 1 | - | 1 | - | - | |
| 2.30 | 300 | 440 | 183 | 425 | 815 | 6.6 | 5.5 | 3.800 | 2 | 486.1 | 2 | 66.5 | 270.8 | TEST I STARTED |
| 2.45 | 299 | 440 | 190 | 425 | 800 | 7.0 | 5.0 | 2.936 | 2 | - | 2 | - | - | |
| 3.00 | 296 | 450 | 185 | 415 | 785 | 7.0 | 5.0 | 2.280 | 2 | - | 2 | - | - | |
| 3.15 | 296 | 450 | 185 | 425 | 780 | 7.0 | 6.0 | 1.585 | 2 | - | 2 | - | - | |
| 3.30 | - | - | - | - | - | - | - | 1.000 | 2 | 153.1 | 2 | 173.8 | 329.4 | FEED 2 REPLACED BY 1. |
| | | | | | | | | EMPTIED | | | | | | HOLDER EMPTIED. TEST I ENDS. |
| 3.46 | 297 | 440 | 183 | 420 | 770 | 7.0 | 6.0 | 4.700 | 3 | 448.2 | 3 | 46.3 | 329.4 | TEST II BEGINS. |
| 4.15 | 295 | 425 | 180 | 410 | 762 | 7.5 | 7.0 | | 3 | - | 3 | - | - | |
| 4.34 | 297 | 430 | 180 | 410 | 763 | 10.0 | 9.0 | 2.710 | 3 | - | 3 | - | - | |
| 4.50 | 299 | 430 | 180 | 410 | 765 | 10.0 | 9.0 | 2.040 | 3 | 159.4 | 3 | 175.7 | 391.4 | TEST II ENDS. FEED STOPPED. |
| | | | | | | | | | | | | | | FURNACES OFF. |
| 5.16 | - | - | - | - | - | - | - | - | - | - | - | - | - | GAS CIRCULATION STOPPED. |

# SUMMARY OF RESULTS - Run 9.

| | Test I | Test II |
|---|---|---|
| Duration of test | 60 mins. | 65 mins. |
| Feed | 273.0 gms. | 288.8 gms. |
| Product | 107.3 gms. | 129.4 gms. |
| Pitch | 58.6 gms. | 62.0 gms. |
| Gas | 2.800 cu.ft. | 2.660 cu.ft. |
| **From 100 gms. Feed.** | | |
| Product | 39.3% | 44.8% |
| Pitch | 21.5% | 21.5% |
| Gas. cu.ft. | 1.025 | 0.921 |
| Mean Temperature | 780°C | 750°C |
| **Mass Balance.** | | |
| Feed | 273.0 gms. | 288.8 gms. |
| Product | 107.3 gms. | 129.4 gms. |
| Pitch | 58.6 gms. | 62.0 gms. |
| Gas (at S.G.1.8, air=1.) | 93.5 gms. | 85.8 gms. |
| Total | 259.4 | 277.2 |

# CHAPTER 5

## VAPOUR PHASE CRACKING OF CANNEL TARS.

## II - CRACKING IN A STRAIGHT PACKED TUBE.

### CRACKING CONDITIONS.

#### Cracking Tube.

The tube used in the experiments described in this chapter was the straight packed tube shown in Fig. 12. Details of this tube and of the properties of the packing are given in the preceding chapter.

#### Temperature.

This was the only variable studied in this series of experiments, as the supply of suitable tar was rather limited. The temperature was varied from about 400° to 800°C, being recorded by a chromel-alumel thermocouple placed inside the silica furnace tube, in the annular space between it and the cracking tube. No attempt was made to determine the temperature of the gases in the cracking zone as, apart from the extra complication involved, it was felt that no useful purpose would be served, since such a temperature would never be used in practice for control purposes. In many runs, the temperatures of the vapours entering and leaving the cracking tube were noted. These temperatures were invariably very much lower than the temperature of the furnace. The vapour entering the tube was normally at 200°C, and as the temperature of the vaporiser was constant, this was true for all the runs of the plant. The exit vapours, at $T_5$ (Fig. 9), were normally only slightly higher in temperature than those of the entering vapours, and were always much below the temperature of the furnace.

As the contact between vapour and packing in the cracking tube would be fairly intimate, and as the packing should cause considerable turbulence, it must be assumed that the vapour passing through the tube reached a fairly high temperature, losing heat quickly as it emerged from the furnace. It is possible, of course, that under the conditions of short time of contact holding in the tube, the temperature of the cracking vapours would be much lower than that of the furnace. It is more probable, however, that the temperatures changes of the gases in the tube would be rapid, and, especially at the surface of the packing material, the temperature would be fairly close to that recorded outside the tube.

## Feed.

The feed material in these experiments was the Dysart Cannel Tar described in Chapter 3. The crude tar had been topped to 190°C, and the topped tar was used. It is important to distinguish between the crude tar feed, and the vaporised material which formed the actual cracking stock. The tar entering the vaporiser is separated into (a) pitch and (b) tar vapour. This vapour which passes through the cracking tube is different in distillation range from the topped tar supplied to the vaporiser, since the heavy fractions have been run off as pitch. The true composition of the vapour feed can be seen by extrapolating the curves in Fig. 18 towards the lower temperatures. It will be seen that the percentage composition of the product remains fairly constant until cracking starts (as shown by gas formation). Thus, assuming that the feed vapour is unaltered in its passage through the tube below the cracking temperatures, the composition arrived at by this method may be taken as that of the vapour feed.

These two sets of figures are given in Table XV.

## TABLE XV

Composition of Tar Feed and Vapour Feed.

| Fraction °C | Tar Feed | Vapour Feed | |
|---|---|---|---|
| | | from curves | condensed |
| 0 - 180° | nil | nil | 0.5% |
| 180° - 300° | 25% | 37.2% | 36.2% |
| 300° - 350° | 17% | 28.7% | 30.8% |
| over 350° | 58% | 34.1% | 32.5% |

An attempt was made to obtain a sample of the vapour feed by disconnecting the cracking tube and replacing it with a worm condenser. The condensate had the composition shown in column 3 of the table. When account is taken of the different method of condensing, the figures show good agreement.

### Gas Rate.

The current of gas passing through the apparatus was intended to sweep the tar vapours through the tube, and to assist vaporisation in the coil and vaporiser. The rate of flow was adjusted during the preliminary runs to give steady working conditions, and was then kept constant during the subsequent runs. The gas before entering along with the tar feed, passed through a flowmeter which was used to control the rate of flow.

The rate corresponding to the normal head across the flowmeter was determined by closing the inlet to the gasholder at M (Fig. 9), and running the apparatus with the connection from the condensing system open to the air. As the holder was normally kept at atmospheric pressure, this did not introduce any errors due to pressure differences in the system. The rate so determined was found to be 0.03 cu.ft. per minute.

## Cracking Time.

An estimate was made of the time the vapour remained in the cracking zone. The method used to arrive at this was as follows.

### (a) Free Space in Packing.

A graduated cylinder of approximately the same internal diameter as the cracking tube was used to determine the percentage free space in the packing. The cylinder was half filled with water and the exact volume noted. Packing material was then added, and the volume occupied by the packing was noted. The increase in the volume shown by the rise in water level gives a measure of the actual volume of the porcelain chips.

Then:- Percentage free space is given by -

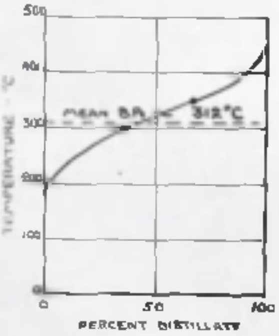

Fig. 16.

Approximate Distillation Curve of

Vapour Feed from Date in Table XV

(An end point of 420°C is assumed)

To face page 119

$$\frac{\text{Vol. occupied by packing - increase in water vol.}}{\text{Vol. occupied by packing}} \times 100$$

This was repeated several times and the mean value taken. The free space for the porcelain chips used was 50.0%

### (b) Molecular Weight of Vapour Feed.

From the data given in Table XV the approximate distillation curve shown in Fig. 16 can be drawn, the end point being taken as 420°. This curve shows the mean boiling point to be 312°C. From the molecular weight of pure hydrocarbons of about this boiling point (see "Science of Petroleum" - Dunstan, Nash, Brooks, and Tizard - Volume II) a figure of 200 was chosen as a reasonable value for the Mean Molecular weight.

### (c) Temperature of Vapour in the Cracking Tube.

Two figures were chosen for this, so that an estimate could be obtained of the limiting conditions. The following calculations were made for temperatures of 400° and 800°C: these temperatures were assumed to be below and above the actual temperatures of cracking.

### (d) Cracking Time.

Nominal bore of cracking tube 1 in.

Volume per foot length $= \frac{12\pi}{4}$ $= 9.43$ in.

At 50% free space, free volume $= 9.43 \times .5$ $= 4.72$ cu.in.

Free sectional area $= \frac{4.72}{12}$ $= 0.392$ sq.in.

Consider the circulating gas.

Rate at 760 mm and 15°C $= 0.03$ cu.ft./min. - as found

" " 760 mm " 400°C $= \frac{0.03 \times 673}{288}$

$= 0.0701$ cu.ft./min. at 400°C

Rate at 760 mm and 800°C $= \frac{0.03 \times 1073}{288}$

$= 0.112$ cu.ft./min. at 800°C

Consider the vaporised tar.

Normal rate of 260 gms./hr. $= 4.33$ gms/min.

Assuming a mean molecular weight of 200, this represents -

at N.T.P. - $\frac{22.4 \times 4.33}{200 \times 28.32}$ cu.ft./min.

At 400°C and 760 mm, rate $= \frac{22.4 \times 4.33 \times 673}{200 \times 28.32 \times 273}$

$= 0.0422$ cu.ft./min.

At 800°C and 760 mm, rate $= \frac{22.4 \times 4.33 \times 1073}{200 \times 28.32 \times 273}$

$= 0.0673$ cu.ft./min.

Therefore:-

Total Flow = 0.0701 + 0.0422

= 0.1123 cu.ft./min. at 400°C

and = 0.112 + 0.0673

= 0.1793 cu.ft./min. at 800°C

At sectional area 0.392 sq.in.

Velocity $= \frac{0.1123 \times 12 \times 12}{0.392}$

= 41.2 ft./min. at 400°C

Velocity $= \frac{0.1793 \times 12 \times 12}{0.392}$

= 65.9 ft./min. at 800°C

But packed length = 2 ft.

Therefore - Contact Time -

$= \frac{2 \times 60}{41.2}$

= 2.91 secs at 400°C

$= \frac{2 \times 60}{65.9}$

= 1.82 secs at 800°C

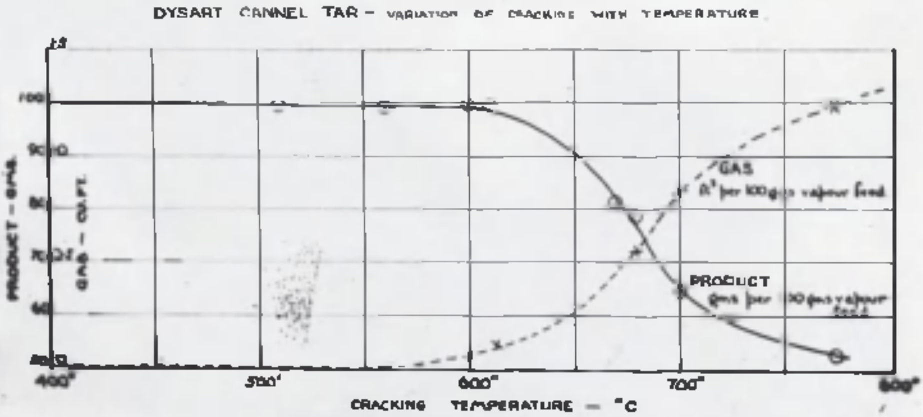

Fig. 17.

Dysart Cannel Tar, cracked in straight packed tube.

To face page 122

## B. Variation of Products with Temperature.

The influence of temperature on the cracking in this apparatus is well shown in Fig. 17. In this, the weight of product formed from 100 gms. of vapour feed is shown, along with the yield of gas produced from the same feed. It is at once evident that up to a temperature of about 600°C, the quantity of product remains largely unaffected, while very little gas is produced. As the temperature is increased beyond this point, however, cracking becomes more and more severe, resulting in the conversion of feed into gas, with consequent reduction in the amount of liquid products. This occurs over a fairly small temperature range, principally between 650° and 750°C.

### Tar.

From the results given in Table XVI, and shown in Fig. 18, it can be seen that there is no substantial change in the character of the liquid product formed below the temperatures which result in gas formation. These figures show the products formed from 100 gms. of vapour feed.

## TABLE XVI

### Cracking of Topped Dysart Cannel Tar.

### Straight Packed Tube.

| Test No. | Furnace Temp. °C | From 100 gms. Vapour Feed | | | | | Composition of Product | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Product gms. | Fraction in cc. | | | | Percent by Volume | | | | S.G. of Fractions | | |
| | | | Up to 180° | 180° 300° | 300° 350° | over 350° | Up to 180° | 180° 300° | 300° 350° | over 350° | Up to 180° | 180° 300° | 300° 350° |
| 10 | nil | 100 | 0.5 | 38.0 | 32.4 | 34.1 | 0.5 | 36.2 | 30.8 | 32.5 | .880 | .894 | 1.00 |
| 12 | 560° | 99.2 | 0.6 | 38.8 | 28.5 | 36.4 | 0.6 | 37.2 | 27.3 | 34.9 | - | .893 | .931 |
| 14 | 612° | 90.9 | 3.1 | 39.2 | 20.6 | 32.8 | 3.2 | 41.0 | 21.5 | 34.3 | .777 | .896 | .946 |
| 7 | 665° | 81.1 | 5.8 | 35.8 | 21.4 | 22.4 | 6.7 | 42.0 | 25.0 | 26.3 | .786 | .904 | .915 |
| 8 | 680° | 78.8 | 9.1 | 35.2 | 16.6 | 21.9 | 11.0 | 42.5 | 20.0 | 26.5 | .777 | .925 | 1.00 |
| 15 | 700° | 65.1 | 10.6 | 29.8 | 11.9 | 16.2 | 15.6 | 43.4 | 17.4 | 23.6 | .777 | .900 | 1.00 |
| 9 | 773° | 53.5 | 3.1 | 18.3 | 6.4 | 28.5 | 5.6 | 32.5 | 11.3 | 50.6 | .891 | 1.02 | 1.09 |

To face page 123

As the temperature is raised, the yield of spirit distilling up to 180°C increases, reaching a maximum of 15.6% of the product at 700°C. This figure corresponds to 10.6 cc. of spirit per 100 gms. of vapour feed. It will be seen that the spirit is produced at the expense of the higher fractions, especially the material boiling over 350°C, which is much reduced in quantity at 700°C. The product at this temperature is thus a very much lighter material than the feed.

Above 700°C cracking becomes more and more severe, the principal products being a heavy residue and gas.

The curves in Fig. 18 give an excellent picture of the properties of the different fractions, since they show how 100 gms. of feed are disturbed after passing through the cracking tube.

### Fraction up to 180°C

This fraction is absent in the feed, and is produced as a result of the cracking process up to 700°C. Beyond this, conditions are more favourable for gas formation, and gas is produced in preference to spirit as the temperature is increased further.

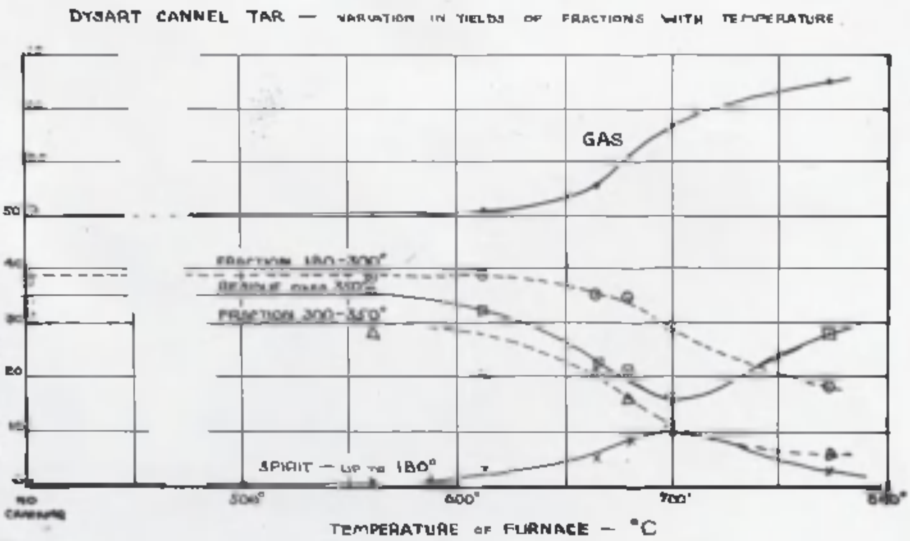

Fig. 18.

Dysart Cannel Tar, cracked in straight packed tube.

To face page 124

### Fraction 180° - 300°C

Until a temperature of about 650°C, this fraction would seem to be unaffected. The products of cracking are at first spirit and gas, since there is no increase in the heavier fractions. Above 700°C, cracking tends to produce gas and heavy material, with less and less spirit.

### Fraction 300° - 350°C

This behaves in much the same way as the previous fraction, cracking first to lighter material and gas, and then, as the temperature rises, to gas and heavy residue.

### Residue over 350°C

At about 700°C, this fraction seems to be readily cracked to lighter material and gas. As the temperature is increased, the cracking of the other fractions, and of the material originally forming this fraction, evidently becomes more severe, resulting in the formation of gas and pitch.

### Maximum Conditions.

It is obvious that a temperature of 700°C is the most effective under the conditions of these experiments. At this temperature:-

(a) Spirit production is a maximum

(b) Pitch production is a minimum.

This can be seen when the percentage composition of the product is compared with that of the vapour feed.

## TABLE XVII

| Fraction | Feed | Product at 700°C |
|---|---|---|
| Up to 180°C | 0.5% | 15.6% |
| 180° - 300° | 36.2% | 43.4% |
| 300° - 350° | 30.8% | 17.4% |
| over 350° | 32.5% | 23.6% |

### Spirit.

Owing to the small quantities of spirit available, no exhaustive tests were possible on this light fraction. The only properties noted were (a) Specific Gravity and (b) where possible, the percentage of unsaturateds and aromatics. The gravity was fairly constant between 0.777 and 0.794, though in the case of the most severe cracking - at 773°C - the gravity rose to 0.891. The unsaturated content, estimated by absorption in 80% sulphuric acid, was about 10%. The aromatic content - estimated with 100% acid - was between 36% and 46%. As very small quantities were used for these determinations, the figures give only a general idea of the constituents present in the spirits.

Gas.

The gas, where evolved in sufficient quantity, was analysed on a modified Podbielniak Unit. Typical analysis are given in Table XVIII.

## TABLE XVIII

Gases formed by Vapour-phase Cracking.

| | Cannel Tar | | | Gas Oil * at 690°C | Topped * Crude |
|---|---|---|---|---|---|
| | Run 8 680°C | Run 5 720°C | Run 9 773°C | | |
| $CH_4$ | 26.7 | 41.3 | 44.6 | 34.7 | 33.2 |
| $C_2H_4$ | 34.1 | 32.8 | 32.6 | 22.6 | 26.7 |
| $C_2H_6$ | 12.0 | 8.0 | 5.8 | 15.3 | 12.8 |
| $C_3$ | 16.6 | 12.1 | | 17.9 | 20.2 |
| | | | 17.1 | | |
| Resid. H'carbs | 10.6 | 6.0 | | 9.5 | 7.1 |

* From, "Science of Petroleum, Dunstan and others, Vol. III page 2115.

An interesting feature of the gases formed by the cracking process described in this chapter is the high content of ethylene. The highly unsaturated nature of the gas is also shown by the total percentage of unsaturated hydrocarbons as estimated by absorption in Bromine water. This value varied from 35% to 45%.

Wagner (Science of Petroleum, Dunstan and others, Volume III, page2115.), pointing out that the gases produced by the vapour phase cracking of different charging stocks show a surprisingly uniform composition, gives figures for a variety of charging stocks, cracked by various methods. Some of these are included in Table XVIII, where it can be seen that the gas from the cannel tar, while it shows some resemblance to the others, differs markedly in the proportions of ethylene and methane. The highly unsaturated nature would seem to be characteristic of cannel products, though in this case it is probably accentuated by the high cracking temperatures used, and by the short contact time.

# CHAPTER 6

## VAPOUR PHASE CRACKING OF CANNEL TARS.

## III - CRACKING IN STAINLESS STEEL TUBES.

The experiments described in the previous chapter showed that the packing possessed definite properties which minimised coking. At a temperature which caused coking on asbestos wool, for example, no coking took place on the porcelain chips which formed the packing material. This effect was even more noticeable when metal gauze was used to hold the packing in position in the tube, the gauze coking up almost immediately. (see p. 103) In view of the fact that cracking operations are normally carried out in metal tubes, without packing, a set of stainless steel tubes was made up to replace the straight packed tube. This arrangement gave three passes through the furnace, thus giving a total length of 7 ft. 6 in. A description of these cracking tubes and their headers is given in Chapter 4.

# CRACKING CONDITIONS.

## Cracking Tubes.

Two series of runs were carried out, in order to investigate the effect of this change in cracking conditions,

(1) Using the straight packed tube, and

(2) Using the stainless steel tubes.

## Temperature.

With each type of cracking tube, runs were carried out at several temperatures, in order to determine the conditions for maximum yield of spirit distilling to 180°C.

## Condensing System.

In these two series of runs, the condensing system used in the experiments described in the previous chapter was modified slightly. It was found that the tar vapours emerging from the cracking tube, and passing through a condenser before entering the precipitator, gave a mist composed of viscous particles which did not flow easily down the walls of the precipitator. The position of the precipitator and condenser were therefore reversed, so that the bulk of the tar was precipitated hot. In case the chilling effect of the condenser caused a further mist, a second precipitator was added instead of the second condenser. At the end of the first series of runs, it seemed that the spirit yield was rather low, and it would probably have been desirable to add another condenser. As this would have introduced a further variable in the second series of runs, it was not done. Thus the two series of runs described in this chapter are strictly comparable from this point of view. The actual yield of light spirit could be increased by a more efficient condensing system.

In order to obtain an estimate of the amount of light material carried off in the gas, an active carbon scrubber was introduced in certain of the tests. This, while it did not disturb the condensing system, showed what percentage of the light spirits remain uncondensed. The scrubber is discussed later.

### Feed.

The material used as a feed was a Cannel Tar obtained by Low Temperature Carbonisation of a Newbattle Cannel. The cannel was carbonised at the Fuel Research Station in the Fuel Research Board Low Temperature Vertical Brick Retort. (see King and Jamieson, Inst. Gas. Eng. Publication No. 162, 1937, for details of the carbonisation.) A sample of the tar was kindly supplied by Dr. J.G. King.

The crude tar was topped to 190°C, giving a charging stock with the properties shown below.

Newbattle Cannel Tar - Topped to 190°C

| | | S.G. |
|---|---|---|
| Distilled to - 180°C | nil | - |
| " 180° - 300°C | 29% | 0.8997 |
| " 300° - 350°C | 20% | 0.9422 |
| Residue over 350°C | 51% | - |

Circulating Gas.

The gas rate was exactly as in previous runs, and was kept constant at 0.03 cu.ft./minute.

Cracking Time.

The cracking time for the stainless steel tubes was estimated as in Chapter 5, the figure of 200 being adopted as before for the molecular weight of the vapour feed. The calculations were made for the same temperature conditions as before.

Nominal bore of tube - $\frac{1}{4}$ in.

$\therefore$ Free sectional area = 0.0491 sq. in.

Consider the circulating gas -

Rate as before, at 760 mm. -

= 0.0701 cu.ft./min. at 400°C and

0.1120 cu.ft./min. at 800°C

Consider the vaporised tar -

Rate as before, at 760 mm. -

= 0.0422 cu.ft./min at 400°C

and 0.0673 cu.ft./min at 800°C

Thus Total Flow = 0.1123 cu.ft./min at 400°C

and 0.1793 cu.ft./min at 800°C

At sectional area 0.0491 sq. in.

Velocity is $\frac{0.1123 \times 12 \times 12}{0.0491}$

= 329.5 ft./min. at 400°C

and $\frac{0.1793 \times 12 \times 12}{0.0491}$

= 526.0 ft./min. at 800°C

Total heated length = 7' 6".

Therefore - contact time -

$= \frac{7.5 \times 60}{329.5}$

= 1.41 secs. at 400°C

$= \frac{7.5 \times 60}{526.0}$

= 0.866 secs. at 800°C

The figures for cracking times are thus -

| | at 400°C | at 800°C |
|---|---|---|
| Series 1 - Straight packed tube | 2.91 secs | 1.82 secs |
| Series 2 - Stainless steel tubes | 1.41 secs | 0.866 secs |

# TABLE XIX

## Cracking of Topped Newbattle Cannel Tar.

| Test No. | Furnace Temp. °C | From 100 gms. Vapour Feed | | | | | Composition of Product | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Product gms. | Fraction in cc. | | | | Percent by Volume | | | | S.G. of Fractions | | |
| | | | Up to 180° | 180° 300° | 300° 350° | over 350° | Up to 180° | 180° 300° | 300° 350° | over 350° | Up to 180° | 180° 300° | 300° 350° |
| Cracking in Straight Packed Tube - Series 1 | | | | | | | | | | | | | |
| 26 | 585° | 99.4 | 0.9 | 42.8 | 25.2 | 35.6 | 0.9 | 41.0 | 24.0 | 34.1 | .792 | .903 | .963 |
| 28 | 640° | 89.6 | 2.5 | 36.8 | 18.6 | 36.4 | 2.5 | 39.0 | 19.8 | 38.6 | .802 | .908 | .966 |
| 29/30 | 655° | 82.7 | 3.8 | 35.3 | 20.0 | 37.0 | 3.4 | 37.0 | 21.0 | 39.0 | .810 | .912 | .972 |
| 27 | 680° | 77.4 | 3.3 | 31.3 | 15.3 | 30.4 | 4.3 | 39.0 | 19.0 | 37.8 | .813 | .923 | .994 |
| 31 | 695° | 76.8 | 1.2 | 29.5 | 13.3 | 36.8 | 1.5 | 36.5 | 16.5 | 45.5 | .815 | .932 | .995 |
| Cracking in Stainless Steel Tubes - Series 2 | | | | | | | | | | | | | |
| 32 | 565° | 100.0 | 0 | 35.8 | 24.8 | 44.8 | - | 34.0 | 23.5 | 42.5 | - | .903 | .944 |
| 34 | 680° | 91.6 | 1.3 | 35.8 | 19.8 | 39.7 | 1.3 | 37.0 | 20.5 | 41.2 | .823 | .929 | .988 |
| 33 | 695° | 86.0 | 2.4 | 35.3 | 16.8 | 36.2 | 2.6 | 39.0 | 18.5 | 39.9 | .809 | .911 | .970 |
| 36 | 720° | 77.1 | 4.7 | 30.8 | 14.6 | 31.0 | 5.8 | 38.0 | 18.0 | 38.2 | .812 | .920 | .981 |
| 35 | 735° | 74.3 | 2.7 | 29.0 | 12.9 | 33.7 | 3.5 | 37.0 | 16.5 | 43.0 | .823 | .929 | .988 |

To face page 133

## THE COMPARISON OF TWO TYPES OF CRACKING TUBE.

The results for the two series of runs are given in Table XIX. The yields of product, spirit and gas are those given by 100 gms. of vaporised tar entering the cracking tube. The composition of the product shown is the actual distillation range by volume, the specific gravity of the fractions being also given.

### Product.

It can be seen from the data in Chapter 5, that the amount of cracking can be judged approximately from the quantity of product and gas formed from a given amount of vapour feed, cracking being most effective just after gas production starts. This point is shown by an inflexion in the product/temperature curves. When this curve is plotted for the two types of cracking tube, the stainless steel tube shows a difference from the packed tube, a higher temperature being necessary for a given amount of cracking. The cracking range, also is narrowed down. This is shown more effectively when the yield of spirit is plotted against temperature. (Fig. 19)

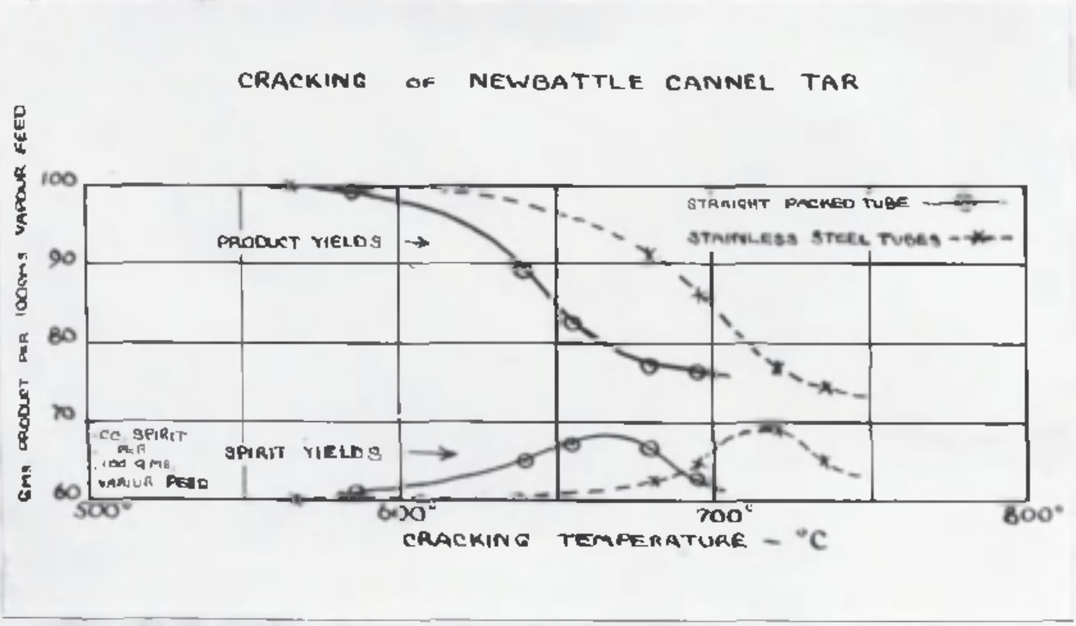

Fig. 19.

Newbattle Cannel Tar.

Comparison of types of cracking tubes.

To face page 154

## Spirit.

The maximum spirit yield shown is approximately 5 cc. per 100 gms. vapour feed. This occurs at 660° - 670°C for the packed tube, and 710° - 720°C for the stainless steel tubes. It was not possible to examine the spirit in detail in order to find any differences in properties between the two types, but the specific gravity of the spirits show great similarity. For example, the gravities of the spirits produced at 680°C in the packed tube and 720°C in the steel tube are 0.813 and 0.812 respectively. The positions occupied by the two spirits on the curve are very similar - both occur just after the point of maximum spirit production. Again, taking another pair of roughly similar points, the specific gravity of the spirits at 655°C (series 1) and 695°C (series 2) are 0.810 and 0.809 respectively.

## Gas.

No hydrocarbon analysis were done on the gases formed during these runs, but analysis were made using a partial combustion furnace for methane and ethane. Typical analysis are given in Table XX.

## TABLE XX

| | Series 1 | | Series 2 |
|---|---|---|---|
| Temperature | 655°C | 695°C | 695°C |
| $CO_2$ | 1.1% | 1.3% | 1.2% |
| $O_2$ | 0.5% | 0.6% | 0.7% |
| Unsats. | 49.3% | 45.3% | 51.6% |
| $H_2$ | 2.2% | 3.4% | 2.2% |
| CO | 0.2% | 0.6% | 0.5% |
| $CH_4$ | 41.7% | 40.9% | 35.5% |
| $C_2H_6$ | 5.1% | 7.9% | 8.4% |

These figures show, as before, a high percentage of unsaturated hydrocarbons of from 45 - 50%. Further aspects of this are discussed later.

Scrubber.

The spirit scrubbed from the gas in these experiments was fairly constant in amount, and varied from 4 - 6 cc. per 100 gms. vapour feed. The addition of this to the spirit yield increases the maximum obtained to approximately 10 cc. per 100 gms. feed. The exact amount of this spirit would of course depend on the efficiency of the scrubber used.

## Conclusions.

There seems little difference between the two types of tube which could not be explained by the difference in cracking time. As can be seen from the figures given on page 135, this time lies between 2.91 and 1.82 secs. for Series 1, as against 1.41 and 0.86 in Series 2. This gives a cracking time for the steel tube of approximately half that for the packed tube. This would probably account for. -

(1) The narrower cracking range in the case of the steel tubes.

(2) The higher temperature necessary in this case owing to the shorter contact time.

The packed tube, however, showed a much greater freedom from coking. In this respect it was definitely superior to the steel tube.

The practicability of using the packing in a large tube would of course require investigation. In addition, a larger diameter tube such as would be used would not coke so readily as the narrow steel tubes used in Series 2. It is possible, then, that the advantages of the packing would be apparent only on the small scale used in these experiments. The freedom from coking is, however, an interesting property of the packing . (see below, p. 139 )

## CRACKING OF CANNEL TARS.

Published work on the cracking of cannel tars has been confined to that of Morrel and Faragher (Ind. Eng. Chem. 1929, 21, 1084.) and Jamieson and King (Proc. I.P.T. Conference on Oil Shales and Cannel Coals, 1938, 452.) The main results of these workers, along with those described in this and preceding chapters, are summarised in Table XXI.

Table XXI

| | Present Work | Jamieson and King | Morrel and Faragher |
|---|---|---|---|
| Conditions | Vapour Phase Atmospheric 700°C | In an Autoclave 400 lbs/sq.in. 430°C | Continuous Unit 125 lbs/sq.in. |
| Cracking Stock | Dysart Tar distillate | Newbattle tar distillate | Kentucky Cannel Tar |
| Distillate to 180°C | 18.4% | 2.5% | - |
| <u>On Cracking</u> | | | |
| Up to 180°C | 6.0% | 9% | 22.2% |
| 180°- 300°C | 19.3% | 9% (to 170°) | 19% |
| 300°- 350°C | 8.6% | 2% (300°-360°) | - |

It will be seen that neither of the Scottish cannels give a yield of spirit comparable to that from the Kentucky cannel. Whether this is due to the properties of the tars, or to the cracking methods is difficult to say. Certainly the conclusions reached by Morrel and Faragher were - "A crude cannel coal tar has been cracked to give high yields of gasoline excellently suitable for motor spirits." On the other hand, Jamieson and King conclude that the products obtained by simple distillation are preferable to the cracked product since, although the yield of spirit is less, it is only slightly so, while distillation gives a greater yield of diesel oil, of better quality. In the case of the vapour-phase cracking, the yield of diesel oil would be larger, but is still not up to that obtained by simple distillation (about 25%).

If, then, the low yield of spirit is attributable to the method of cracking, the process used by Morrel and Faragher might be profitably employed. If, the tar itself is an unsuitable cracking stock, the straight distillation of the tar must be the most effective method of producing products for use as motor fuels.

The large yield of unsaturated gases shown in this and the preceding chapter might direct attention to the gaseous products. In this connection the work of Tropsch, Thomas, Egloff and Morrel (Ind. Eng. Chem. 1938, 169) is of interest. This work, which is mentioned on page 103 in connection with the packing of the cracking tube, was carried out with the object of producing dienes by cracking a gas oil. The conditions assumed to favour the reaction were:-

1. Since aromatics can be produced from almost any source, then conjugated dienes should also.
2. Aromatics are formed usually above 600°C in vapour-phase cracking. This should hold for dienes.
3. Conjugated dienes are highly reactive: therefore a short reaction time is necessary.
4. The reaction dienes aromatics is bimolecular. Low pressures should hinder this secondary reaction.

Accordingly a gas oil, vaporised at 400°C under a pressure of 175 mm., was passed through a tube at 900°C, and the products condensed in solid $CO_2$ and liquid nitrogen. The tube was packed with a porcelain packing similar to that described in Chapter 4, and the same freedom from coking, along with the deposition of a carbon film, was noted. The contact time was 0.05 seconds.

The products formed at 950°C and 175 mm. were:-

| | |
|---|---|
| Hydrogen | 0.6% by wt. |
| Methane | 3.0% |
| Ethylene | 24.6% |
| Ethane | 9.2% |
| Propylene | 17.2% |
| Propane | 0.9% |
| Butadiene | 5.8% |
| Butenes | 7.3% |
| Butanes | 0.3% |
| $C_5$ Conjugated dienes | 2.7% |
| Other Hydrocarbons | 3.2% |

<u>Gasoline</u>.

| | |
|---|---|
| Conjugated dienes | 6.3% |
| Other Hydrocarbons | 3.2% |
| Residue at 200°C | 12.5% |
| Unaccounted for | 0.2% |

Such unsaturated compounds would be very useful starting materials for synthesis, and in view of the highly unsaturated nature of the gases from cannel tar at temperatures below 700°C, the application of a high temperature and a low pressure should give a satisfactory yield of dienes. Instead of using a pump to produce a low pressure, the introduction of circulating gas as is done in the apparatus described in Chapter 4 should reduce the partial pressure of the tar vapours in a simple and easily controllable manner.

In contrast to this method, the polymerisation of such unsaturated gases offers a possibility. The products in this case are generally aromatic spirits suitable for use as motor fuels. An excellent summary of the processes used is given by Howes, in "The Science of Petroleum" - (Dunstan and others) Volume III page 2045.

# CHAPTER 7.

## OILS FROM CANNEL COAL.

It is probably true to say that the chief reason for the interest in the various oil from coal processes in this country at present is a desire to provide a source of home produced oil for use in a national emergency. That this will always hold is decidedly unlikely. Were the possible need for national self sufficiency removed, the high present production of natural petroleum would ensure a plentiful supply of the various oil fuels. World petroleum resources, however, are very much more limited than those of coal, and the possibility remains that, failing the supply of new types of fuel, the oil demand of the world will have to be supplied from the resources of coal and other oil-yielding minerals.

But these were not primarily the motives which prompted the Scottish Development Council to investigate the problems of oil production from coal. Nor could they be discussed adequately in such a thesis as this. From the scientific and technical standpoint, the main interest in such questions lies in the possibilities of new processes and new methods applied to our natural resources.

During the greater part of the work described in this thesis, the latter consideration could not be the only one. The interests of the Development Council lay in setting up new industries in Scotland: and apart from the technical success of any new process, its economic possibilities had to be examined. For this reason many points were investigated which were not of immediate scientific interest.

## Bituminous Coal.

Present work on oil production in this country has been almost entirely directed to the carbonisation of bituminous coal. Of this there are large proved reserves, of well known properties, and which are easily and constantly available - in Britain at any rate. Bituminous coal, from the oil production point of view, has the disadvantage that the oil yield is low, so that for a given oil production a large quantity of coal must be retorted, with the production of a corresponding amount of coke. Although the latter is saleable as a smokeless fuel, the economic success of the process is dependent on the price obtained for this residue.

The development of hydrogenation led to an increased interest in other methods of treating coal. The most important of these being:

(a) Hydrogenation

(b) Hydrocarbon Synthesis from Water Gas.

In this country, only the hydrogenation has been tried out on a large scale. The Billingham plant of Imperial Chemical Industries started operation in 1935, to produce 45 million gallons of petrol per annum. The laying down of plant on this scale is a desirable feature, as the results of such full scale operation give accurate data for assessing the value of the process.

Although the Fischer-Tropsch synthesis is carried on in Germany, no large plant for the production of oils by such a process has been erected in this country. Some experience of the similar Robinson-Bindley process will be gained in Britain, however, by Messrs Synthetic Oils Ltd., at the plant they are erecting at Bedley Colliery near Glasgow.

The economic and practical features of the various oil from coal processes are discussed in the report issued by a sub-committee of the Committee of Imperial Defence. This committee, under the chairmanship of Viscount Falmouth, reported in 1938. Their terms of reference were "To consider and examine the various processes for the production of oil from coal and certain other materials indigenous to this country, and to report on their economic possibilities, and on the advantages to be obtained by way of security of oil supplies in an emergency".

The most important conclusion reached by the committee was that in time of war it would be more advantageous to import and store petroleum than to produce it at home from coal and similar materials. They recommend strongly, however, that the possibilities of the low pressure synthetic processes such as the Fischer-Tropsch and Robinson-Bindley should be investigated in this country on a scale of at least 20 - 30,000 tons of product per annum.

The possibilities of such processes as applied to Scottish bituminous coals, are discussed in the First and Third Reports of the Oil from Coal Committee (Scottish National Development Council, Economic Series Nos. 12 and 14.)

## Other Minerals.

When the oil industry was founded in this country the materials used for retorting gave oil yields which enabled the process to be carried out economically without reliance on the sale of a residue. This is still a feature of the shale industry, although some of the spent shale is at present used for brick-making.

Thus the choice of raw material so far as carbonisation is concerned lies between:-

(a) Bituminous coal, which although easily available gives a small oil yield and a large residue, the sale of which must therefore be a deciding factor in the economics of the process.

(b) Oil bearing minerals such as shale and cannel, which give a high yield and can be retorted for oil primarily, with the sale of a residue as a secondary consideration.

The continued existence of the shale industry in Scotland is evidence that the retorting of this mineral can be carried out efficiently, and, though perhaps not in direct competition with natural petroleum, then on a profitable basis under present Government preference for home produced oils.

To what extent, then, could this be assumed for a possible cannel industry? And what are the factors which would favour the establishment of such an industry?

(1) From the point of view of the Scottish Development Council, cannel is a material indigenous to Scotland, and its exploitation would be of assistance to Scottish industry. This was of course the main consideration underlying the Cannel Inquiry.

(2) From the national point of view, cannel is a possible source of home-produced oil, and its potentialities should therefore be examined.

(3) From a practical point of view, the criterion is the availability. Shale reserves have the advantage of being proved and are at present in production. In the case of cannel, seams formerly worked are now closed, and in the case of some seams now proved to exist, new workings would be required. Here economic considerations predominate in deciding on the merits of various seams.

(4) The reserves are important when the rate of production and length of life of a possible cannel industry are considered.

(5) The quality and yield of the various products is the factor which would control the ultimate success of such an industry. At present information on this subject is meagre:

particularly so with regard to the application of modern refining methods to these oils. This factor seems to complete a vicious circle:- such experiments could give conclusive results only when carried out on adequate quantities of cannel oil. But the plant necessary for production of the oil would not normally be laid down until conclusive results are available.

The work described in this thesis was concerned largely with such physical and chemical properties of the oils obtained from cannel coal as indicated their suitability for use as fuels of various kinds. Nevertheless, all these considerations had to be borne in mind when the results were interpreted. From the results obtained it was attempted:-

To estimate the yield of motor spirit, diesel oil etc., likely to be obtained from Scottish Cannels. To estimate the quality of the motor spirit, diesel oil, and lubricating oil which might be obtained.

and taking these into account, to indicate the general methods by which the products might be obtained to best advantage.

Attention was concentrated on the oil product. The gas and the residue on carbonisation were not extensively examined.

On this basis the main conclusions to be drawn were:-

(1) The existence of large reserves of cannel of good oil yield, and in sufficient thickness for economic working is shown by the survey carried out by the Oil from Coal Committee of the Scottish Development Council. The results of this survey are indicated briefly in Chapter 1.

(2) The quality of the oils from the different cannels, although variable, was good in the case of all the samples examined as described in Chapter 2. Under the conditions of carbonisation described there, there were produced:-

From 0.74 to 1.84 gals/ton of spirit from the gas

From 5.3 to 14.2 gals/ton of spirit from the tar

From 3.9 to 28.3 gals/ton of oil distilling $180^{\circ}$-$300^{\circ}$C (i.e. potential diesel oil)

From 9.4 to 15.1 cwts/ton of residue, in most cases non-coking, and varying in ash from 3% to 60%

Approximately 2000 cu.ft. of gas of high C.V. (about 1000 B.Th.U.)

(3) It is possible to obtain a motor spirit, in good yield, from the tar, exclusive of the light material available by scrubbing the gas. This latter, according to the experience of Jamieson and King (as quoted p. 95 ) should be of excellent quality and could be used as a first grade spirit, or could be blended with the spirit from the tar to give a larger yield of satisfactory motor fuel. The properties of the tar spirit are given on page 94 , and show it to be a spirit of indifferent quality,

but which could be used as a blending material. The yield of the refined spirit in the case of the oil examined, was large - 12 gallons per ton.

(4) A satisfactory diesel oil could be obtained, but some refining would be necessary. This need not be excessive, however, as thorough refining with furfural, on a heavy fraction, gave a diesel oil in excess of the specifications demanded at present. The untreated diesel fraction, also, was almost identical with that obtained from Scottish shale oil. The ignition quality, was not so good, but it must be borne in mind that the cannel oil tested was quite untreated, while the shale diesel oil is a refined product. There is no doubt that the ignition quality could be improved substantially, without a serious loss. The yield of crude diesel oil from the cannel investigated was 20 gallons per ton. By including a heavier fraction in the diesel oil, this could be raised to over 30 gallons.

(5) Attempts to isolate a satisfactory lubricating oil were unsuccessful. Oils were obtained which had satisfactory viscosity characteristics but the resistance to

oxidation was poor. In view of the recent advances in solvent refining methods it is very probable that the unstable fractions could be extracted, to give a small yield of oil suitable for use as a lubricating oil. The extracted material could then be cracked, or used as a fuel oil.

(6) An increased yield of spirit can be obtained by cracking, but the cannel tar does not seem to be a good cracking stock, tending to crack to gas and pitch rather than to lighter liquid products. The maximum conditions found among those investigated gave 6.0% by weight of the tar as the yield of cracked spirit distilling below 180°C. This compares unfavourably with the yield of 9.0% obtained by Jamieson and King, from a Newbattle cannel tar, and even more so with that of 22.2% obtained by Morrell and Faragher. These figures are discussed in greater detail on pages .

(7) Reference has been made (p. 63 ) to the vapour phase hydrogenation-cracking of cannel tar and to the report by the Director of Fuel Research that the tar - a Dysart cannel tar - was an excellent material for this purpose. In their paper Jamieson and King mention that

a quantity of their high temperature tar was sent to Imperial Chemical Industries at Billingham for test in their hydrogenation plant. No details have been published as to whether this test showed the same promise as the small scale test at the Fuel Research Station. It is known that large quantities of creosote are being hydrogenated at Billingham, and according to an I.C.I. witness before the Falmouth committee, (Falmouth Report, p. 24.) this organisation prefers the high temperature product to low temperature tar. As long as creosote was available in adequate amount it would seem that the preference would lie in the direction of its use.

While cannel tar may yield comparatively large volumes of low grade motor spirit either by low or high temperature carbonisation, it is obvious that the economics of the process, if production of motor spirit is the aim, must rest on the hydrogenation of the liquid fractions. Even if a good yield of spirit were obtained by cracking, there would remain a cracked residue for disposal, presumably by hydrogenation.

Imperial Chemical Industries (Falmouth Report p. 34.) do not propose to extend their present hydrogenation activities, as present economics would appear to be against such a course. If the nation required oil during war time, however, the economics would be considerably modified. Under such circumstances, if the hydrogenation of low or high temperature tar were contemplated, cannel tar, produced in two to three times the volume per ton of raw material, would appear to be a raw material well worth investigating from this angle.

(8) The tendency of cannel tar to crack readily to gas is discussed in Chapter 6. The cracking of the tar to yield this highly unsaturated gas is a possibility in view of the interest now being taken in polymerisation and "reforming" these hydrocarbon gases. This it might well be that the production of saleable oils could be achieved by first cracking the tar to gas of suitable composition, followed by synthesis from the gas of a spirit which could be used as a motor fuel. The possibility also exists that the gas might be used, not to produce petroleum hydrocarbons, but as a starting point for the synthesis of other organic compounds. It must be borne in mind ,that when cracking is

considered, it can easily be applied only to such fractions of the tar as are not directly suitable for use as fuels. Solvent refining processes, for example, which would give satisfactory motor spirit, diesel oil, and lubricating oil would also give low grade residues which might form suitable material for cracking to gas.

Within the past few years, estimates of the possible yields of products from the carbonisation of cannel have been made by Jamieson and King (Inst. Gas. Eng. Communications No. 135, 1936, and 162, 1937. also Proc. Conference on Oil Shales and Cannels, 1938, p. 452.) by the Oil from Coal Committee of the Scottish Development Council (2nd. Report, Economic Series No. 13.) and by Cumming and Nisbet (Proc. I.P.T. Conference on Oil Shales and Cannel Coals, 1938, p. 423.) The main products envisaged in all these estimates are motor spirit and diesel oil, the residue being considered as suitable for sale as a smokeless fuel, or as a source of water gas for synthesis. Such heavy fractions of the oil as have been isolated have proved unsuitable for use as lubricating oils.

The methods suggested for treating the cannel include:-

Hydrogenation of the cannel coal.

Carbonisation followed by - distillation of the tar

- cracking

or hydrogenation-cracking.

The solid residue to be - sold as fuel

or gasified to yield further oils by synthesis.

These methods are illustrated diagramatically in Fig. 20, which shows most of the processes so far advanced for dealing with cannel. In the scheme are included a variety of different possibilities: which of these would be adopted in practice would depend on the products desired, and on the market conditions and technical merits of the individual methods.

It will be seen that the principle products are motor spirit and diesel oil. More extensive investigations might make it possible to produce small amount of more valuable by-products which might well alter the balance from economic failure to success.

This idea is expressed by Col. Auld in a discussion at the Institute of Petroleum Conference on Oil Shales and Cannels. He states (Proceedings,

p. 439) "--- it looks to me as if special technique or the development of special products might be the only real likelihood of making the exploitation of cannels a success. ------- every effort must be made to produce specific products or intermediates capable of carrying the high cost of exploitation."

From such estimates, also, as those of A.J.V. Underwood (Ibid. p. 468) it can be seen how many unknown factors there are. Yields of products can be given with a fair accuracy. Their properties, however, are not well established. When they are refined, the methods are simply those in normal use: methods specifically adapted to cannel oil have not been evolved.

The reason for this lack of information seems to lie in the concentration on methods of carbonisation rather than on examination of the products. Alteration in the retort used, so long as heating conditions are not unduly severe, need not seriously alter the properties of the cannel tar produced at a given temperature. The most urgent need is for information on the properties of the tar. The present method of approach to this problem, too, is to ask "What yields of normal petroleum products can be obtained?". Would it not be better to frame the question "What are the distinctive properties and constituents of cannel tar, and how may they best be utilised?".

From this latter point of view, the examination of cannel might be directed along such lines as:-

(1) The production of adequate amounts - several hundred gallons - of cannel tar in a retort of normal design, so long as excessive heating of the cannel and the evolved vapours is avoided. Owing to the non-coking character of most cannels, their heat conductivity is good, and avoidance of excessive temperatures is simplified.

(2) The extraction of the tar acids and tar bases, and their identification. This has been carried out to some extent on shale oil, (see for example, Gray, J.S.C.I. 1902, 21, 845.), and might give interesting evidence as to their relationship.

(3) The separation of three or four main fractions from the neutral oil by topping and flash vaporisation, and vacuum flash vaporisation, or by such methods as would reduce thermal decomposition to a minimum.

(4) Further fractionation of these wide cuts, and estimation of aromatics, naphthenes, etc., by normal absorption methods, or by such methods

as those of Vlugter, Watermann and Van Westen (J.I.P.T. 1935, 661.) This, by giving an idea of the type of oil, would direct attention to a suitable refining method.

(5) Production of a diesel oil of a satisfactory ignition quality by refining a suitable fraction. Various solvents for extraction could be investigated, particularly liquid $SO_2$.

(6) Preparation of solvent extraction data for the heavy fractions using a variety of solvents - Liquid propane, liquid $SO_2$, furfural, nitrobenzene, chlorex, and other solvents, to produce a fraction suitable for use as a lubricating oil and possessing the required oxidation resistance. Dewaxing could be carried out by normal methods, or by the use of a suitable dewaxing solvent.

(7) Treatment of the heavy fractions by hydrogenation to produce stable lubricating oils. Such processes are in commercial use and might lend themselves to such unsaturated material as cannel oil.

(8) Cracking of the tar or tar fractions at a high temperature in the vapour phase to produce a gas which might be used for polymerisation or for synthesis. Such products

as styrene and indene have been obtained by pyrolysis of natural gas at 800° - 1000°C (Birch and Hague, Ind. Eng. Chem. 1934, 26, 1008 and Dunstan, Hague and Wheeler, Ibid, 1934, 26, 319.) Such products would be valuable and might have a great effect on the economics of a cannel industry.

(9) Examination of the cannel residue for use in water gas producers: with the ultimate object of producing a synthesis gas for treatment by the Robinson-Bindley or Fischer-Tropsch process.

The cracking of the gas, particularly that produced at high temperatures (see Jamieson and King's papers quoted elsewhere) might well receive attention, if processes such as the Fischer-Tropsch were under consideration. In addition to the oil and motor spirit obtained, the large volumes of gas could be cracked under controlled conditions to give a suitable synthesis gas, while the coke left in the retorts could be utilised for heating the cracking units and for production of steam, etc. The synthesis processes which, in spite of many authoritative recommendations (see Falmouth Report, 1938, p. 62.) have not been tried out on a large scale in this country, would appear to be one of the methods which could make use of the cannel resources for the production of oil

When there are so many such problems still to be investigated, it is difficult to reach a satisfactory conclusion as to the possibilities of cannel. Such problems as have been answered, however, would show:-

(a) That the competition of natural petroleum is sufficiently great to make the home production of oil from cannel a purely emergency measure.

(b) That useful products can be obtained from cannel in the form of motor spirit and diesel oil.

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# APPENDIX.

## A PROPOSED LABORATORY CARBONISING UNIT.

## A PROPOSED LABORATORY CARBONISING UNIT.

The following drawings are preliminary sketches of a carbonising unit which it is proposed to erect in the Technical Chemistry Department of the Royal Technical College, Glasgow. It was originally intended for the laboratory carbonisation of cannel. As it would also be used for instructional purposes, it was designed for very varied conditions, so that high temperature as well as low temperature carbonisation could be carried out.

### REQUIREMENTS.

As originally envisaged, the retort would have a capacity of 40 lbs. of cannel. This, however, would be an unwieldy unit for laboratory work, so that a charge of 11.2 lbs. (i.e. 1/200 ton) was allowed for. A cannel giving 40 gallons per ton would therefore yield about a litre of tar for examination.

The retort would require to stand up to varied temperature conditions, as temperatures of only 400°C might be used for cannels, while work under modern gas-making conditions would involve temperatures of over 1000°C. Thus the temperature range is very great.

## MATERIAL.

The decision as to the material to form the retort proper was therefore difficult, as silica, for example, which has excellent heat-resisting powers, would be rather fragile. A suggestion made subsequent to the drawing of the plans shown, was that the liner should be made removable, so that the low temperature work could be carried out in a cast iron retort, which could be replaced by a silica one for high temperatures. This plan would necessitate changes in the design of the furnace, otherwise the flues would not be gas-tight and draught would be interfered with.

In the drawings shown, the retort is made of silica. On inquiry it was ascertained that a retort could be made. in this material. By allowing room under the furnace for withdrawing the tapered silica liner, it might be possible to replace it if desired with a liner of another material.

Materials which might be used, and the important properties, are:-

| | |
|---|---|
| (1) Silica. | Very good resistance to heat, and to temperature changes. Heat conduction satisfactory. Mechanically weak. |
| (2) Silimanite. | Similar to silica, though not quite so fragile. |
| (3) Cast Iron. | Suitable for use up to about 500°C, though prolonged heating at this temperature would cause serious scaling. The advantages would be the mechanical strength and good heat conduction. |
| (4) Iron Alloys. | Such materials as the Iron-Silicon alloys would give greater heat resistance and would enable higher temperatures to be used - say up to 700°C. The higher temperatures, however, would still be outwith their useful range. |
| (5) Nichrome. | Special alloys such as Nichrome would give satisfactory service up to 1000°C. If much higher temperatures were used, the life would be shortened. The use of such materials is limited chiefly by the expense. |

## RETORT.

The suggested form of retort is shown in drawing R2. The retort proper is 3ft. 6in. long, with a cross-section of 6in. x $2\frac{1}{2}$in. at the bottom, tapering to 5in. x 2in. at the top. The ends are closed by cast iron end-pieces as described below, and shown in drawing R3.

The slightly tapered shape was adopted to aid discharging. With the non-coking cannels, no difficulty was anticipated, but if the more readily coking bituminous coals were used, the taper in the retort would be of great importance.

In the design suggested here, it is proposed to have the retort made of silica. While this is quite suitable for low temperatures, it would also enable conditions holding in high temperature gas practice to be reached.

## CHARGING GEAR.

The details of the fittings for charging and discharging are shown in drawing R3. With the bottom door closed, the material is charged by removing the cover-plate on the top header. As this merely involves removing four easily accessible bolts, it was felt that more complicated swinging gear was unnecessary. The vapour offtake is in the side of the header so that no connections are disturbed. As the cover is away from the hot zone, a normal jointing material such as "Walkerite" would be used.

To discharge, the bolt B would be slackened, and knocked aside. This would then allow the strap S to fall, taking with it the end piece P which it carries. This leaves the full width of the retort free, and the charge would either drop out or be assisted out by a metal poker. The machined faces of this joint, coated with red lead, should give a gas tight joint.

In the drawings shown, no provision is made for steaming. If high temperature gas-making conditions were to be reproduced, this would be necessary. The steam connections could then be made at the points on the bottom header marked C.C.

## HEATING.

Electrical heating was considered on account of the convenience and ease of control, but was rejected as being expensive. In addition, difficulty would be experienced in reaching the higher temperatures. If the heating chamber is properly lagged, and other precautions are taken to avoid heat loss, the gas heating should be satisfactory and should not heat up the room unduly. In addition some of the gas produced during the carbonisation might be used for heating.

In the design shown, gas and air enter together at the bottom of each side of the retort (at G, G) as shown in the sectional plan AA, drawing R2. The products of combustion then travel up one half of each side of the retort, and down the other. Then, via the flues F,F, the gases pass up the end walls of the retort, the two streams joining as shown in section CC, and emerging at the top to waste.

In a laboratory unit it would be desirable to cut down the brickwork to a minimum, so that heating up of the unit will occupy as little time as possible. Where possible (for example as shown by the dotted line in the sectional elevation XX, drawing R2,) it may be possible to cut away some brickwork which would be replaced by metal reinforcing, the space being filled with asbestos or slag wool to give insulation with low heat capacity.

It is difficult to estimate the amount of heat required, as losses can be very heavy. In such a unit it is desirable to have a large reserve of heating power, so that the temperature can be raised quickly. The laboratory supply of gas is considered adequate for the unit.

## CONDENSING EQUIPMENT.

### AIR CONDENSER.

The air condenser shown in the layout (drawing R8) would be necessary when cannel coal was being carbonised, as the wax content of cannel tar is very high. This condenser could be allowed to warm sufficiently to keep the wax molten. The heavier material, including the wax, would then condense in the air condenser. The main condenser could then be kept as cold as possible to condense the lighter fractions.

It consists of a tube bent as shown in the layout, with a nipple welded into the bottom of the U to carry a cock.

## WATER CONDENSER.

The condenser is shown in drawing R4. It consists of a vessel 5in. internal diameter, closed at the bottom, where a cock is fitted, and flanged at the top. The top flange carries a header for the water tubes. The tubes are made in a U - shape, the cooling water flowing from one side of the header, down and up the U - shaped tubes, and out at the other side of the header. This arrangement, although more complicated than the normal straight tube condenser, allows the tubes to be lifted out for cleaning. Where small quantities are dealt with, as on this unit, it is important to minimise hold-up, and where this is unavoidable, cleaning should be possible.

The condenser contains five steel tubes of $\frac{3}{4}$in. O.D., giving a total surface area of 3.45 sq.ft.

Assume a total gas evolution of 2000 cu.ft. per ton of coal. This represents 10 cu.ft. of gas from this retort in a period of say 4 hours. The mean rate of gas evolution is thus 2.5 cu.ft./hr.

Allow a maximum gas evolution of 5 cu.ft./hr. If a total of 1000 gms. of tar is obtained, this 5 cu.ft. would contain 500 gms. of tar. If the gas enters the condenser at 200°C and leaves at 30°C, then

Heat to be removed

$$= (170 \times 5 \times 12.75) + (170 \times 500 \times 0.4) + (500 \times 84.8) \text{ calories.}$$

where:-

12.75 = Specific Heat of gas, in cals/cu.ft./°C

0.4 = Specific Heat of tar and tar vapour in cals./gm./°C

84.8 = Latent Heat of Condensation of tar in cals./gm.

This gives a total of 97,220 calories/hr.
or 385 B.Th.U./hr.

At a water temperature of 20°C, the approximate log. mean temperature difference is 60°, so that at an overall heat transfer coefficient of 20 B.Th.U./sq.ft./°C/hr. (which is very low)

$$\text{Surface area reqd.} = \frac{385}{20 \times 60}$$

$$= 3.2 \text{ sq.ft.}$$

This surface area is exceeded slightly, (by 0.25 sq.ft.) so that the conditions assumed (which are considered as the absolute maximum conditions) are within the capacity of the condenser even at the low figure of 20 for conductivity. These figures are based on low temperature carbonisation, but will hold for higher temperatures as less liquid products will be formed as the gas yield increases

PRECIPITATOR.

Owing to the success of the small precipitators described in Chapter 4, it was decided to use an electrostatic precipitator for tar mist dispersal in this unit. The design is shown in the drawing R5.

It consists simply of a length of 2 in. pipe fitted for entrance and exit of the gas, and for running off the tar. The central electrode is suspended from an insulating bush. This is shown in the drawing as being made of ebonite. It is possible that this material might absorb tar, and soften. For this reason it might be better to use another material, such as fibre.

The important feature in the design of this insulator is the prevention of surface leakage, as the high tension current could leak very easily from the electrode along the film of moist tar which will certainly collect on the surface. With this in view the design shown in drawing R5a is proposed. In this the leakage path is increased in length as much as possible by

(a) carrying the insulation well down the electrode and

(b) grooving the surfaces.

As the voltage used is nearing the sparking voltage, also, all corners are rounded, to prevent point discharges.

The electrode itself would consist of a length of ¼ in. diameter wire of star section. This, however, is only a suggestion, as no information is available as to the behaviour of small precipitators. Certainly, on the small ones already described, the character of the electrode proved of great importance. Some interesting results could be obtained by investigating the efficiency of various electrodes.

Inquiries as to the dimensions and voltage required showed that some electrical firms dealing in large precipitators considered that one on such a small scale was impossible. In view of successful experience gained in this laboratory with the small precipitator it was decided to proceed with the design. The precipitators shown on page 106 had a diameter of 1 in. and the voltage was of the order of 2,000 volts. At 2 in. diameter, the voltage required would lie between 5,000 and 10,000 volts.

Accordingly, it is proposed to supply the high tension voltage by means of a transformer fed from the laboratory 220 volt 50 cycle supply, the output being rectified by Westinghouse metal rectifiers. The elctrical circuit is shown in drawing R5a and is that suggested by Messrs Westinghouse. By varying the input as shown, the output voltage can be varied between 5,000 and 10,000 volts to give efficient precipitation without sparking.

## AMMONIA SCRUBBER.

This is shown in drawing R5 and is of normal design. Its principal function would be to scrub out the ammonia prior to the passage of the gas through the active carbon scrubber. Its washing action would not be needed, as all dust and mist would be removed by the precipitator.

As the scrubbing is very efficient, the dimensions shown are well above those required theoretically. The dimensions are chosen principally for ease of fabrication and operation.

## PURIFIER.

The scrubbed gas then passes through a purifier containing iron oxide. This is shown in drawing R7. It consists of a circular box containing three trays. These are made of 20 mesh wire gauze, supported on frames of $\frac{1}{4}$in. diameter wire. The purifier is recharged by taking off the cover plate, when the trays can be lifted out. The gas off-take, shown on the cover, might be more effectively situated in the side of the box, just under the flange. The cover could then be taken off without disturbing pipe connections. This is not an important point, as the purifier will not need recharging very often.

In a commercial unit, for a gas containing 63 grains of sulphur per 100 cu.ft. of gas a volume of 35.5 cu.in. of oxide was allowed per cu.ft. of gas per hour. Allowing a sulphur content of four times this, at a rate of 5 cu.ft. per hour, the volume of oxide required would be 710.0 cu.in. The volume of oxide in the purifier shown is 1060 cu.in.

## ACTIVE CARBON SCRUBBER.

This is intended to be operated in the normal manner. During the carbonisation, the gas enters via cock A, passes up through the carbon, and out through cock B. These are shown in drawing R5.

To recover the spirit, cocks A and B are closed, and the cock leading to the condenser is opened. Live steam is then admitted through the steam pipe S. After steaming the carbon is dried by means of the steam coil, in a current of nitrogen. The nitrogen could enter as shown, the moist gas leaving through the main gas outlet. This would be possible only when steaming after a run. If two scrubbers were being used alternately, the nitrogen would best be added through the top of the scrubber, at N. This is the position shown in the layout sketch (R8). In this case the nitrogen passes down through the carbon and out through the condenser.

The body of the scrubber would of course be lagged. This is not shown in the drawing.

The maximum quantity of spirit obtained is about 2 gallons/ton. One charge would therefore give 50 cc. of spirit - say 50 gms.

Allowing an absorptive capacity of 5 gms. spirit per 100 gms. active carbon, one charge would require 1,000 gms. carbon. At an apparent density of 0.5, this requires a space of 2000 cc. i.e. 122 cu.in.

The volume in the scrubber shown is

$$\frac{6 \times 6^2 \times \pi}{4} = 170 \text{ cu.in.}$$

Less space occupied by coil, which

$$= \frac{4 \times \pi \times \cancel{4} \times \pi \times .5^2}{\cancel{4}}$$

$$= 9.85 \text{ cu.in.}$$

Giving volume of carbon

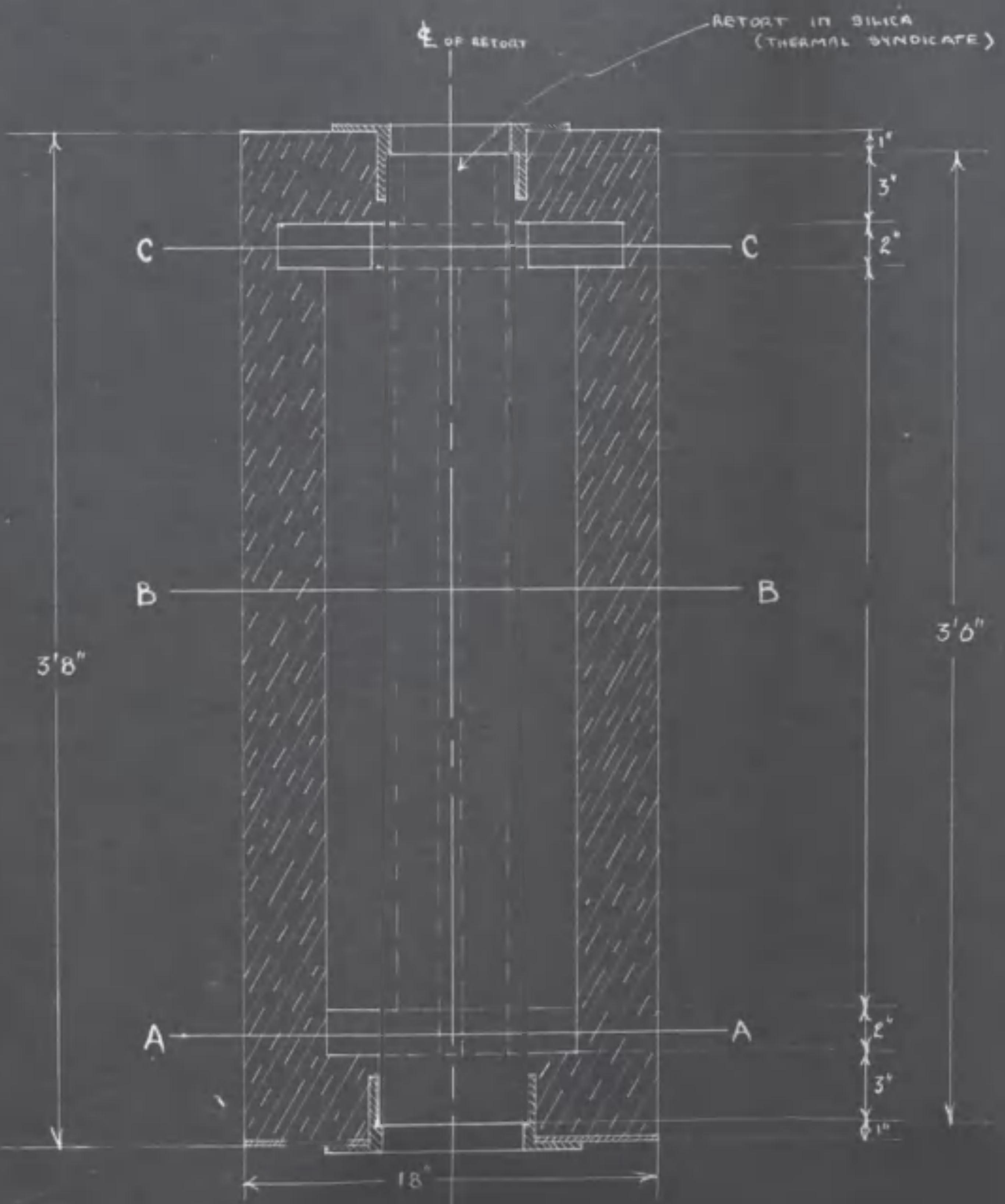
$$= 170 - 9.85$$
$$= 160 \text{ cu.in.}$$

Which gives a reasonable margin over these maximum conditions.

## ASSEMBLY.

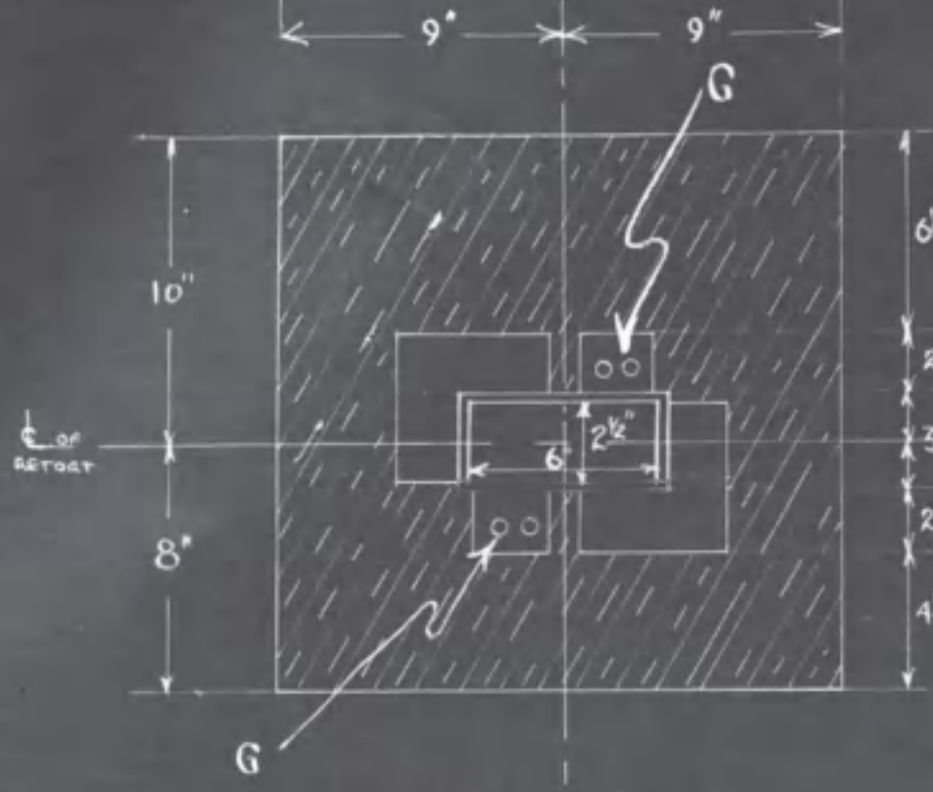
The complete assembly is shown in the drawing R8. The products of carbonisation pass from the retort through the air condenser, the water-cooled condenser, and the precipitator. They then enter an exhauster, and are forced through the ammonia scrubber, oxide purifier, and one or other of the active scrubbers. The gas then passes through a meter, and is split up at an orifice plate, where a fraction of possibly one tenth is led to a holder for analysis, the remainder being burnt as waste.

It is proposed to erect this unit during the winter term of 1939.



SECTN. ELEVATION THRO' LONG. AXIS OF RETORT

SECTN. ELEVATION THRO' XX



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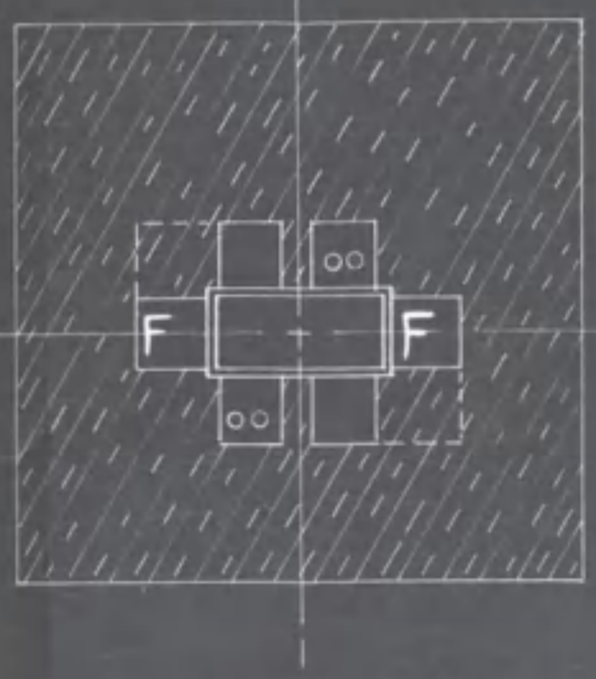
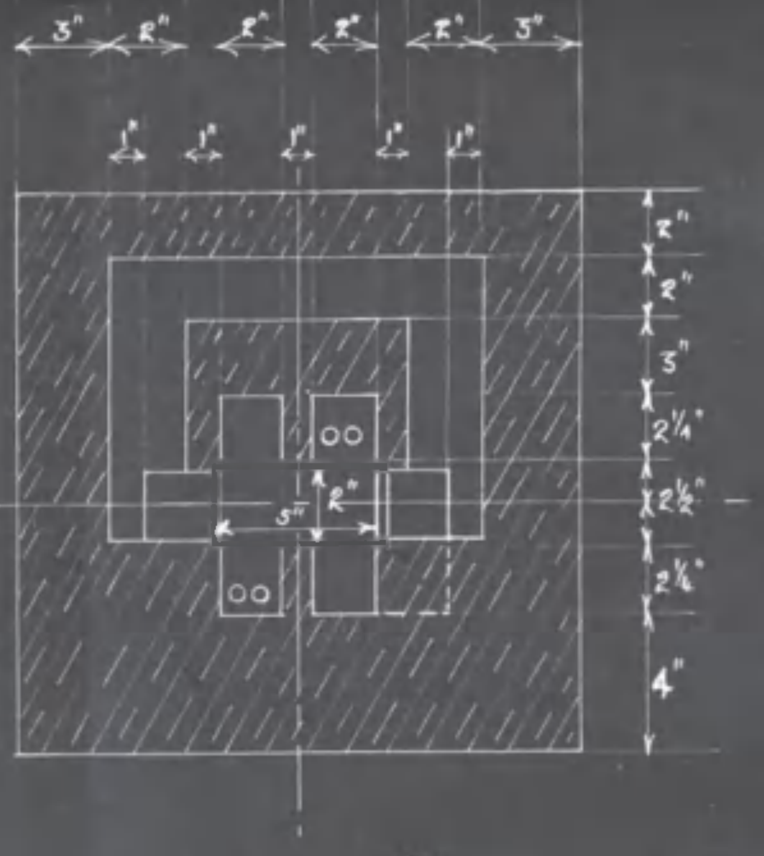


SECTN. PLAN BB



SECTN. PLAN CC

## ~ EXPERIMENTAL RETORT ~

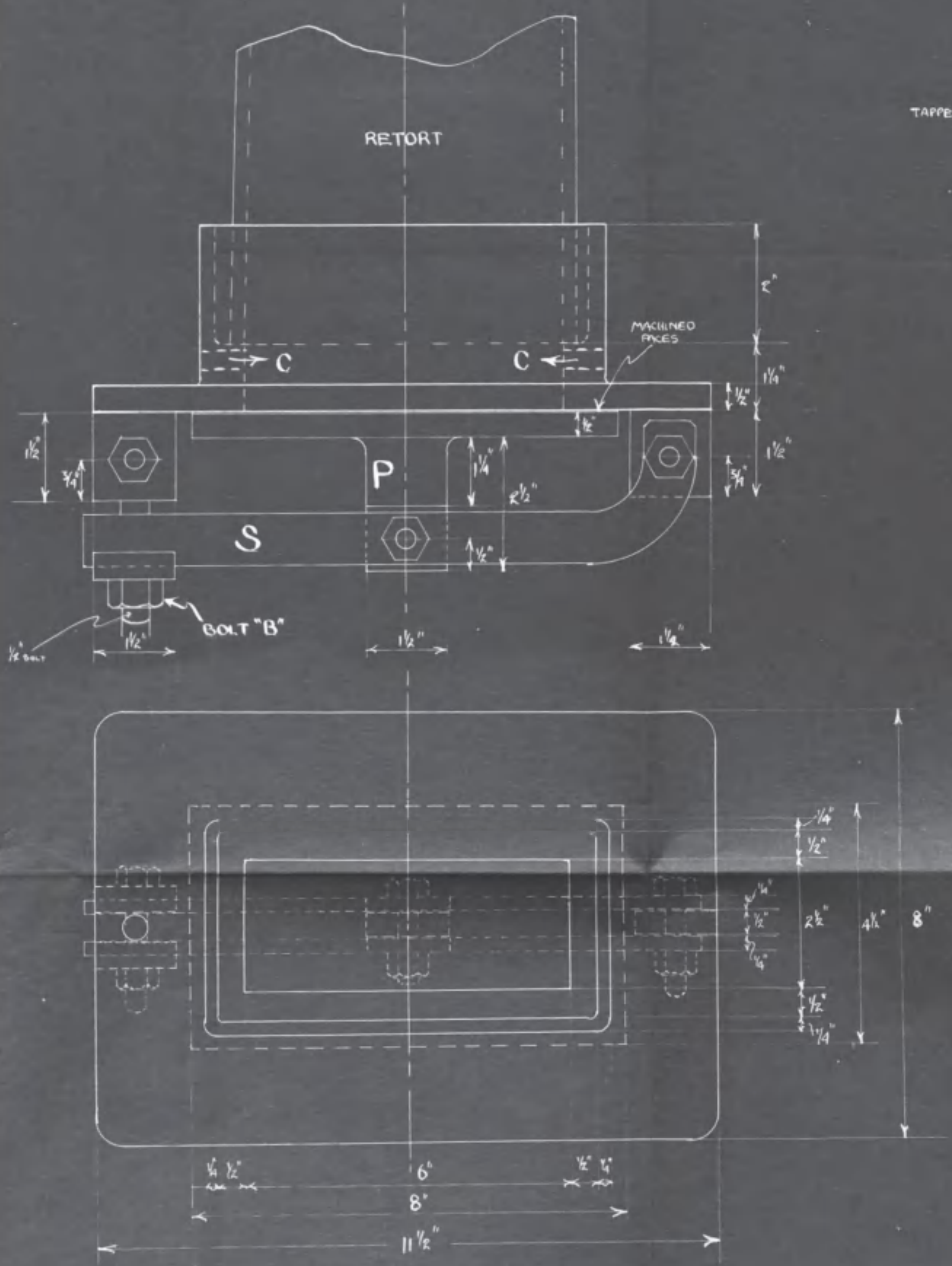
ARRANGEMENT OF BRICKWORK

(METAL CASING AND OTHER METALWORK OMITTED)

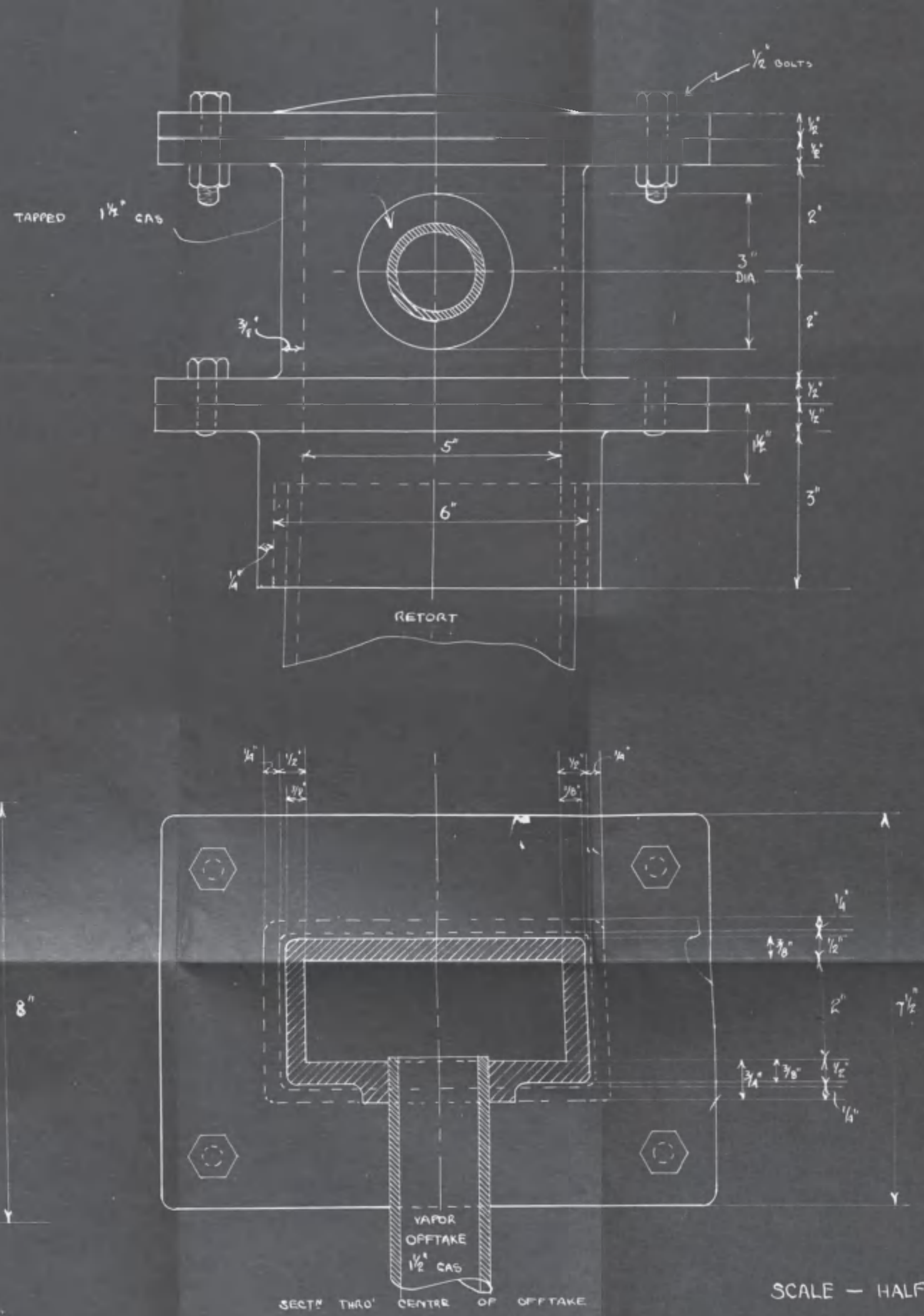
SCALE —

TWO INCHES = ONE FOOT

| | | |
|---|---|---|
| ROYAL TECHNICAL COLLEGE<br>TECHNICAL CHEMISTRY DEPARTMENT. | | |
| DRAWING No | R-2 | DATE 29TH JUNE '36 |
| DRAWN BY | David L. Wilson. | |
| CHECKED | | |



- DISCHARGE DOOR AND END PIECE -

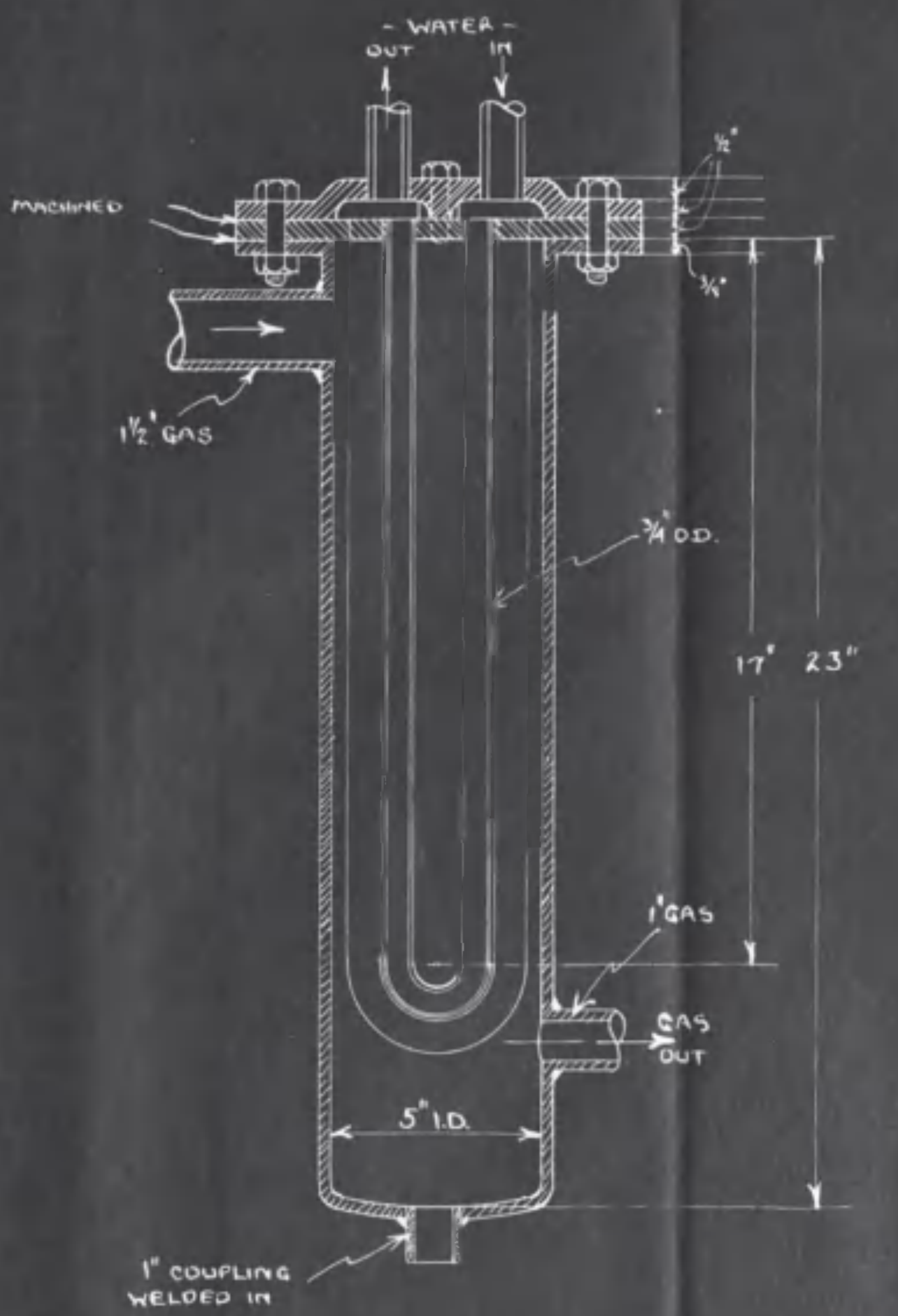


SCALE — HALF SIZE

- TOP-PIECE AND OFFTAKE HEADER -

GENERAL ARRANGEMENTS OF FEED AND DISCHARGE GEAR

| ROYAL TECHNICAL COLLEGE<br>TECHNICAL CHEMISTRY DEPT | |
|---|---|
| DRAWING No. R.3 | DATE 29TH JUNE 1938 |
| DRAWN BY | Wilson |
| CHECKED | |



SECTION

PLAN

COVER OF HEADER REMOVED

# - CONDENSER -

FOR CARBONISING UNIT

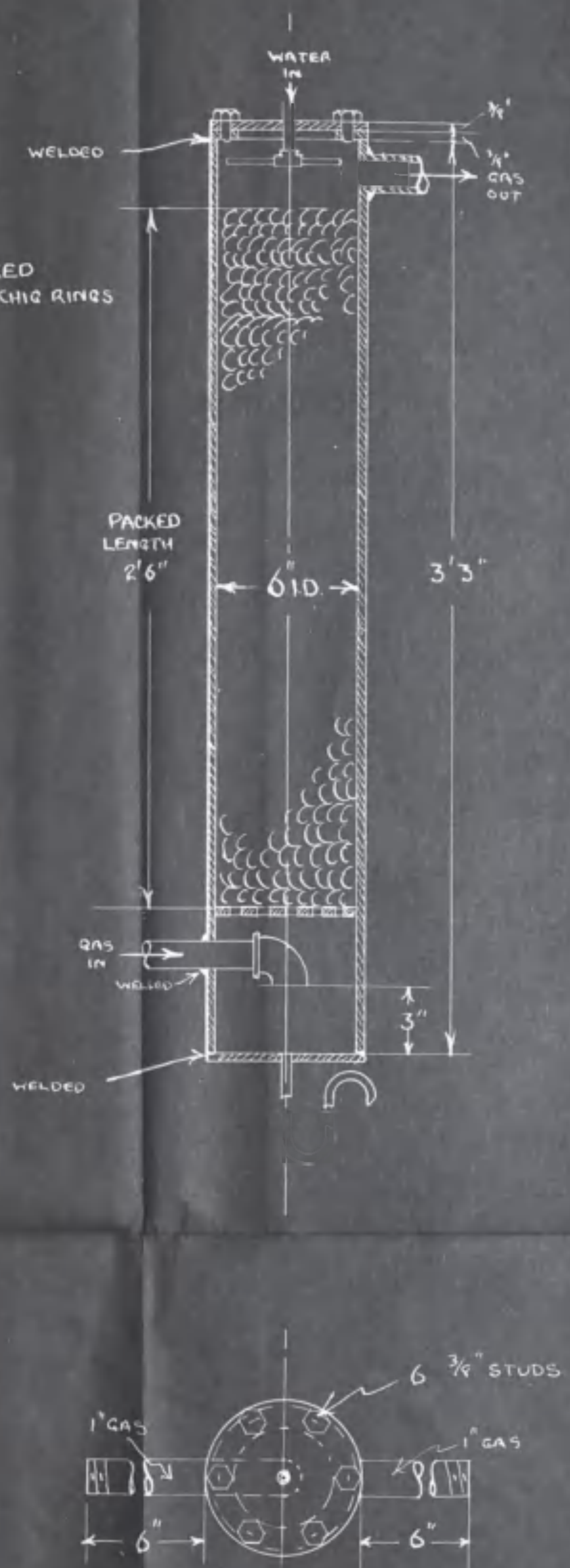
SCALE - 3" = 1 FOOT

| | |
|---|---|
| ROYAL TECHNICAL COLLEGE<br>TECHNICAL CHEMISTRY DEPARTMENT | |
| DRAWING No. R 4 | DATE 1ST JULY 1938 |
| DRAWN BY | David L. Wilson |
| CHECKED | |

## PRECIPITATOR

FOR CARBONISING UNIT

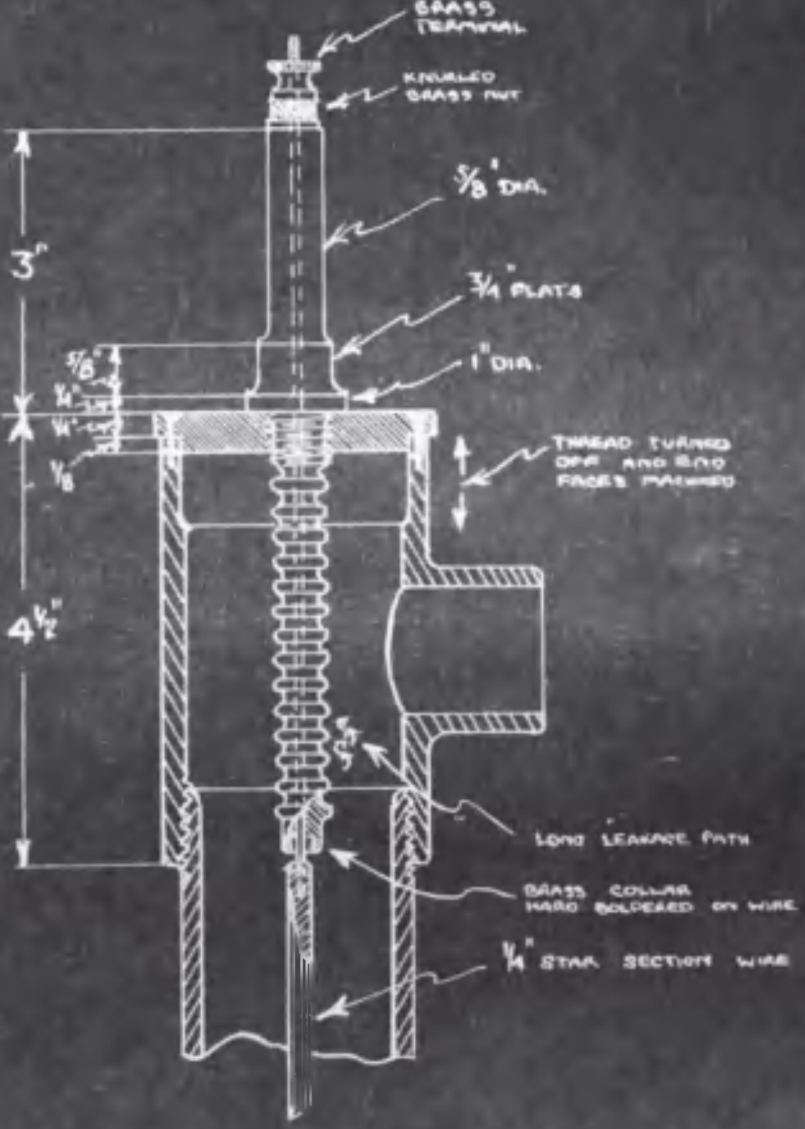
SCALE - 2" = 1 FOOT



## AMMONIA SCRUBBER

FOR CARBONISING UNIT

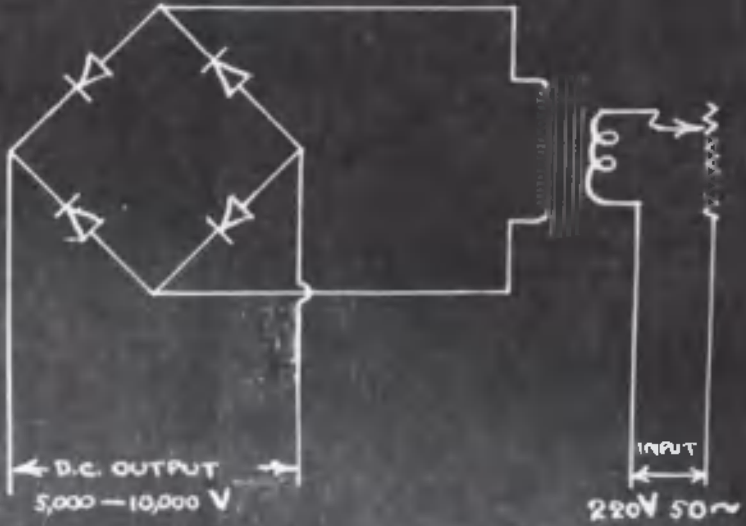
| ROYAL TECHNICAL COLLEGE<br>TECHNICAL CHEMISTRY DEPARTMENT | |
|---|---|
| DRAWING No. R.5 | DATE 1ST JULY 1938 |
| DRAWN BY | David A. Wilson. |
| CHECKED | |



- SECTION OF PRECIPITATOR HEAD -

CENTRAL ELECTRODE
IN PART SECTION

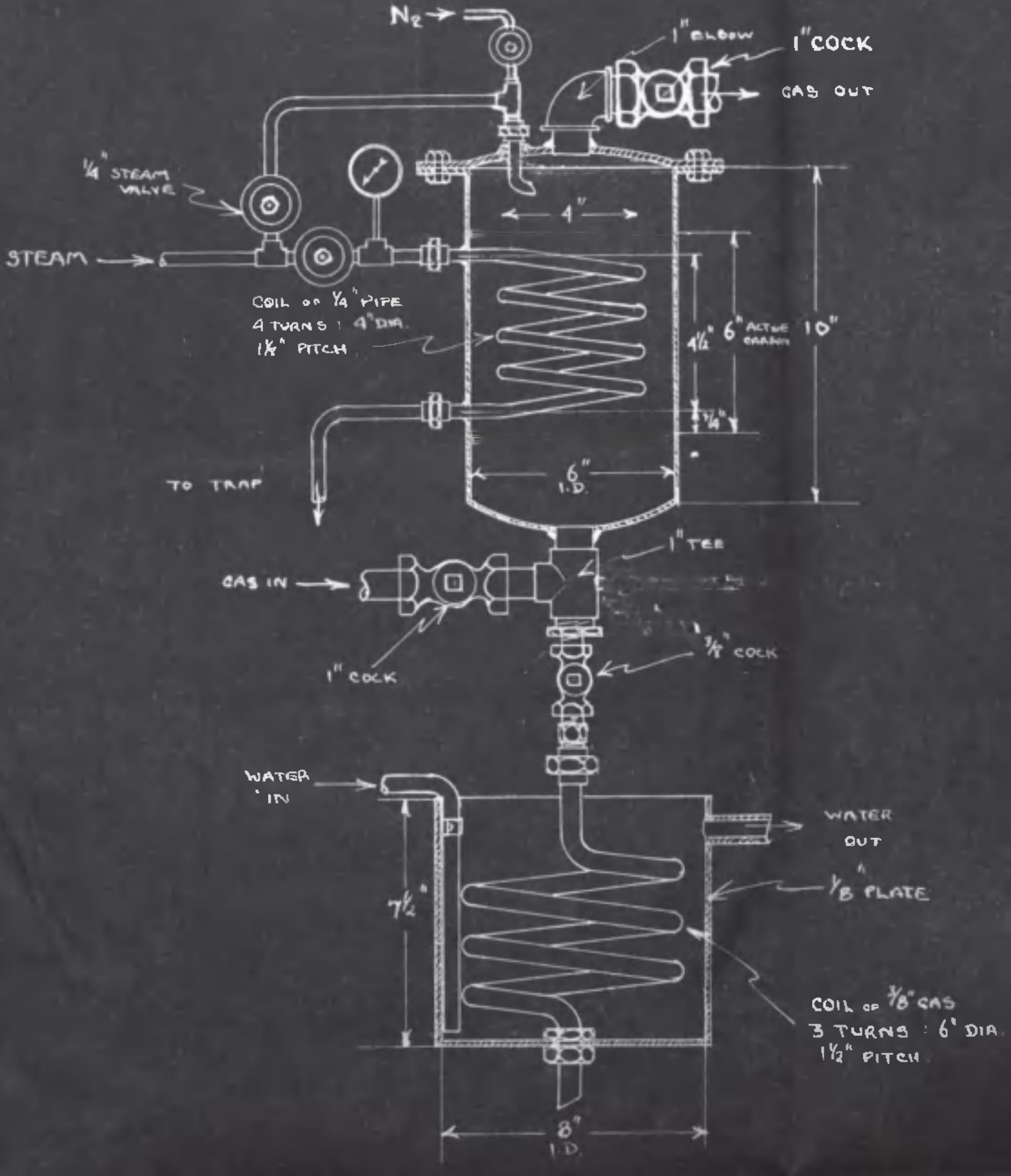
SCALE — HALF SIZE.



HIGH VOLTAGE SUPPLY CIRCUIT

USING METAL OXIDE RECTIFIER

| | | | |
|---|---|---|---|
| ROYAL TECHNICAL COLLEGE, GLASGOW.<br>TECHNICAL CHEMISTRY DEPARTMENT. | | | |
| DRAWING No. | RSA. | DATE. | SEPT. 1939 |
| DRAWN BY | David L. Adam. | | |
| CHECKED BY | | | |



## ACTIVE CARBON SCRUBBER

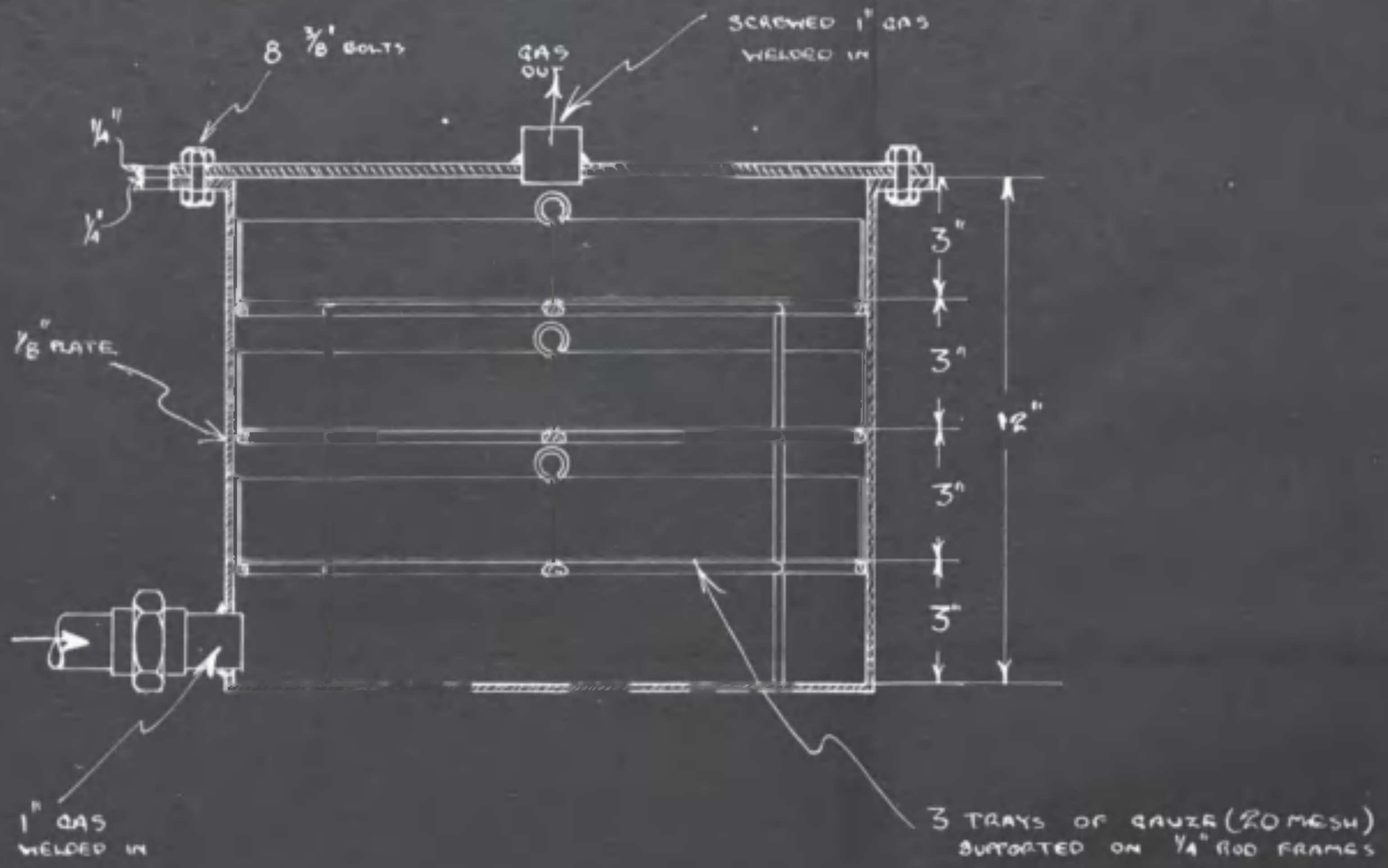
FOR

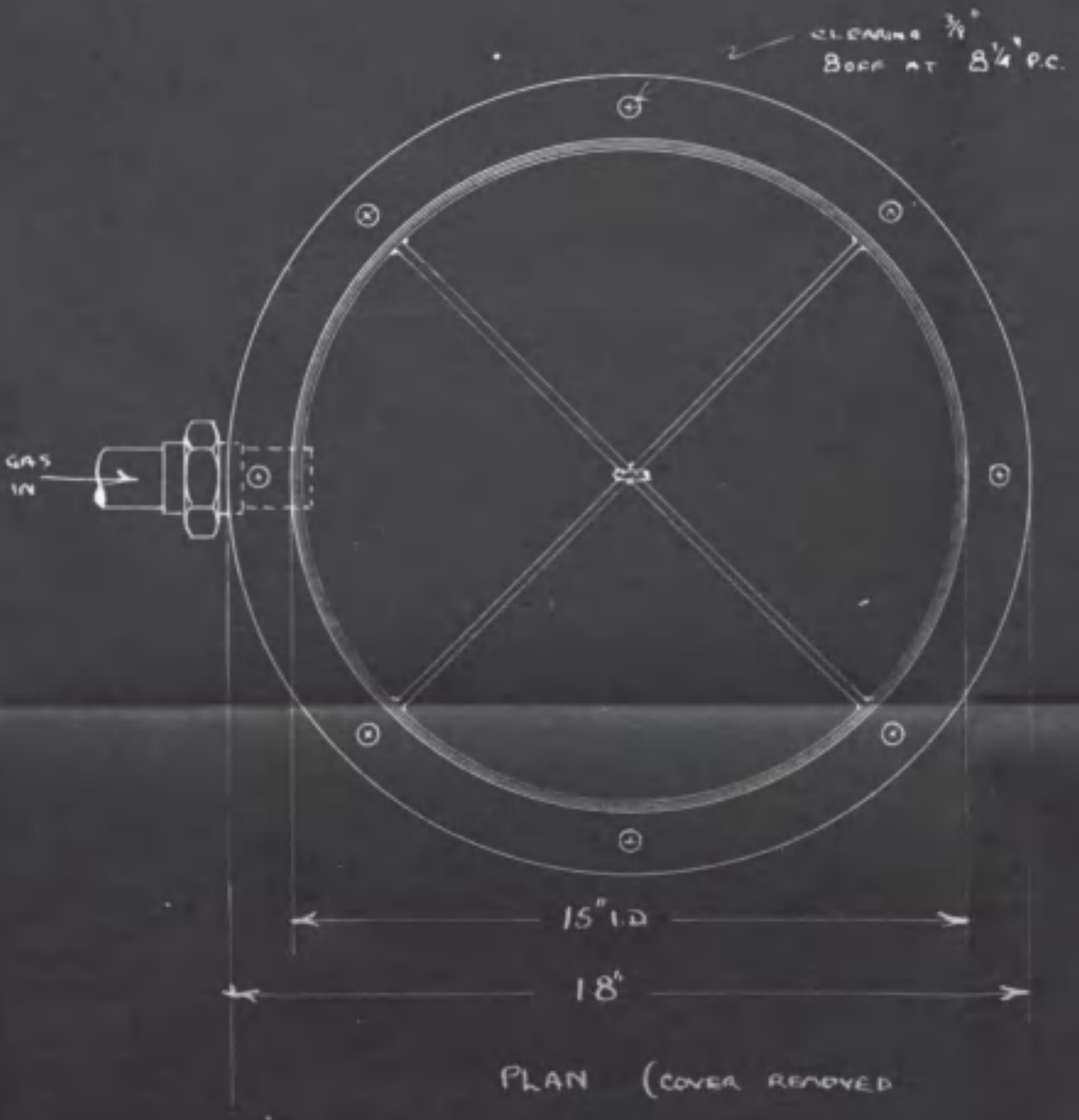
## CARBONISING UNIT

SCALE – 3" = 1 FOOT

ROYAL TECHNICAL COLLEGE
TECHNICAL CHEMISTRY DEPARTMENT

| | | |
|---|---|---|
| DRAWING NO | R 6 | DATE 6TH JULY 1938 |
| DRAW BY | David L. Wilson | |
| CHECKED BY | J. R[illegible] | |



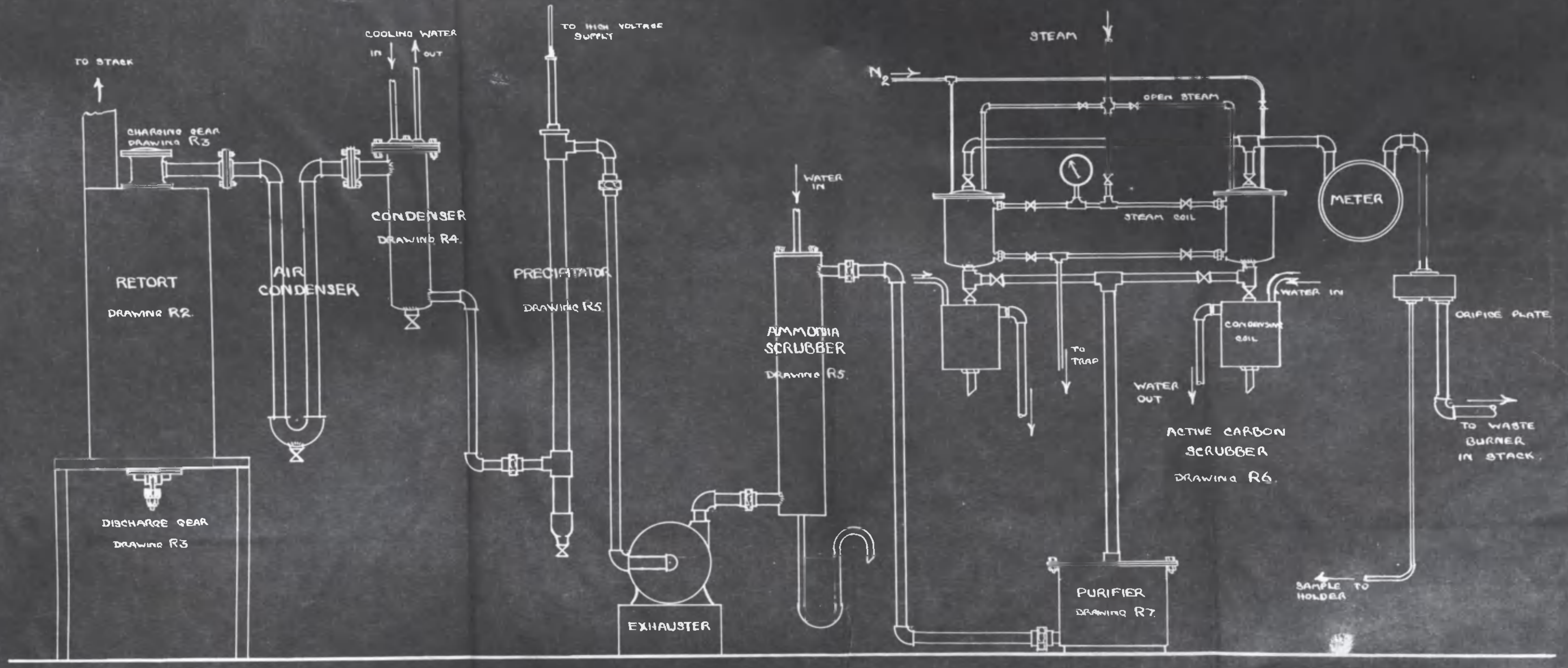


PLAN (COVER REMOVED

## ~ PURIFIER ~
FOR
CARBONISING UNIT

SCALE – 3" = 1 FOOT

| | |
|---|---|
| ROYAL TECHNICAL COLLEGE<br>TECHNICAL CHEMISTRY DEPARTMENT | |
| DRAWING NO. 2. Y | DATE 6TH JULY 1938 |
| DRAWN BY | David L [illegible] |
| CHECKED BY | |



## - ARRANGEMENT OF CARBONISING UNIT -

SCALE — ONE INCH TO THE FOOT.

| | | | |
|---|---|---|---|
| ROYAL TECHNICAL COLLEGE, GLASGOW. | | | |
| TECHNICAL CHEMISTRY DEPARTMENT | | | |
| DRAWING NO. | R 8 | DATE | SEPT. 1930 |
| DRAWN BY | David M Wilson | | |
| CHECKED BY | | | |