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HOWARD ZUMIN
VIA AIR

MINES BRANCH INVESTIGATIONS OF
FUELS AND FUEL TESTING, 1923

The investigations described hereunder have been carried out under the direction of B. F. Haanel, Chief of Division of Fuels and Fuel Testing, and the immediate supervision of R. E. Gilmore, Superintendent of Fuel Testing Laboratories.

I

THE CARBONIZATION OF LIGNITE AND SUB-
BITUMINOUS COALS

Harold Kohl

INTRODUCTORY

Previous Mines Branch Summary Reports¹ contain carbonization results on lignites from both Saskatchewan and Alberta. The brown lignites from Saskatchewan were experimented with on both the small and large laboratory scale, while only small scale tests were made on the Alberta lignites. This report describes carbonization experiments with 4½ to 6½-pound samples carried out on five different Alberta coals.

The purpose of these carbonization experiments was to obtain information on the comparative yield and value of the carbonized residue or char as the main product, and of the gas, tar oils, ammonia, etc., as by-products. With this information the merits of the carbonization of these Alberta coals, with or without the subsequent briquetting of the char, may be discussed. The lignite coals from this province are known as black lignites in contrast with the brown Saskatchewan lignite, and the terms "black lignite" and "sub-bituminous" are often used as interchangeable.

The general characteristics of the lower grade Canadian coals vary considerably. The so-called black lignites of Alberta may be classed as low, medium and high grade depending mainly on the moisture content of the coal as mined. All these three grades are, however, superior to the brown Saskatchewan lignites, which when freshly mined contain over 30 per cent moisture. By low-grade Alberta lignites are meant those coals ranging from 20 to 25 per cent moisture and which are termed non-storage coals. They are similar to the Saskatchewan lignites in that they will disintegrate or slack on exposure, although not to the same extent. The medium-grade Alberta lignites ranging from 15 to 20 per cent moisture

¹ Summary Report of the Mines Branch, 1918, pages 87 to 105; 1919, pages 30 to 37; 1920, pages 39 to 42, and 1921, pages 205 to 225.

as mined are known in Alberta as limited storage coals, which apparently means that although they will crumble on exposure to extreme weather conditions they will not deteriorate if stored under cover after being mined. The high-grade lignites, showing from 10 to 15 per cent moisture when freshly mined, may be termed storage coals, and in this respect are similar to the sub-bituminous, by which name they are often called.

Without going into the details of chemical classification the sub-bituminous coals for the purpose of this report may be defined as those non-coking (bituminous) coals having less than 10 per cent moisture when freshly mined and having a hygroscopic moisture in the neighbourhood of 6 to 7 per cent when air dried to constant weight in an atmosphere of approximately 60 per cent humidity. They may be distinguished from the true bituminous coals by their non-coking qualities and also by their calorific value on the "unit coal" or dry ash-free basis. The dividing line between the bituminous coals proper and the sub-bituminous has been drawn at 14,000 B.T.U. per pound on the unit coal basis which line is quite serviceable. On the other hand the Alberta sub-bituminous coals may be differentiated from the high-grade lignites in that they are usually found in pitched seams whereas the latter coals are mined from flat seams.

The Alberta lignites and sub-bituminous coals are high in oxygen content and are known as domestic or household coals. They are not smokeless in the sense that they will burn with no smoke the same as anthracite or coke, but when compared with bituminous high volatile matter steam coals the smoke coming out of the chimneys is not noticeably black and disagreeable. Instead of the dense black smoke from ordinary soft coals the smoke from the sub-bituminous coals may be described as white or blue resembling that from peat or wood.

The brown lignites when freshly mined can be used either for household or steam-raising purposes and find general use locally. When shipped and stored under cover there is no apparent reason other than economic why they should not find general use. The cost of mining, the necessity of paying freight on an excessive water content, and finally the value of the fuel on a calorific value basis are apparently the economic reasons why the brown lignites of Saskatchewan do not find more general use. The same comments also apply to Alberta lignites, but of course to a much less and varying degree. The lower grade black lignites for the above reasons may not be expected to find as general a use as the higher grades.

For the lower grade Canadian coals the feasibility of carbonizing and briquetting has received much attention. The aim of the carbonization process would be to obtain a carbonized residue or char with a heating value approaching the high-grade fuels, and the purpose of briquetting would be to improve the general handling, storage and burning qualities so that the manufactured fuel may compare favourably with high-grade household fuels such as anthracite, by-product coke, or anthracite briquettes. If the brown lignites are amenable to carbonizing and briquetting it does not necessarily follow that the higher grade black lignites and sub-bituminous coals should be so treated. It would seem that the importance of carbonizing (and briquetting) the different grades of black lignites and sub-bituminous coals varies indirectly with the degree to which they can be used in their raw state.

EXPERIMENTAL

Carbonization runs were made on five different coals at low temperature and on three of these at high temperature. All five coals were from Alberta, a list of which is as follows:—

| No. | Marked | Description | Lab. No. |
|-----|-----------|--|----------|
| A | Cardiff | From Cardiff Collieries, Cardiff, Alta..... | 1456 |
| B | Rose Deer | From Rose Deer Coal Mfg. Co., Wayne, Alta..... | 2257 |
| C | Taber | From Canada West Coal Co., Taber, Alta..... | 1824 |
| D | Coalspur | From McLeod River Hard Coal Co., Coalspur, Alta..... | 2621 |
| E | Harlech | From Harlech Coal Co., Harlech, Alta..... | 2255 |

The Cardiff and Taber coals carbonized were the same as used in the small scale experiments reported in the Mines Branch Summary Report for 1919 and 1921 respectively, the sample in the meantime having been kept in air-tight containers. The Rose Deer and Harlech samples used were from car lots shipped to Ottawa in April 1923 as demonstration Alberta Domestic fuels. The Coalspur coal used was from a fresh hundred-pound lump sample.

The Cardiff Collieries are in the Edmonton area; the Rose Deer coal is from the lower seam of the Drumheller area, and the Taber sample is from the Taber—Bow Island area in southern Alberta. All these three samples are lignites, and may be graded according to the moisture content either on the freshly mined or air-dried basis. The Coalspur coal from the Coalspur or Yellowhead area and the Harlech coal from the Saunders area are known as sub-bituminous. They are similar to the high-grade black lignites in that they are non-coking, and in this respect they may be differentiated from the true bituminous, which are practically all coking coals.

The low temperature carbonization runs were made in the lead bath temperature control electric furnace described in detail on pages 99 to 101, Mines Branch Summary Report for 1918. The retort, condenser and tar fog scrubber used were the same as described in the above reference. Duplicate runs were made on 3,000-gramme samples of each coal, and a weight balance made for each run. The maximum temperature at which the coal was carbonized was in the neighbourhood of 600°C. The temperature of the lead bath during the carbonization period ranged from about 400°C., and the time taken to reach the above-mentioned maximum temperature averaged 4½ hours.

The liquid distillate from the duplicate runs were combined and by means of gravity settling after warming followed by recovery of the last traces of oil by ether, a separation of the tar oil from the aqueous distillate was made. The carbonized residue or char, gas and the liquid products were analysed according to standard laboratory practice. In the high temperature runs the furnace used was the same as employed in the fusibility of ash determinations according to the U.S. Bureau of Mines standard method—namely a No. 3 melters furnace. A retort 10 inches long made from a 5-inch diameter iron pipe was fitted with a 2-inch gas off-take pipe, which in turn was connected to the same gas condensing and scrubber train as used in the low temperature runs. 2,000-gramme samples were used and the heating continued until the temperature in the coal reached approximately 950°C.

The low temperature carbonization results for the five coals used are given in Tables I to VI inclusive under the numbers A, B, C, D and E. In table I the analyses are given on both the "as used" and "dry basis." Table II is a summary of the yields and partial analyses of the char, gas, dry tar, and aqueous liquor products. Table III is a weight balance, and tables IV and V are the analyses of tar and gas respectively. In table VI is given a thermal balance where the calorific values of the products are given as percentages of the heating value of the raw coal carbonized.

Tables VII and VIII are the summarized results of the three coals, viz.: B, D, and E carbonized at high temperature. Table VII is a summary of the yields and analyses of the char and other products, table VIII a combined weight and thermal balance sheet.

Discussion of Results

The five coals carbonized may be graded or arranged according to their moisture contents, which as shown in table I ranged from 21.4 per cent in A to 6.7 per cent in coal E. The coals used had been exposed either in transit, or in storage (under cover) so that the moisture contents indicated are for air-dried lump coal rather than for freshly mined. Coals A, B, and C, as mentioned above, are termed black lignites and D and E sub-bituminous. The calorific value of the coal as charged, which of course varied indirectly as the moisture content, is also a means of grading these coals. Still another method of grading is according to calorific value on the dry basis, which in turn will correspond to the grading on the dry ash-free basis as the ash contents are fairly uniform. While the calorific value of the three black lignites varied from 11,040 to 11,910 B.T.U. per pound their average is roughly 11,500 as compared with an average of nearly 12,500 for the two sub-bituminous coals.

Lignites and sub-bituminous coals are non-coking and when carbonized yield an incoherent residue or char in contrast to the coherent coke residue resulting from true bituminous coals. For the latter coals the burning qualities in respect to smoke are improved by coking and the calorific value per pound of the coke may or may not be greater than that of the raw coal carbonized. For the sub-bituminous and lignite coals carbonization does not improve the burning qualities in respect to smoke to the same extent as with the bituminous coals, but the calorific value per pound of the char is materially increased over that of the raw coal carbonized. In the following table is a comparison of this increase for the coals carbonized.

| Coals | A | B | C | D | E |
|--|---------------|----------|--------|----------------|----------|
| Calorific value of coals used— | | | | | |
| B.T.U. per lb. as charged..... | 8,680 | 9,860 | 10,480 | 11,280 | 11,750 |
| B.T.U. per lb. dry basis..... | 11,040 | 11,800 | 11,910 | 12,240 | 12,590 |
| Calorific value of char— | | | | | |
| Low temperature tests..... | 12,750 | 13,320 | 13,040 | 12,920 | 13,430 |
| High temperature tests..... | | (12,810) | | (12,520) | (12,855) |
| Increase in calorific value of low temperature char— | | | | | |
| (a) over that of coal charged.....% | 47.0 | 35.5 | 24.5 | 14.5 | 14.5 |
| (b) over that of dry coal.....% | 15.5 | 13.0 | 8.5 | 5.5 | 6.5 |
| Class of coal..... | Black lignite | | | Sub-bituminous | |

For the low temperature carbonization tests the maximum temperature was controlled in the neighbourhood of 600°C. At approximately this temperature the char reached its maximum calorific value per unit weight as indicated by small scale experiments already referred to. Carbonization at higher temperature gives a lower yield of char and a higher gas yield. The heating value per pound of the char from the high temperature tests was appreciably lower than that for the low temperature char as shown in the above table.

A study of the distribution of the heat of combustions of the different products in the high temperature tests as compared with that of low temperature tests is worth while and may be given as follows:—

| Thermal value of products as per cent of that in the raw coal as charged | B Black lignite | | Average of D and E Sub-bituminous | |
|--|--------------------|---------------------|--------------------------------------|---------------------|
| | Low temperature | High temperature | Low temperature | High temperature |
| | Char.....% | 78.9 | 75.3 | 81.3 |
| Tar.....% | 5.1 | 3.1 | 6.6 | 3.6 |
| Gas.....% | 11.5 | 20.1 | 7.8 | 20.5 |
| Loss.....% | 4.4 | 1.5 | 4.3 | 7.8 |

In the high temperature tests the coal was not charged into a hot retort similar to high temperature coking practice. The quality of the tar therefore will not be wholly typical of high temperature tar. The yield was however materially lower in the high temperature runs than in those at low temperature. A comparison of the tar oils may be made as follows:—

Comparison of Tar Oils at Low and High Temperature

| — | B Black lignite | | | | E Sub-bituminous | | | |
|--|--------------------|------------|-----------|------------|---------------------|------------|-----------|------------|
| | Low | | High | | Low | | High | |
| | % Vol. | Sp. Gr. | % Vol. | Sp. Gr. | % Vol. | Sp. Gr. | % Vol. | Sp. Gr. |
| Yield of dry tar oils (Imp. gals. per ton) | 6.2 | | 3.8 | | 8.6 | | 4.1 | |
| Crude tar oils..... | 25 | 0.994 | 27 | 0.983 | 20 | 1.015 | 22 | 1.045 |
| Fraction 200-300°C..... | 35 | 0.960 | 36 | 0.974 | 42 | 0.996 | 33 | 0.917 |
| Fraction 300-350°C..... | 17 | 0.980 | 19 | 1.014 | 20 | 1.018 | 18 | 1.053 |
| % Wt. (pitch)—350° up..... | 21 | | 17 | | 17 | | 26 | |
| Imp. gals. per ton of fraction to 200° C..... | 1.6 | | 1.0 | | 1.7 | | 0.9 | |
| Imp. gals. " " 200-300° C..... | 2.2 | | 1.4 | | 3.6 | | 1.3 | |
| Imp. gals. " " 300-350° C..... | 1.0 | | 0.7 | | 1.7 | | 0.7 | |
| Lbs. of pitch 350° C. up..... | 13 | | 6 | | 15 | | 11 | |

The purpose of the above table is to show the low yields of crude tar oils and their fractions resulting from high temperature carbonization in comparison with those in the low temperature tests rather than to show the difference in the nature of the tar. The results are to be considered as comparative only, and any detailed interpretation should not

be attempted, as different results would more than likely be obtained by charging the coal into a hot retort, and by carbonizing on a larger scale. Averaging the tar yields first from the lignites B and C and second from the sub-bituminous coals D and E, and comparing these averages with the tar yields from peat and brown lignites on the one hand and with bituminous coals on the other the following table is of interest:—

| Fuel | Moisture content | Low temperature carbonization | |
|-----------------------------------|------------------|-------------------------------|--------------------|
| | | Sp. Gr. of tar | Imp. gals. per ton |
| Peat (air-dried)..... | 25.0% | 0.97 | 14.0 |
| Brown lignites (Sask.)..... | 33.0 | 1.00 | 5.5 |
| Black lignites (Alta.)..... | 14.5 | 1.00 | 6.0 |
| Sub-bituminous coals (Alta.)..... | 7.5 | 1.00 | 9.0 |
| Bituminous coals (Ill.)..... | 7.0 | 1.07 | 15.0 |
| Bituminous coals (English)..... | 5.0 | 1.04 | 22.0 |

The yield of tar for peat is that given in Mines Branch Report for 1922, page 204, and the yield from brown lignites is the average of coals A to E inclusively as given on page 105, Summary Report for 1918. The 15-gallon tar yield was on 6 to 8-pound samples of (poor coking) Illinois coal—see University of Illinois Bulletins 60 and 79—and the 22-gallon yield for the English coals was calculated from the results given on page 33—Report of the Fuel Research Board for 1920 and 1921. It will be noticed that in the latter reference the yields of tar from the large scale horizontal retorts was only about 60 per cent of the above laboratory yield. The tar results, therefore, in this report must be interpreted only as that obtainable on small laboratory scale. The yield of tar at low temperature on a commercial scale from these lignite and sub-bituminous coals would be much lower than indicated and would be still lower if carbonized commercially at high temperatures.

The carbonized residue or char was not examined as to suitability for briquetting. Future work on the samples of char from both the low and high temperature tests is planned. A study of the structure of the char under hand lens and microscope, the determination of porosity, the apparent and true specific gravity along with briquetting experiments to determine the amount of different kinds of binder necessary to make a good domestic briquette in comparison with anthracite fines on the one hand and with char from brown lignites on the other, is to be included in the work still to be carried out on the char from the five coals reported.

SUMMARY

1. Laboratory carbonization tests have been carried out on Alberta lignite and sub-bituminous coals at both high and low temperature. For the low temperature tests 3,000-gramme samples were carbonized at approximately 600°C., while in the high temperature tests on 2,000-gramme samples the carbonizing temperature used was 900°C. to 950°C. The results, which are given in tables, include: analyses of coals; summary of carbonization tests; analyses of gas and tar oils along with weight and thermal balances.

2. The Alberta lignites may be designated black lignites in contrast to the brown lignites of Saskatchewan. The terms black lignites and sub-bituminous are often used as interchangeable but for the purpose of this report they have been defined and treated as two different classes of coals. The three lignites and two sub-bituminous coals (here reported) may be graded according to (a) the moisture content either on the freshly mined or air-dried basis, and (b) calorific value on the freshly mined, air-dried or dry ash-free basis.

3. The black lignites and sub-bituminous coals are termed domestic fuels, and are used extensively for this purpose. The lignites are graded non-storage or limited storage coals depending on the moisture content as mined, while the low moisture black lignites along with the sub-bituminous coals are considered of storage quality, the same as bituminous coals in general. When carbonized, an incoherent char is produced which requires to be briquetted to improve its burning and handling qualities as a household fuel. For the lump coal (irrespective of the liquid and gas by-products) the necessity and desirability of carbonizing would seem to bear an indirect relation to their use in the raw state.

4. The increase in calorific value per unit weight of the char over that of the raw coal charged was roughly 47, 35, and 25 per cent respectively for the three lignites tested and 14.5 per cent each for the two sub-bituminous coals. Brown lignites give an average increase of about 75 per cent. On the dry basis the corresponding increase is 18 per cent for brown lignites; 15.5, 13.0 and 8.5 per cent respectively for the three black lignites and 6.0 per cent average for the two sub-bituminous coals.

5. The yield of char was roughly 54, 58, and 63 per cent of the coal charged respectively for the three coals carbonized at low temperature, compared with 68 and 72 per cent for the two sub-bituminous coals. The medium-grade lignite giving 58.4 per cent char at low temperature gave 55.8 per cent at high temperature, while the average yield of char from sub-bituminous coals at high and low temperatures was 71.0 per cent and 61.7 per cent respectively.

6. The yield of gas varied from 4,500 to 5,000 cubic feet per (2,000 lb.) ton for the three lignites at low temperature compared with an average of 3,100 cu. ft. for the two sub-bituminous coals. At high temperature this relation was reversed, viz.: 8,800 cu. ft. from the lignite tested and an average of over 10,000 for the two sub-bituminous coals.

7. The tar yields were 3.3, 6.2 and 6.3 Imp. gallons respectively for the three lignites at low temperature compared with 9.9 and 8.6 gallons for the sub-bituminous coals. At high temperature the tar yield fell to less than 4 gallons for the lignite tested and an average of about 5 gals. for the sub-bituminous coals. The average laboratory yield for the five brown lignites has previously been reported as 5.5 Imp. gallons per ton of coal as charged. Inasmuch as it is doubtful if over 60 per cent of these tar yields could be realized on the commercial scale, the tar oil by-product from these lignite and sub-bituminous coals may not be considered as important in comparison with that from peat on the one hand and bituminous coals on the other.

8. The carbonized residue or char was not examined as to suitability for briquetting, but further work along this line, particularly on char from low price fines or slack coal, is included in future working plans for carbonization and briquetting experiments.

TABLE I
Analyses of Coals Carbonized

| Coals | A Cardiff | B Rosedeer | C Taber | D Coalspur | E Harlech |
|------------------------|--------------|---------------|------------|---------------|--------------|
| Analyses (as charged)— | | | | | |
| Moisture.....% | 21.4 | 16.5 | 12.0 | 7.9 | 6.7 |
| Ash.....% | 6.4 | 7.2 | 7.7 | 8.0 | 7.4 |
| Volatile matter.....% | 32.5 | 34.1 | 33.7 | 34.5 | 32.0 |
| Fixed carbon.....% | 39.7 | 42.2 | 46.6 | 49.6 | 53.0 |
| 100.0 | | | | | |
| Nitrogen (1).....% | 0.9 | 1.2 | 1.4 | 0.8 | 1.0 |
| Sulphur.....% | 0.2 | 0.4 | 0.9 | 0.2 | 0.2 |
| B.T.U. per lb..... | 8,680 | 9,860 | 10,480 | 11,280 | 11,750 |
| Analyses (dry basis)— | | | | | |
| Ash.....% | 8.1 | 8.6 | 8.8 | 8.7 | 7.9 |
| Volatile matter.....% | 41.4 | 40.8 | 38.3 | 37.5 | 35.3 |
| Fixed carbon.....% | 50.5 | 50.6 | 52.9 | 53.8 | 56.8 |
| B.T.U. per lb..... | 11,040 | 11,800 | 11,910 | 12,240 | 12,590 |

(1) Nitrogen and sulphur are average for coals from the area.

TABLE II
Summary of Low Temperature Carbonization Tests
(Yields per 2,000 lbs. of coal as charged)

| Coals | A | B | C | D | E |
|--|--------|--------|--------|--------|--------|
| Maximum temperature of carbonization as per lead bath pyrometer reading..... | 600°C. | 630°C. | 610°C. | 600°C. | 590°C. |
| Carbonized residue or char:— | | | | | |
| Per cent of coal carbonized..... | 54.0 | 58.4 | 63.1 | 69.7 | 72.3 |
| Proximate analyses— | | | | | |
| Ash.....% | 11.2 | 11.7 | 12.6 | 11.5 | 10.0 |
| Volatile matter.....% | 11.8 | 8.9 | 9.0 | 12.1 | 10.8 |
| Fixed carbon.....% | 77.0 | 79.4 | 78.4 | 76.4 | 79.2 |
| Calorific value B.T.U. per lb..... | 12,750 | 13,320 | 13,040 | 12,920 | 13430 |
| Gas (as per meter readings)— | | | | | |
| Cu. ft. per ton..... | 4,500 | 4,540 | 5,000 | 3,290 | 3,095 |
| B.T.U. per cu. ft. (calculated)..... | 448 | 502 | 575 | 519 | 582 |
| Density (air=1)..... | 86 | 70 | 73 | 81 | 65 |
| Tar (dry)— | | | | | |
| Imp. gals. per ton..... | 3.34 | 6.21 | 6.28 | 9.90 | 8.60 |
| Sp. Gr. at 15.5° C..... | 0.981 | 0.994 | 1.020 | 0.994 | 1.015 |
| Liquor (aqueous)— | | | | | |
| Imp. gals. per ton..... | 608 | 513 | 432 | 309 | 311 |
| (NH ₄) ₂ SO ₄ lbs. per ton..... | 5.6 | | 13.2 | 5.6 | 9.2 |

TABLE III

Weight Balance—Low Temperature Carbonization Tests
(Parts by weight for 100 parts of coal charged)

| Coal | A | B | C | D | E |
|-----------------------------------|------|------|-------|------|------|
| Coke..... | 54.0 | 58.4 | 63.1 | 69.7 | 72.3 |
| Tar..... | 1.6 | 3.2 | 3.1 | 4.9 | 4.4 |
| Liquor..... | 30.4 | 25.7 | 21.6 | 15.5 | 15.5 |
| Gas and loss (by difference)..... | 14.0 | 12.7 | 12.2 | 9.9 | 7.8 |
| | | | 100.0 | | |
| Gas (calculated)(1)..... | 14.7 | 12.0 | 12.8 | 10.1 | 7.6 |

(1) The figures used here were calculated from the yields in Table II as at 60°F. and 30'' pressure.

TABLE IV

Analyses of Tar Oils—Low Temperature Carbonization Tests

| Coal | A | | B | | C | | D | | E | |
|----------------------|-------|------------------|--------|------------------|--------|------------------|--------|------------------|--------|------------------|
| Tar (dry)— | | | | | | | | | | |
| Sp.Gr. at 15.5° C. | 0.981 | | 0.994 | | 1.020 | | 0.994 | | 1.105 | |
| B.T.U. per lb..... | | | | | 16,400 | | 16,240 | | 16,355 | |
| Distillation | % Vol | Sp. Gr. 15.5° C. | % Vol | Sp. Gr. 15.5° C. | % Vol | Sp. Gr. 15.5° C. | % Vol | Sp. Gr. 15.5° C. | % Vol | Sp. Gr. 15.5° C. |
| To 200° C..... | 25 | 0.817 | 25 | 0.88 | 20 | 0.894 | 21 | 0.846 | 20 | 0.918 |
| 200-300° C..... | 37 | 0.972 | 35 | 0.96 | 36 | 0.985 | 35 | 0.961 | 42 | 0.996 |
| 300-350° C..... | 15 | 0.992 | 17 | 0.98 | 19 | 0.013 | 18 | 1.006 | 20 | 0.018 |
| Pitch by weight..... | 22 | | 21 | | 23 | | 25 | | 17 | |
| M.P. of pitch..... | | | 79° C. | | | | | | 74° C. | |
| To 170° C..... | 15 | | 8 | | 9 | | 10 | | 6 | |
| 170-230° C..... | 21 | | 28 | | 23 | | 21 | | 28 | |
| 230-270° C..... | 16 | | 15 | | 15 | | 17 | | 17 | |
| 270-310° C..... | 12 | | 12 | | 12 | | 11 | | 12 | |
| 310-350° C..... | 12 | | 14 | | 16 | | 16 | | 17 | |
| Pitch (by wt.)..... | 22 | | 21 | | 23 | | 25 | | 17 | |

TABLE V

Analyses of Gases from Low Temperature Carbonization Tests

| Coal | A | B | C | D | E |
|------------------------|------|------|------|------|------|
| Density (air = 1)..... | 0.86 | 0.70 | 0.73 | 0.81 | 0.65 |
| CO ₂ | 33.8 | 18.0 | 24.3 | 16.7 | 8.0 |
| Illuminants..... | 0.8 | 1.9 | 2.2 | 1.7 | 1.0 |
| O ₂ | 2.2 | 1.9 | 0.7 | 1.4 | 3.5 |
| CO..... | 7.1 | 6.1 | 5.2 | 6.1 | 5.2 |
| CH ₄ | 37.0 | 37.2 | 46.9 | 44.6 | 43.4 |
| H ₂ | 13.2 | 25.6 | 17.0 | 8.1 | 19.8 |
| N ₂ | 5.9 | 10.3 | 3.7 | 21.4 | 14.1 |
| B.T.U. per cu. ft.— | | | | | |
| Net..... | 405 | 440 | 519 | 469 | 526 |
| Gross..... | 448 | 502 | 575 | 519 | 582 |

The density and B.T.U. per cu. ft. are calculated values.

TABLE VI
Thermal Balance—Low Temperature Carbonization Tests
(Thermal value of products as percentage of that in raw coal charged)

| Coal | A | B | C | D | E |
|------------|------|------|------|------|------|
| Coke.....% | 79.6 | 78.9 | 78.5 | 79.9 | 82.6 |
| Tar.....% | 3.1 | 5.1 | 5.0 | 7.1 | 6.1 |
| Gas.....% | 11.6 | 11.6 | 13.7 | 7.9 | 7.7 |
| Loss.....% | 5.7 | 4.4 | 2.8 | 5.1 | 3.6 |

TABLE VII
Summary of High Temperature Carbonization Tests
(Yields per 2,000 lbs. of coal as charged)

| Coal | B | D | E |
|--|--------|--------|--------|
| Carbonized residue or char— | | | |
| Per cent of coal as charged..... | 55.8 | 60.4 | 63.1 |
| Pounds per ton..... | 1,116 | 1,208 | 1,262 |
| Proximate analysis of char— | | | |
| Ash.....% | 12.4 | 12.1 | 11.4 |
| Volatile matter.....% | 2.3 | 1.3 | 1.1 |
| Fixed carbon.....% | 85.3 | 86.6 | 87.5 |
| B.T.U. per lb..... | 12,810 | 12,520 | 12,855 |
| Gas (as per meter reading)— | | | |
| Cu. ft. per ton..... | 8,790 | 9,420 | 11,030 |
| Density calculated (air = 1)..... | 0.65 | 0.61 | 0.50 |
| Analyses— | | | |
| CO ₂ | 14.9 | 7.1 | 5.9 |
| Illuminants..... | 1.6 | 2.8 | 2.1 |
| O ₂ | 1.6 | 4.6 | 5.4 |
| CO..... | 9.0 | 10.2 | 12.1 |
| CH ₄ | 28.1 | 22.6 | 23.4 |
| H ₂ | 32.0 | 33.9 | 45.7 |
| N ₂ | 12.8 | 18.8 | 5.4 |
| B.T.U. per cu. ft.— | | | |
| Gross..... | 439 | 413 | 454 |
| Net..... | 393 | 371 | 405 |
| Liquor-aqueous (Imp. gals. per ton)..... | 41.7 | 30.6 | 25.1 |
| Ammonium sulphate (lbs. per ton)..... | 19.9 | | |
| Tar (dry)— | | | |
| Imp. gals. per ton..... | 3.8 | 6.1 | 4.1 |
| Specific gravity at 15.5°C..... | 0.983 | | 1.045 |
| B.T.U. per lb..... | 16,240 | | 15,710 |
| Distillation (% Volume)— | | | |
| Up to 170°C..... | 14 | | 12 |
| 170-230°C..... | 26 | | 25 |
| 230-270°C..... | 13 | | 10 |
| 270-310°C..... | 13 | | 9 |
| 310-350°C..... | 16 | | 17 |
| Pitch (by wt.)..... | 17 | | 26 |

TABLE VIII
Weight and Thermal Balance—High Temperature Carbonization Tests
(Weight and thermal value of products as percentage of that in raw coals as charged)

| Coal | Weight balance | | | Thermal balance | | |
|------------------------------------|----------------|------|------|-----------------|------|------|
| | B | D | E | B | D | E |
| Coke.....% | 55.7 | 60.4 | 63.1 | 75.3 | 67.1 | 69.1 |
| Tar.....% | 1.9 | 3.0 | 2.2 | 3.1 | 4.2 | 2.9 |
| Aqueous liquor.....% | 20.8 | 15.3 | 12.6 | | | |
| Gas and loss (by difference.)..... | 21.6 | 21.3 | 22.1 | | | |
| Gas (calculated)..... | 21.7 | 21.8 | 20.9 | 19.8 | 19.7 | 21.3 |
| Loss.....% | | | | 1.8 | 9.0 | 6.7 |

II

SURVEY OF MARITIME PROVINCES COALS

J. H. H. Nicolls

INTRODUCTION

This report is a description of samples taken by the writer during the summer of 1923 in the principal coal fields of New Brunswick and Nova Scotia. It is proposed that within the next few years the Division of Fuels and Fuel Testing, either alone or with the co-operation of the Provincial Laboratories, shall carry out a general survey of Canadian coal fields, and the samples herein described represent the first section of such a survey, inasmuch as they furnish information as to the quality of coal now being mined in the Maritime Provinces.

During the years 1908-1909 a comprehensive survey of the coals of Canada was carried out in connexion with the "Government Coal Tests" conducted at McGill University by Dr. J. B. Porter and a considerable staff. A description of this survey, and of the laboratory and commercial scale tests which followed it, is to be found in "An Investigation of the Coals of Canada"¹ by Porter and others. Subsequently this work was transferred from McGill University to the Fuel Testing Station at Ottawa, and in 1918 the principal analyses carried out in either laboratory were published as "Analyses of Canadian Fuels" compiled by Stansfield and Nicolls.²

The present survey was carried out along very much the same lines as that made by Denis 15 years ago. In all 65 samples were collected, of which 14 were from New Brunswick and 51 from Nova Scotia. The field notes taken during the survey and the chemical analyses of the samples have been of special interest to the Dominion Fuel Board in that body's recent investigation as to the suitability of coals from the Maritime Provinces for the manufacture of domestic coke.

COLLECTION OF SAMPLES

In planning and carrying out the survey it was decided that the most reliable samples were those obtained by cutting the coal seams in the mines. In this way samples of coal almost entirely free from impurities were obtained, which serve as a fairly reliable basis upon which to compare the coal from the different seams. These samples have been designated as "mine" samples, as distinguished from the "tipple" samples taken above ground. Whenever in the description of a sample it is stated that partings or impurities were discarded, the sampler has been following the practice used as far as possible in mining the seam. In the few instances where a portion of the seam was sampled, only that particular portion was being worked at the time of sampling.

At a great many mines there is considerably more foreign matter in the coal as brought above ground than there is in the seam. This is due partly to thin intrusions, which could be almost completely removed in

¹Mines Branch Publication No. 83--In six volumes.

²Mines Branch Bulletins Nos. 22--26.

cutting a small sample, but which could not be entirely taken out in mining without considerable loss of time and labour. Furthermore, shale and rock from the roof and floor of the mine sometimes come to the surface with the coal, and it is often due to the removal of these that some mines produce coal superior to others on the same seam. Therefore, because they represent coal as shipped from the mines and may be slightly inferior to the "mine" samples, "tipple" or surface samples were taken in order to supplement the information obtainable from the principal or "mine" samples.

The survey was not as thorough as is generally advisable owing to the necessity of its completion within a limited time. Generally speaking, only one mine and one tipple sample were taken at each mine, whereas in a very careful and detailed survey at least four mine samples should be taken, and one or more tipple samples according to the production capacity of the mine. At each mine the manager or principal foreman was requested to indicate the part of the seam then being mined that, to the best of his knowledge, represented its average composition, and there the sample was taken. If the coal had not been freshly cut, that is within the last few days, an inch or more (according to the time elapsed since last cutting) of surface coal was removed before taking the sample. In addition any adjacent loose coal or rock was removed lest it should fall and contaminate the sample. For the sample a uniform section or "channel" was cut across the seam from top to bottom and allowed to fall on to a tarpaulin stretched on the floor of the mine so as to exclude mine dust or impurities. The area of the cross-section of the channel was varied with the height of the seam and with its apparent uniformity or content of impurities. Where intrusions of shale or "partings" were left out, a note of this and any other physical characteristics of the seam is to be found in the description of the individual samples. If the sample was too large for a 125-pound sack it was quartered on the tarpaulin. This was also done in the few instances where the sacks had to be carried to the hoists.

Tipple samples were usually taken from railway cars either in the course of or after loading. At one colliery the cars were specially loaded, and samples taken as the coal came from the chutes to the cars. The practice generally followed was to take the samples from all over the surface of a full or partly loaded car, usually the former. If there were any reason for taking a sample representative of more than one car-load all the coal was thrown on to the tarpaulin, where it was thoroughly mixed and then quartered. In a few instances where no coal was being shipped, the tipple samples were taken from loading bins or from storages. Usually these contained a large quantity of coal, and the samples were considered to be representative. On the other hand, samples taken from a small pile or a stationary picking belt, while they may be quite representative, cannot be considered as satisfactory as those taken from a larger quantity of coal, and are therefore designated as "grab" samples.

DESCRIPTION OF SAMPLES

As is the case in "Analyses of Canadian Fuels," the samples are described under the numbers allotted to them on arrival at the Fuel Testing Station, which are not necessarily consecutive. For the sake of uniformity the mine samples are listed before the corresponding tipple samples. It

was primarily intended to list the areas in the same order as in Bulletin No. 22 (just referred to), that is beginning with the Sydney area, Nova Scotia. However, as this survey was largely brought about by representations of New Brunswick interests, since the sample numbers run in order from Minto where the survey was begun to Sydney where it finished, and since even in the Sydney area the numerical order would be entirely upset by listing the samples as in Bulletin No. 22, it was decided to tabulate the samples almost entirely in chronological order.

The names of areas are identical with those previously used, except that the former "Grand Lake Area" is called "Minto (Grand Lake) Area." It is proposed to replace the name Grand Lake by the better known Minto when referring to the area in any future publications, and the combination of the two names is an intermediate step.

In addition to the samples collected by the writer, seven more from the Minto area have been included in this report. These were taken by W. S. Dyer, of the Geological Survey, and should be classed as field survey samples. Furthermore, four samples from Caraquet, Gloucester co., N.B., and two from Riversdale, Colchester co., N.S., have been included because of their being the first samples received from the deposits which they represent. These were not taken by representatives of the Department, but at least indicate the general nature of the deposits. All these samples were taken during the past summer, with the possible exception of those from Riversdale. As these were received in July, it seems probable that they also were taken during the summer of 1923.

NEW BRUNSWICK

Minto (Grand Lake) Area

Fourteen samples were collected from six mines.

Minto Coal Co., Ltd., Minto, No. D 3 shaft, South Minto.

No. 2289—From No. 2 by-level, north side of workings, 140 feet from shaft bottom, 37 feet from surface.

Channel cut across 29-inch seam at one end of a freshly cut bench. Shale was removed from sample.

No. 2288—Over $\frac{5}{8}$ -inch screen, from box car loading at tippie.

Welton & Henderson, Ltd., Minto, No. C 2 shaft, South Minto.

No. 2291—From No. 1 room, No. 1 level, west side of workings, 70 feet from shaft bottom, 58 feet from surface.

Channel cut across 26-inch seam in a 14-foot working face. All shale and foreign matter excluded.

No. 2290—Over $\frac{5}{8}$ -inch screen, from freshly loaded open car.

H. and W. Welton, Minto, No. 6 shaft, South Minto.

No. 2298—Over $\frac{5}{8}$ -inch screen, from freshly loaded open car.

No. 2299—Slack, through $\frac{5}{8}$ -inch screen, from freshly loaded open car.

The coal appeared dry as it came from the mine, but the cars sampled were wet owing to a recent shower.

Rothwell Coal Co., Ltd., Minto, No. 10 shaft, South Minto.

No. 2301—From 50 feet northeast of shaft bottom, 48 feet from surface.

Channel cut across 18-inch seam after a layer of about 2 inches of coal had been stripped off. Owing to pressure of time the sample was taken in one of the tunnels from a place that had not been worked for almost a year, where the mine was very wet.

No. 2300—Over $\frac{5}{8}$ -inch screen, from freshly loaded open car.

The coal appeared wet as it came from the mine, and was still wetter in the car due to a recent shower.

Miramichi Lumber Company, Minto, No. 7 shaft on old King property, North Minto.

Following two samples from No. 4 level, raise side of main level; 188 feet from shaft bottom, 47 feet from surface.

No. 2293—24-inch upper seam.

No. 2294—6-inch lower seam.

After stripping off about 1 inch of coal, channels practically in alignment were cut across the two seams.

No. 2292—Over $\frac{5}{8}$ -inch screen, from car loading at tippie.

Minto Coal Co., Ltd., Minto, No. 2 B shaft, North Minto.

Following two samples from face of crosscut in preparation for longwall mining, No. 1 by-level, No. 2 place right, 80 feet north of shaft bottom, 101 feet from surface.

No. 2296—22-inch upper seam.

About 1 inch was stripped off face of seam before cutting channel.

No. 2297—6-inch lower seam.

Face was freshly cut, so that coal was not stripped before cutting channel. Upper 2 inches of seam are bone coal, which was excluded from sample.

No. 2295—Over $\frac{3}{4}$ -inch screen, from car loading at tippie.

The aforesaid five companies produce almost all the coal that comes on the general market from this area. The coal is sold either screened (one size only, generally over $\frac{5}{8}$ -inch) or as slack. A large part of the screened output goes to the railways, while a great deal of it is consumed in the province or shipped to the state of Maine for manufacturing or domestic use. The slack is employed for power-raising, very largely by pulp and paper companies.

In addition to samples taken by the writer, the following were taken by Mr. Dyer from holdings already developed.

Avon Coal Co., Ltd., Newcastle Creek (South Minto).

No. 2402—Upper seam.

No. 2400—Lower seam.

No. 2401—Tippie sample.

McDougal Bros., Minto

No. 2403—From mine at South Minto.

McPherson's Mine, Coal creek, near Chipman.

No. 2541—General sample.

Prospect samples were taken by Mr. Dyer from Eighteen creek, 7 miles southwest of Minto, as follows:—

No. 2542—Middle seam.

No. 2543—Upper seam.

Gloucester Area

The following samples were taken by Mr. J. W. Nixon from a property which he is developing for local fuel supply at Caraquet:

No. 2485—Prospector's sample.

Following samples taken after development was under way:

No. 2599—Top half of 24-inch seam, 18 feet underground, 150 feet down slope.

No. 2600—Bottom half of same 24-inch seam.

No. 2601—Same as last, but only one corner of seam, which appeared to contain clay.

NOVA SCOTIA

Joggins-Chignecto Area

The following eight samples were taken from four mines.

River Hebert Coal Company, River Hebert, Marsh mine, Kimberley seam.

No. 2308—From No. 5 room west of slope, 575 feet down slope, 125 feet from slope bottom.

Channel cut across two 18-inch sections of seam with 1 or 2 inches of parting dividing them. The parting was discarded.

No. 2309—Run-of-mine coal from loading pocket.

Maritime Coal, Railway & Power Co., Ltd., Maple Leaf Colliery, River Hebert, Beech Grove mine, Old Joggins seam.

No. 2310—From No. 2 sheet, No. 7 room west of slope, 450 feet down slope, 150 feet from slope bottom.

Channel cut across two 27-inch portions of seam, divided by 4-foot parting of shale.

No. 2311—Run-of-mine coal from box car loading at tipple.

Minudie Coal Co., Ltd., Victoria Colliery, River Hebert, No. 1 slope, Queen seam.

No. 2312—From 2,000 feet west of slope, 1,000 feet down slope, 100 feet from slope bottom.

Channel cut across two 15-inch sections of seam, divided by about the same thickness of shale

No. 2313—Unscreened coal, over picking belt, from pile on the ground under the tipple.

This was stated by the mine manager to be representative of the average output.

Fundy Coal Co., Ltd., Joggins, Fundy seam.

No. 2315—From east side of slope, 1,075 feet down slope and practically at the bottom.

Cut channel across the following section: 4 inches poor coal—30 inches coal, including a little parting—10 inches of shale—3 inches of coal.

The poor coal, any lumps of parting from the 30-inch section and all the 10 inches of shale were discarded.

No. 2314—Run-of-mine coal from "run-of-mine" pocket.

According to information gathered from the above companies, the coal sold from this area is principally screened, though a considerable amount of run-of-mine is also sold. Three of the four companies employ $\frac{3}{4}$ -inch screens, so that this size may be considered as representative. A considerable percentage of the output, largely run-of-mine, is sold to the railways or loaded into ships on the bay of Fundy, while screened coal is sold for domestic use. Whatever slack is produced is generally consumed in the various companies' power plants. Only one company uses a picking belt.

Springhill Area

Four samples were taken from two mines.

Cumberland Railway and Coal Co., Ltd. (British Empire Steel Corporation) Springhill.

No. 2 Mine, Springhill, No. 2 seam.

No. 2316—From counter level, 150 feet east of riding slope, 5,400 feet down slope, 1,200 feet from slope bottom.

After stripping about 4 inches from face, cut channel across 9½-foot seam. Sample was reduced once by quartering.

No. 2317—Over $\frac{1}{2}$ -inch screen and picking belt, from 14-ton open car loading at tipple.

No. 6 Mine, Springhill, No. 6 seam.

No. 2318—From 300 feet south of slope, at bottom of slope, 1,600 feet from surface.

Channel cut across 5-foot 8-inch seam.

No. 2319—Run-of-mine coal, from open car loading at tipple.

Part of this coal appeared very wet.

The output of these mines is largely unscreened coal, which at No. 2 mine has been sent over a picking belt. This is sold principally to the railways, which consume a large proportion of the total output. Screened coal is disposed of locally or shipped by water from Parrsboro' on the bay of Fundy, while a certain amount of it is sold to the railways. Slack or culm is either sold to pulp and paper companies or consumed in the company's own power plant.

Colchester Area

The following outcrop samples were taken by the property holders from near Riversdale on the Canadian National railway.

No. 2321—Lump coal.

No. 2321A—Slack coal.

Pictou Area

Thirteen samples were taken from six mines.

Intercolonial Coal Mining Co., Ltd., Drummond Colliery, Westville.

No. 1 Mine, No. 1 or Main seam.

No. 2327—From No. 10 level, 500 feet north of slope, 4,000 feet down slope, 1,000 feet from slope bottom.

Channel cut across lower 8 feet of 17-foot seam; 1 foot of parting omitted.

No. 2 Mine, No. 2 or Scott seam.

No. 2326—From No. 1 room, No. 3 balance north of slope, 1,400 feet from slope, 2,000 feet down slope, 1,500 feet from slope bottom.

Channel cut across central 8 feet of 13-foot seam.

The coal from these two mines is sent to the one bankhead, where it is mixed in proportions which vary somewhat but average 5 parts of Scott to 2 parts of Main. Owing to the difficulty of obtaining a true average without sampling a number of cars, the mine authorities arranged to load the output of the two seams into separate cars. In this way samples were taken representing each individual seam as delivered from the bankhead picking tables. By combining the analyses of these in the correct proportions there was obtained the average composition of the coal as shipped. This has been tabulated as No. 2328-9.

No. 2329—No. 1 Main seam, unscreened coal over picking belt, from box car loading at tipple.

No. 2328—No. 2, Scott seam, unscreened coal over picking belt, from open car loading at tipple.

The samples were taken as the coal was loaded to the cars. The writer was unable to wait till the cars were filled, but each sample covered what was considered to be a representative part of the car load.

As was found to be the case in many Nova Scotia collieries, the bulk of the output of this company is disposed of to the railways. This is mainly unscreened. Some coal is screened over $\frac{3}{4}$ -inch, principally for local consumption, while the slack is used for power-raising. All the coal is sent over a picking belt.

Acadia Coal Co., Ltd. (British Empire Steel Corporation) Stellarton.

Allan Shaft Colliery, Stellarton.

No. 2325—Unscreened coal, over picking belt, from two loaded open cars on siding.

The bulk of this coal comes from the Foord seam, though a little of it is from the Third seam.

Albion Colliery, Stellarton, Albion and Acadia No. 1 Mines.

Albion Mine, Foord seam.

No. 2334—From No. 3 pillar, southwest balance, east incline off Cage seam.

Channel cut across upper 10 feet of 40-foot seam.

No. 2333—From No. 2 pillar, southwest balance, east incline off Cage seam.

Channel cut across upper 8 feet of lower 12 feet of 40-foot seam. A little parting removed from sample.

Albion Mine, Cage seam.

No. 2332—From No. 1 pillar off No. 6 level, northwest balance, 900 feet northeast of slope.

Channel cut across lower 10 feet of 14-foot seam. A rock intrusion was left out.

Albion Mine, Third seam.

No. 2331—From east sinking off No. 6 level, 800 feet east of slope, 3,700 feet down slope, 300 feet from slope bottom.

Channel cut across upper 8 feet of 12-foot seam. A few pieces of rock discarded.

Acadia No. 1 Mine, Acadia No. 1 seam.

No. 2335—From No. 5, main (2,700-foot) level, 1,400 feet west of slope.

Channel cut across the 8-foot seam.

No. 2330—Average of output of Albion bankhead, unscreened coal, over picking belt, from open car on siding.

This average is composed of about 9 parts of Acadia No. 1, 7 of Foord, 7 of Cage, 1 of Third seam.

The output of both the Allan Shaft and Albion collieries is disposed of very largely unscreened; this goes principally to the railway. A considerable amount of coal is sold locally for domestic use, and this is screened into the four sizes, lump, egg, chestnut, and culm. All coal is sent over picking belts.

Acadia No. 3 Colliery, Thorburn, Old Vale or Greener seam.

No. 2323—From No. 1 slant off No. 3 level, west side of slope, 2,500 feet from slope to top of slant, 150 feet down slant, 300 feet west of slant.

Channel cut across longwall face of 50-inch seam, including a little scattered bone coal. This bone occurs irregularly throughout the seam, so that the sample may be either a little better or a little worse than the average of the seam.

No. 2324—Unscreened coal, over picking belt, from open car loading at tipple.

The output of this colliery is entirely unscreened coal, over picking belt, all of which is consumed by the railway.

Inverness Area

Only two samples, one from each of the mines operating at Inverness, were taken in this area. A miners' strike had been called at Sydney and conditions at Inverness were threatening, so that the writer had no opportunity to take samples underground.

Inverness Railway and Coal Co., Inverness

No. 1 Mine, No. 1 seam.

No. 2336—Sample taken across face of seam under direction of mine manager. Sample consisted of lumps of coal (weight about 10 lbs. in all) removed at intervals across the seam. Smalls, fines and partings were excluded, so that the sample is probably better than run-of-mine.

No. 4 Mine, No. 4 seam.

No. 2337—Unscreened coal, over picking belt, from discharge end of travelling belt, from No. 1 bankhead which now serves both mines.

The coal shipped from Inverness is mainly unscreened, and has all passed over picking belts. Occasionally a $\frac{3}{4}$ -inch screen is used. A great deal of the coal is consumed by the railways, while some of it is shipped by water from the company's wharf at Port Hastings.

Sydney Area

Twenty-one samples were collected from ten mines.

During the entire period that the writer was in this area the miners were out on strike. It was therefore impossible to take samples quite as satisfactory as those from areas on the mainland of Nova Scotia or New Brunswick. However, the samples collected cover the principal mines in the area, and ought to be representative in nearly all cases.

Nova Scotia Steel & Coal Co., Ltd. (British Empire Steel Corp.) Sydney Mines.

It was found to be inadvisable to attempt to take samples underground at Sydney Mines. There was, however, a certain amount of coal in storage or in cars, and samples of this were taken as indicating the composition of the coal from the various mines.

No. 5 Jubilee Mine, Sydney Mines, Upper or Indian Cove seam.

No. 2339—Unscreened coal, over picking belt.

No. 5 Jubilee Mine, Lower or Toronto seam.

No. 2338—Unscreened coal, over picking belt.

The coal from which these samples were taken had been stored, as a test for spontaneous combustion, in two covered bins similar to box cars, in the company's main yard.

The Toronto seam is said to average better than the Indian Cove, but it did not appear so in the storage bin.

No. 7 Alexandra Mine, Little Bras d'Or, Lloyd's Cove seam.

No. 2340—Run-of-mine coal, from a 15-ton open car in the company's main yard.

It was subsequently noted that other cars of this coal seemed to be somewhat cleaner than the one sampled. All coal appeared to contain a good deal of impurities.

No. 1 Princess Mine, Sydney Mines, Main seam.

No. 2342—Unscreened coal, over picking belt, from two 15-ton cars on siding in Princess yard.

No. 3 Florence Mine, Florence, Main seam.

No. 2343—Run-of-mine coal; grab sample from the "domestic" pocket.

The pocket, which serves for local delivery, was completely emptied on to the tarpaulin, and half the coal taken for the sample.

Princess and Florence Mines.

No. 2341—Grab sample from along one face of 140,000-ton storage pile at Princess mine. The coal in the pile came from both No. 1 and No. 3 mines.

The company has coke ovens and a steel plant at Sydney Mines, but the ovens had ceased operating and the steel plant had been running at less than full capacity since the merger with the Dominion Iron & Steel Company. Half the total output of the mines used to be consumed by the steel plant.

Dominion Coal Co., Ltd. (British Empire Steel Corporation) Glace Bay.

Conditions at Glace Bay were a little more favourable for taking samples underground than at Sydney Mines. They were not however satisfactory. At four of the collieries the managers took the mine samples, and, from information gathered, the writer considers that they were carefully taken, and fairly represent the part of the seam sampled. They were, however, rather smaller than was desirable.

Dominion No. 22 Colliery, Birch Grove, Gowrie seam.

No. 2347—From No. 5 room, No. 7 deep, No. 4 east level.

No. 2348—From No. 6 room, No. 3 headway, No. 4 east level.

For the above two samples channels were cut across the 5-foot seam by the colliery manager. The samples averaged about 6 lbs. each. At the time of sampling practically all mining operations were carried out in No. 4 east level.

No. 2346—Over $\frac{5}{8}$ -inch round screen and picking belt, grab sample from discharge end of belt.

Dominion No. 11 Colliery, Caledonia Junction, Emery seam.

No. 2352—From No. 1 room, No. 1 headway off No. 5 east level.

No. 2353—From No. 1 room, No. 5 headway off No. 4 west level.

The above samples, of about 10 lbs. each, were taken from the 4-foot 5-inch seam by the colliery manager.

No. 2354—Over $1\frac{1}{4}$ -inch screen and picking belt, from partly loaded open car.

Dominion No. 12 Colliery, New Waterford, Victoria seam.

No. 2355—From No. 6 west level, west landing, 200 feet from slope, 3,500 feet down slope.

After stripping 2 to 4 inches from face, cut channel across 5-foot seam. One piece of shale was picked out from sample, which was taken from the side of a tunnel cut five years ago.

No. 2356—Unscreened coal, over picking belt, grab sample from underneath car dumped at tipple.

Dominion No. 16 Colliery, New Waterford, Lingan seam.

No. 2357—From No. 7 landing, 5,400 feet from the surface.

Sample taken by colliery manager from 5-foot 6-inch seam.

No. 2358—Unscreened coal, over picking belt, grab sample from a small pile in the colliery yard, consisting of slack with a few large lumps.

Dominion No. 4 Colliery, Caledonia, Phalen seam.

No. 2350—From east side of east deep, No. 16 east level, 10,400 feet from shaft.

No. 2351—From west side of east deep, in main deep, 12,000 feet from shaft.

The above two samples, of about 8 lbs. each, were taken from the 6-foot 11-inch seam by the colliery manager.

No. 2349—Unscreened coal, over picking belt, from open car partly loaded at tipple.

Dominion No. 2 Colliery, New Aberdeen, Phalen seam.

No. 2359—From No. 5 room, No. 4 south off No. 1 north deep, about 1 mile from the shaft and near the centre of workings.

Channel cut across 7-foot seam. Sample cut in half on the tarpaulin in the mine.

No. 2360—Unscreened coal, over picking belt, grab sample from discharge end of belt.

The distribution centre for all these collieries is at No. 2 colliery, New Aberdeen. Here there is a large mixing floor and storage pile, which at the time of the survey contained 400,000 tons. A considerable amount of the output is consumed by the allied Dominion Iron and Steel Company at Sydney, principally in the coke ovens. The greater portion of the output comes on to the general market, very largely by water as the company operates the railway between Sydney and Louisburg, which are respectively summer and winter ports.

Arrangements were made with Mr. C. E. Wallin, superintendent of the coke ovens plant at Sydney, to obtain representative samples of the Dominion slack coal and of the coke produced from it. These were taken as soon as operations were fully under way following the strike, and are as follows:—

No. 2383—Raw coal from day's run of August 13.

No. 2384—Coal as above coming from Baum washer.

No. 2385—Coke made from above washed coal.

ANALYSES OF SAMPLES

The chemical analyses of the coal samples above described are shown in the following tables. They were carried out by different members of the staff of the Fuel Testing Laboratories. The proximate analyses were made by C. B. Mohr and J. W. McKinney, and the calorific values were determined by G. B. Frost. The special large scale laboratory coking tests were made by Harold Kohl, and the fusibility of ash determinations by J. W. McKinney.

The large scale laboratory coking tests were made in a large melter's furnace, using 12 pounds of coal in each case. Coal crushed in a coffee mill was slightly moistened and charged into a covered graphite crucible. The crucible and contents were placed in the furnace, which had been previously heated to near the coking temperature, and about three hours allowed for coking. The temperature of coking was approximately 1,000° Centigrade (1,830°F.). Most of the original survey samples were coked on both the large and small laboratory scales, and the coking properties tabulated have in such cases been judged from both of these.

For fusibility determinations the finely ground ash was moulded into a standard size cone with a 10 per cent solution of dextrin, and gradually heated in a No. 3 melter's furnace, according to the official method of the U.S. Bureau of Mines.¹ "I.D." in the tables denotes Initial Deformation, the temperature at which the tip of the cone first showed signs of bending; "Softening," the temperature at which the ash was melted into a spherical

¹ Bulletin No. 209—Fusibility of Ash from Coals of the United States. Selvig and Fieldner.

lump; "Fluid," the temperature at which the ash cone had entirely flattened. For practical purposes the intermediate temperature is usually considered as the fusion point, and is therefore shown on both the Centigrade and Fahrenheit scales. The I. D. and Fluid temperatures are indicated by the number of degrees Centigrade that separate them from the softening temperature.

NEW BRUNSWICK
Minto (Grand Lake) Area

| No. | Description | Moisture, as rec'd. | Analysis, dry basis | | | | | |
|------|--|---------------------|---------------------|-----------------|--------------|------------|---------|----------|
| | | | Ash | Volatile matter | Fixed carbon | Fuel ratio | Sulphur | Nitrogen |
| 2289 | Minto Coal Co., S. Minto—mine..... | 1.1 | 15.5 | 32.1 | 52.4 | 1.65 | 6.9 | 0.7 |
| 2288 | Minto Coal Co., S. Minto—tipple..... | 1.0 | 18.5 | 30.5 | 51.0 | 1.65 | 7.4 | |
| 2291 | Welton & Henderson, S. Minto—mine... | 1.1 | 15.3 | 31.3 | 53.4 | 1.70 | 7.4 | |
| 2290 | Welton & Henderson, S. Minto—tipple... | 1.1 | 18.9 | 30.1 | 51.0 | 1.70 | 8.4 | |
| 2298 | H. & W. Welton, S. Minto—tipple..... | 1.1 | 15.5 | 31.2 | 53.3 | 1.70 | 5.7 | |
| 2299 | H. & W. Welton, S. Minto—slack..... | 2.0 | 18.7 | 30.8 | 50.5 | 1.65 | 6.2 | |
| 2301 | Rothwell Coal Co., S. Minto—mine..... | 1.2 | 13.6 | 31.3 | 55.1 | 1.75 | 7.5 | |
| 2300 | Rothwell Coal Co., S. Minto.—tipple.. | 1.0 | 14.8 | 31.8 | 53.4 | 1.70 | 7.3 | |
| 2293 | Miramichi Lumber Co., N. Minto— upper seam..... | 0.8 | 10.8 | 32.7 | 56.5 | 1.75 | 6.0 | |
| 2294 | Miramichi Lumber Co.—lower seam... | 0.7 | 23.4 | 29.8 | 46.8 | 1.55 | 9.0 | |
| 2292 | Miramichi Lumber Co., N. Minto— tipple..... | 1.0 | 20.3 | 29.9 | 49.8 | 1.65 | 7.1 | |
| 2296 | Minto Coal Co., N. Minto—upper seam | 1.0 | 12.0 | 33.1 | 54.9 | 1.65 | 6.2 | |
| 2297 | Minto Coal Co., N. Minto—lower seam | 0.8 | 16.4 | 32.3 | 51.3 | 1.60 | 7.7 | |
| 2295 | Minto Coal Co., N. Minto—tipple..... | 1.2 | 20.7 | 29.9 | 49.4 | 1.65 | 7.3 | 0.8 |
| 2402 | Avon Coal Co., S. Minto—upper seam. | 1.3 | 18.1 | 30.5 | 51.4 | 1.70 | 11.3 | |
| 2400 | Avon Coal Co., S. Minto—lower seam. | 1.1 | 16.3 | 30.8 | 52.9 | 1.70 | 10.1 | |
| 2401 | Avon Coal Co., S. Minto—tipple..... | 1.1 | 13.7 | 32.3 | 54.0 | 1.65 | 6.7 | |
| 2403 | McDougal Bros., S. Minto..... | 1.4 | 19.5 | 31.0 | 49.5 | 1.60 | 6.3 | |
| 2541 | McPherson's mine, Coal creek..... | 0.8 | 13.7 | 35.1 | 51.2 | 1.45 | 7.0 | |
| 2542 | From Eighteen creek, S.W. of Minto.. | 1.1 | 10.5 | 32.2 | 57.3 | 1.75 | 2.5 | |
| 2543 | From Eighteen creek, S.W. of Minto.... | 1.4 | 5.8 | 33.8 | 60.4 | 1.80 | 2.0 | |

| No. | Calorific value dry basis | | Coking properties | Fusibility of ash | | | |
|------|---------------------------|-------------------------|-------------------|-------------------|-----------|----------|--------|
| | Calories per grm., gross | B. T. U. per lb., gross | | I. D. | Softening | Fluid | |
| 2289 | 7,080 | 12,740 | Fair to good..... | -25°C. | 1,120°C. | 2,050°F. | +50°C. |
| 2288 | | | Fair to good..... | -20°C. | 1,110°C. | 2,030°F. | +40°C. |
| 2291 | 7,010 | 12,610 | Fair to good..... | -25°C. | 1,130°C. | 2,065°F. | +25°C. |
| 2290 | 6,610 | 11,900 | Good..... | -20°C. | 1,135°C. | 2,075°F. | +35°C. |
| 2298 | | | Fair to good..... | -20°C. | 1,110°C. | 2,030°F. | +60°C. |
| 2299 | | | Fair to good..... | -20°C. | 1,120°C. | 2,050°F. | +50°C. |
| 2301 | 7,210 | 12,930 | Fair to good..... | -40°C. | 1,140°C. | 2,080°F. | +20°C. |
| 2300 | | | Fair to good..... | -30°C. | 1,135°C. | 2,075°F. | +25°C. |
| 2293 | | | Fair to good..... | -15°C. | 1,115°C. | 2,040°F. | +30°C. |
| 2294 | | | Fair..... | -20°C. | 1,135°C. | 2,075°F. | +65°C. |
| 2292 | | | Fair to good..... | -20°C. | 1,110°C. | 2,030°F. | +60°C. |
| 2296 | 7,340 | 13,200 | Fair..... | -20°C. | 1,115°C. | 2,040°F. | +30°C. |
| 2297 | 6,990 | 12,530 | Poor..... | -25°C. | 1,120°C. | 2,050°F. | +25°C. |
| 2295 | 6,550 | 11,790 | Fair to good..... | -20°C. | 1,120°C. | 2,050°F. | +55°C. |
| 2402 | 6,660 | 11,930 | Good..... | | | | |
| 2400 | 6,890 | 12,400 | Good..... | | | | |
| 2401 | 7,240 | 13,030 | Good..... | | | | |
| 2403 | 6,680 | 12,020 | Good..... | | | | |
| 2541 | 7,220 | 12,990 | Good..... | | | | |
| 2542 | 7,470 | 13,440 | Good..... | | | | |
| 2543 | 7,870 | 14,170 | Good..... | | | | |

NEW BRUNSWICK
Gloucester Area

| No. | Description | Moisture, as received | Analysis, dry basis | | | | |
|------|------------------------|-----------------------------|---------------------|---------------------|-----------------|---------------|---------|
| | | | Ash | Volatiles matter | Fixed carbon | Fuel ratio | Sulphur |
| 2485 | Caraquet—Prospect..... | 5.8 | 16.9 | 37.1 | 46.0 | 1.25 | 10.8 |
| 2599 | Caraquet—Mine..... | 7.6 | 22.9 | 34.8 | 42.3 | 1.20 | 11.5 |
| 2600 | Caraquet—Mine..... | 7.0 | 12.2 | 39.1 | 48.7 | 1.25 | 6.2 |
| 2601 | Caraquet—Mine..... | 7.4 | 11.7 | 39.5 | 48.8 | 1.25 | 6.3 |

| No. | Calorific value, dry basis | | Coking properties | Fusibility of ash | | | |
|------|-------------------------------|-------------------------------|-------------------|-------------------|-----------|----------|--------|
| | Calories per gm., gross | B. T. U. per lb., gross | | I. D. | Softening | Fluid | |
| 2485 | 6,280 | 11,300 | Poor..... | -120°C. | 1,200°C. | 2,190°F. | +50°C. |
| 2599 | 5,840 | 10,520 | Poor..... | | | | |
| 2600 | 7,000 | 12,600 | Fair..... | | | | |
| 2601 | 7,090 | 12,760 | Fair..... | -15°C. | 1,170°C. | 2,140°F. | +10°C. |

NOVA SCOTIA

Joggins-Chignecto Area

| No. | Description | Moisture, as rec'd | Analysis, dry basis | | | | | |
|------|---|--------------------------|---------------------|--------------------|-----------------|---------------|---------|----------|
| | | | Ash | Volatile matter | Fixed carbon | Fuel ratio | Sulphur | Nitrogen |
| 2308 | River Hebert Coal Co., River Hebert— mine..... | 3.6 | 13.3 | 35.7 | 51.0 | 1.45 | 5.5 | 1.4 |
| 2309 | River Hebert Coal Co., River Hebert— tipple..... | 3.4 | 21.3 | 33.5 | 45.2 | 1.35 | 6.6 | |
| 2310 | Maritime Coal Railway & Power Co., Ltd., Beech Grove mine, River Hebert—mine..... | 4.1 | 13.7 | 36.8 | 49.5 | 1.35 | 6.7 | |
| 2311 | Maritime Coal Railway & Power Co., Ltd., Beech Grove mine, River Hebert—tipple..... | 4.3 | 17.8 | 36.2 | 46.0 | 1.25 | 5.9 | |
| 2312 | Minudie Coal Co., Ltd., River Hebert— mine..... | 2.6 | 13.7 | 37.7 | 48.6 | 1.30 | 6.1 | |
| 2313 | Minudie Coal Co., Ltd., River Hebert— tipple..... | 2.7 | 19.2 | 35.0 | 45.8 | 1.30 | 6.6 | |
| 2315 | Fundy Coal Co., Ltd., Joggins—mine.. | 3.7 | 16.8 | 35.9 | 47.3 | 1.30 | 7.4 | 1.2 |
| 2314 | Fundy Coal Co., Ltd., Joggins— tipple..... | 3.0 | 20.7 | 35.1 | 44.2 | 1.25 | 7.7 | |

NOVA SCOTIA

Joggins-Chignecto Area—concluded

| No. | Calorific value, dry basis | | Coking properties | Fusibility of ash | | | |
|------|-------------------------------|-------------------------------|-------------------|-------------------|-----------|----------|--------|
| | Calories per gm., gross | B. T. U. per lb., gross | | I.D. | Softening | Fluid | |
| 2308 | 6,700 | 12,060 | Fair to good..... | -20°C. | 1,100°C. | 2,010°F. | +30°C. |
| 2309 | | | Fair to good..... | -15°C. | 1,135°C. | 2,070°F. | +30°C. |
| 2310 | 6,500 | 11,700 | Fair..... | -25°C. | 1,105°C. | 2,020°F. | +85°C. |
| 2311 | | | Fair to good..... | -25°C. | 1,140°C. | 2,080°F. | +20°C. |
| 2312 | 6,770 | 12,190 | Fair to good..... | -40°C. | 1,120°C. | 2,050°F. | +35°C. |
| 2313 | 6,240 | 11,240 | Fair to good..... | -35°C. | 1,125°C. | 2,060°F. | +65°C. |
| 2315 | 6,300 | 11,350 | Fair..... | -60°C. | 1,140°C. | 2,080°F. | +35°C. |
| 2314 | | | Fair to good..... | -25°C. | 1,150°C. | 2,100°F. | +65°C. |

Springhill Area

| No | Description | Mois- ture, as rec'd. | Analysis, dry basis | | | | | |
|------|--|--------------------------------|---------------------|-------------------------|-----------------|---------------|--------------|---------------|
| | | | Ash | Vola- tile matter | Fixed carbon | Fuel ratio | Sul- phur | Nitro- gen |
| 2316 | Cumberland Railway & Coal Co., Ltd., Springhill, No. 2 mine—mine..... | 2.6 | 8.1 | 31.1 | 60.8 | 1.95 | 1.6 | 1.8 |
| 2317 | Cumberland Railway & Coal Co., Ltd., Springhill, No. 2 mine—tipple..... | 2.4 | 11.0 | 30.2 | 58.8 | 1.95 | 1.6 | |
| 2318 | Cumberland Railway & Coal Co., Ltd., Springhill, No. 6 mine—mine..... | 3.1 | 8.0 | 33.4 | 58.6 | 1.75 | 1.6 | 2.4 |
| 2319 | Cumberland Railway & Coal Co., Ltd., Springhill, No. 6 mine—tipple..... | 4.1 | 13.2 | 32.3 | 54.5 | 1.70 | 2.1 | |

| No. | Calorific value, dry basis | | Coking properties | Fusibility of ash | | | |
|------|-------------------------------|-------------------------------|-------------------|-------------------|-----------|----------|--------|
| | Calories per gm., gross | B. T. U. per lb., gross | | I.D. | Softening | Fluid | |
| 2316 | 7,660 | 13,700 | Fair to good..... | -50°C. | 1,240°C. | 2,265°F. | +40°C. |
| 2317 | 7,410 | 13,340 | Good..... | -80°C. | 1,275°C. | 2,325°F. | |
| 2318 | 7,520 | 13,540 | Good..... | -50°C. | 1,210°C. | 2,210°F. | +65°C. |
| 2319 | | | Fair..... | -50°C. | 1,195°C. | 2,185°F. | +85°C. |

Colchester Area

| No. | Description | Mois- ture, as rec'd. | Analysis, dry basis | | | | | Calorific value | |
|-------|--------------------------------------|--------------------------------|---------------------|-------------------------|-----------------|---------------|---------|----------------------------------|-------------------------------|
| | | | Ash | Volat- ile matter | Fixed carbon | Fuel ratio | Sulphur | Calories per gm., gross | B. T. U. per lb., gross |
| | | | | | | | | | |
| 2321 | From near Rivers- dale—lump..... | 1.7 | 21.4 | 11.5 | 67.1 | 5.85 | 5.6 | 6,440 | 11,600 |
| 2321A | From near Rivers- dale—slack..... | 6.9 | 16.5 | 20.0 | 63.5 | 3.20 | 1.1 | 6,250 | 11,260 |

NOVA SCOTIA

Pictou Area

| No. | Description | Moisture, as rec'd. | Analysis, dry basis | | | | | |
|------|--|---------------------|---------------------|-----------------|--------------|------------|---------|----------|
| | | | Ash | Volatile matter | Fixed carbon | Fuel ratio | Sulphur | Nitrogen |
| 2327 | Intercolonial Coal Mg. Co., Westville, Main—mine..... | 2.1 | 10.8 | 26.9 | 62.3 | 2.30 | 0.8 | 2.1 |
| 2326 | Intercolonial Coal Mg. Co., Westville, Scott—mine..... | 2.3 | 20.9 | 25.8 | 53.3 | 2.05 | 1.0 | 1.7 |
| 2329 | Intercolonial Coal Mg. Co., Westville, Main—tipple..... | 1.8 | 14.6 | 26.5 | 58.9 | 2.20 | 0.8 | |
| 2328 | Intercolonial Coal Mg. Co., Westville, Scott—tipple..... | 2.3 | 22.6 | 26.2 | 51.2 | 1.95 | 1.3 | |
| 2328 | Intercolonial Coal Mg. Co., Westville, -9 Main and Scott—tipple..... | 2.1 | 20.3 | 26.3 | 53.4 | 2.05 | 1.1 | |
| 2325 | Acadia Coal Co., Stellarton, Allan Shaft—tipple..... | 1.8 | 13.5 | 30.1 | 56.4 | 1.85 | 0.9 | |
| 2334 | Acadia Coal Co., Stellarton, Albion-Poord seam..... | 2.3 | 13.9 | 31.8 | 54.3 | 1.70 | 0.6 | |
| 2333 | Acadia Coal Co., Albion—Poord seam..... | 2.2 | 13.1 | 30.3 | 56.6 | 1.85 | 1.1 | 2.3 |
| 2332 | Acadia Coal Co., Albion—Cage seam..... | 2.1 | 10.0 | 23.2 | 61.8 | 2.20 | 1.2 | 2.1 |
| 2331 | Acadia Coal Co., Albion—Third seam..... | 1.8 | 12.1 | 23.9 | 59.0 | 2.05 | 1.2 | 1.8 |
| 2335 | Acadia Coal Co.—Acadia No. 1 seam..... | 1.2 | 12.9 | 28.7 | 58.4 | 2.05 | 3.1* | 1.7 |
| 2330 | Acadia Coal Co., Albion bankhead—tipple..... | 2.4 | 12.2 | 30.2 | 57.6 | 1.90 | 0.8 | |
| 2323 | Acadia No. 3 Colliery, Thorburn, Vale or Greener seam—mine..... | 2.7 | 12.2 | 31.7 | 56.1 | 1.75 | 1.1 | 1.8 |
| 2324 | Acadia No. 3 Colliery, Thorburn, Vale or Greener seam—tipple..... | 2.6 | 12.8 | 31.6 | 55.6 | 1.75 | 1.1 | |

*According to the analysis of No. 2330, the average output of Albion bankhead, and to that of a similar sample subsequently received by the Dominion Fuel Board, this sulphur content seems higher than the average.

| No. | Calorific value, dry basis | | Coking properties | Fusibility of ash | | | |
|--------|----------------------------|-------------------------|-------------------|-------------------|-----------|----------|--------|
| | Calories per grm., gross | B. T. U. per lb., gross | | I.D. | Softening | Fluid | |
| 2327 | 7,510 | 13,520 | Good..... | -50°C. | 1,315°C. | 2,400°F. | |
| 2326 | 6,490 | 11,690 | Poor..... | -65°C. | 1,440°C. | 2,625°F. | |
| 2329 | 7,160 | 12,890 | Good..... | -35°C. | 1,240°C. | 2,265°F. | +80°C. |
| 2328 | 6,300 | 11,340 | Poor..... | -25°C. | 1,260°C. | 2,300°F. | +65°C. |
| 2328-9 | 6,550 | 11,790 | | -85°C. | 1,255°C. | 2,290°F. | +65°C. |
| 2325 | | | Fair..... | -60°C. | 1,345°C. | 2,450°F. | +70°C. |
| 2334 | | | Poor to fair..... | -30°C. | 1,145°C. | 2,095°F. | +70°C. |
| 2333 | 7,160 | 12,880 | Poor to fair..... | -100°C. | 1,260°C. | 2,300°F. | +90°C. |
| 2332 | 7,490 | 13,480 | Good..... | 1,370°C. | 1,470°C. | 2,680°F. | |
| 2331 | 7,330 | 13,200 | Fair to good..... | -70°C. | 1,345°C. | 2,450°F. | |
| 2335 | 7,160 | 12,890 | Good..... | -40°C. | 1,330°C. | 2,425°F. | |
| 2330 | 7,170 | 12,900 | Poor..... | -80°C. | 1,235°C. | 2,255°F. | +40°C. |
| 2323 | 7,210 | 12,980 | Fair..... | -50°C. | 1,305°C. | 2,380°F. | +40°C. |
| 2324 | | | Poor..... | -35°C. | 1,310°C. | 2,390°F. | |

NOVA SCOTIA

Inverness Area

| No. | Description | Moisture, as rec'd. | Analysis, dry basis | | | | |
|------|---|---------------------|---------------------|-----------------|--------------|------------|---------|
| | | | Ash | Volatile matter | Fixed carbon | Fuel ratio | Sulphur |
| 2336 | Inverness Railway & Coal Co., Inverness, No. 1 seam—mine..... | 4.3 | 9.2 | 40.9 | 49.9 | 1.20 | 5.7 |
| 2337 | Inverness Railway & Coal Co., Inverness, No. 4 seam—tipple..... | 9.9 | 12.8 | 39.8 | 47.4 | 1.20 | 7.8 |

| No. | Calorific value, dry basis. | | Coking properties | Fusibility of ash | | | |
|------|-----------------------------|-------------------------|-------------------|-------------------|-----------|----------|--------|
| | Calories per grm., gross | B. T. U. per lb., gross | | I.D. | Softening | Fluid | |
| 2336 | | | Poor..... | | 1,145°C. | 2,095°F. | |
| 2337 | | | Non-coking..... | -110°C. | 1,170°C. | 2,140°F. | +40°C. |

Sydney Area

| No. | Description | Moisture, as rec'd. | Analysis, dry basis | | | | | |
|------|---|---------------------|---------------------|-----------------|--------------|------------|---------|----------|
| | | | Ash | Volatile matter | Fixed carbon | Fuel ratio | Sulphur | Nitrogen |
| 2339 | N.S. Steel & Coal Co., Ltd., Jubilee, Indian Cove seam—tipple..... | 3.0 | 8.4 | 36.1 | 55.5 | 1.55 | 4.6 | 1.4 |
| 2338 | N.S. Steel & Coal Co., Ltd., Jubilee, Toronto seam—tipple..... | 2.9 | 13.3 | 37.2 | 49.5 | 1.35 | 6.8 | 1.4 |
| 2340 | N.S. Steel & Coal Co., Ltd., Alexandra, Lloyd's Cove seam—tipple..... | 6.1 | 9.4 | 36.9 | 53.7 | 1.45 | 5.6 | 1.3 |
| 2342 | N.S. Steel & Coal Co., Ltd., Princess, Main seam—tipple..... | 2.2 | 7.0 | 37.3 | 54.8 | 1.45 | 2.0 | 1.5 |
| 2343 | N.S. Steel & Coal Co., Ltd., Florence, Main seam—grab..... | 4.6 | 7.3 | 37.7 | 55.0 | 1.45 | 2.4 | |
| 2341 | N.S. Steel & Coal Co., Ltd., Princess & Florence—grab..... | 2.9 | 8.7 | 36.9 | 54.4 | 1.45 | 2.2 | |
| 2347 | Dominion No. 22, Gowrie—mine..... | 3.4 | 5.5 | 36.7 | 57.8 | 1.55 | 2.1 | |
| 2348 | Dominion No. 22, Gowrie—mine..... | 3.2 | 7.4 | 36.2 | 56.4 | 1.55 | 3.1 | 1.2 |
| 2346 | Dominion No. 22, Gowrie—grab..... | 3.1 | 11.1 | 35.0 | 53.9 | 1.55 | 3.7 | |
| 2352 | Dominion No. 11, Emery—mine..... | 2.6 | 3.8 | 36.8 | 59.4 | 1.60 | 1.7 | 1.5 |
| 2353 | Dominion No. 11, Emery—mine..... | 2.2 | 10.5 | 35.9 | 53.6 | 1.50 | 2.2 | |
| 2354 | Dominion No. 11, Emery—tipple..... | 2.3 | 10.6 | 34.6 | 54.8 | 1.60 | 2.3 | |
| 2355 | Dominion No. 12, Victoria—mine..... | 3.7 | 5.9 | 38.9 | 55.2 | 1.40 | 3.6* | 1.6 |
| 2356 | Dominion No. 12, Victoria—grab..... | 2.0 | 5.3 | 37.6 | 57.1 | 1.50 | 2.2 | |
| 2357 | Dominion No. 16, Lingan—mine..... | 3.0 | 5.6 | 33.5 | 60.9 | 1.80 | 2.2 | 1.5 |
| 2358 | Dominion No. 16, Lingan—grab..... | 2.7 | 6.4 | 34.1 | 59.5 | 1.75 | 3.2 | |
| 2350 | Dominion No. 4, Phalen—mine..... | 1.5 | 9.4 | 33.8 | 56.8 | 1.70 | 4.9 | |
| 2351 | Dominion No. 4, Phalen—mine..... | 1.8 | 6.5 | 35.1 | 58.4 | 1.65 | 2.8 | 1.4 |
| 2349 | Dominion No. 4, Phalen—tipple..... | 1.5 | 9.1 | 34.8 | 56.1 | 1.60 | 4.6 | |
| 2359 | Dominion No. 2, Phalen—mine..... | 1.7 | 6.8 | 33.1 | 60.1 | 1.80 | 2.3 | |
| 2360 | Dominion No. 2, Phalen—grab..... | 1.4 | 10.0 | 33.6 | 56.4 | 1.65 | 4.2 | |
| 2383 | Dominion—slack..... | 3.1 | 8.3 | 34.6 | 57.1 | 1.65 | 2.7 | |
| 2384 | Dominion—slack, washed..... | 3.6 | 5.1 | 35.6 | 59.3 | 1.65 | 2.0 | |
| 2385 | Coke from washed slack..... | 0.2 | 6.4 | 0.9 | 92.7 | 98.5 | 1.7 | |

*This value is almost double the normal sulphur content of Victoria coal.

NOVA SCOTIA
Sydney Area—concluded

| No. | Calorific value, dry basis | | Coking properties | Fusibility of ash | | | |
|------|-------------------------------|-----------------------------|-------------------|-------------------|-----------|----------|---------|
| | Calories per gm., gross | B.T.U. per lb., gross | | I.D. | Softening | Fluid | |
| 2339 | 7,370 | 13,270 | Fair..... | -60°C. | 1,145°C. | 2,090°F. | +65°C. |
| 2338 | 6,880 | 12,390 | Fair..... | -25°C. | 1,120°C. | 2,050°F. | +60°C. |
| 2340 | 6,960 | 12,530 | Poor to fair..... | -40°C. | 1,130°C. | 2,065°F. | +55°C. |
| 2342 | 7,610 | 13,690 | Good..... | -65°C. | 1,190°C. | 2,175°F. | +85°C. |
| 2343 | | | Fair to good..... | -30°C. | 1,135°C. | 2,075°F. | +65°C. |
| 2341 | | | Good..... | -130°C. | 1,240°C. | 2,265°F. | |
| 2347 | 7,720 | 13,890 | Good..... | -50°C. | 1,170°C. | 2,140°F. | |
| 2348 | 7,600 | 13,680 | Fair..... | -60°C. | 1,135°C. | 2,075°F. | |
| 2346 | 7,200 | 12,950 | Good..... | -45°C. | 1,160°C. | 2,120°F. | +100°C. |
| 2352 | 8,070 | 14,530 | Good..... | -45°C. | 1,140°C. | 2,085°F. | +110°C. |
| 2353 | 7,510 | 13,510 | Good..... | -60°C. | 1,215°C. | 2,220°F. | |
| 2354 | | | Good..... | -80°C. | 1,220°C. | 2,225°F. | +90°C. |
| 2355 | 7,630 | 13,740 | Fair to good..... | -50°C. | 1,135°C. | 2,075°F. | +70°C. |
| 2356 | | | Fair to good..... | -50°C. | 1,115°C. | 2,040°F. | +45°C. |
| 2357 | 7,790 | 14,020 | Good..... | -60°C. | 1,135°C. | 2,075°F. | +50°C. |
| 2358 | | | Good..... | -30°C. | 1,085°C. | 1,985°F. | +30°C. |
| 2350 | | | Fair..... | -50°C. | 1,090°C. | 1,995°F. | +60°C. |
| 2351 | 7,850 | 14,130 | Good..... | -55°C. | 1,160°C. | 2,120°F. | +50°C. |
| 2349 | | | Fair to good..... | -25°C. | 1,085°C. | 1,985°F. | +50°C. |
| 2359 | 7,820 | 14,090 | Fair to good..... | -50°C. | 1,140°C. | 2,085°F. | +35°C. |
| 2360 | 7,600 | 13,680 | Fair to good..... | -25°C. | 1,085°C. | 1,985°F. | +70°C. |
| 2383 | 7,620 | 13,720 | Good..... | -55°C. | 1,150°C. | 2,090°F. | +40°C. |
| 2384 | 7,920 | 14,250 | Good..... | -45°C. | 1,150°C. | 2,090°F. | +100°C. |
| 2385 | 7,300 | 13,140 | | -40°C. | 1,155°C. | 2,100°F. | +95°C. |

III

A STUDY OF THE NATURE OF SULPHUR IN COAL AND COKE FROM THE MARITIME PROVINCES

J. H. H. Nicolls

The preceding paper describes a survey of the coal fields of the Maritime Provinces, and contains tables of analyses of the samples collected. If the analyses are compared with those of other Canadian coals it will at once be evident that, as a general rule, the coals of New Brunswick and Nova Scotia contain a higher percentage of sulphur than those from any other part of the Dominion. They have therefore been selected as the most suitable Canadian coals for a study of the various forms of coal sulphur.

One of the principal reasons for the survey was the determination of the coking properties of the coals as a guide to the Dominion Fuel Board in preparing for their subsequent commercial scale tests on the production of household coke from Canadian coal. It is essential that the sulphur in metallurgical coke be low, and even with household coke there is a limit above which it is undesirable, so that the question of the sulphur in coal, and more particularly of that in the coke produced from it, is of considerable import. This is particularly true in dealing with the Maritime Provinces, which produce 50 per cent to 60 per cent of the coking coal mined in Canada, the remainder coming from the extreme west, where the country is thinly populated, and where there is little industrial development as compared with that in Ontario and Quebec.

In this paper attention has been principally directed to the sulphur in coal, since, as will be shown later, the amount of sulphur in a coke is chiefly dependent upon the total quantity of sulphur in the coal from which it was made. There is however no reason why, in many cases, some of the sulphur in coal should not be removed before coking. Coal sulphur may be primarily divided into two groups, namely organic and inorganic, which, as will be shown later, may again be sub-divided. Organic sulphur forms part of the coal proper, and probably originated in the vegetable and animal substances from which the coal was metamorphosed. It cannot be removed by any processes of mechanical separation as it is comparatively evenly distributed among the coal constituents. This was shown by Wheeler and Clark¹, who employed pyridine and chloroform to separate coal into two forms of cellulosic compound and one resinic compound, and found about the same amount of sulphur in each case. On the other hand the inorganic sulphur cannot be considered to form part of the coal proper. It consists mainly of iron pyrites or marcasite, and has been probably formed from the interaction of the iron of the mineral matter in the coal with percolating water containing hydrogen sulphide; it may also have been formed by some combination of iron with the organic sulphur. A large percentage of it can be removed by such a mechanical separation as washing.

In the course of investigations carried out at Urbana, Ill., with several coals from various parts of the United States, Yancey² and his respective colleagues, Parr and Fraser, have shown that, although in comparing various

¹ Jour. Chem. Soc.,—103, 1704-1913.

² Jour. Ind. Eng. Chem., 13, 1, 35—1921, 16, 5, 501—1924.

coal deposits it may be stated that high inorganic is generally accompanied by high organic sulphur, the same condition does not hold good throughout any particular coal bed. They have clearly shown that although the organic sulphur varies very little throughout a bed, the inorganic is continually altering. Most of the pyritic sulphur occurs as crystals segregated in lumps or nodules, which are readily removable by washing, while a certain amount occurs as finely disseminated microscopic globules distributed throughout the coal. By means of float and sink solutions Yancey and Parr have estimated this finely disseminated sulphur, and have proved by a further series of tests that this, together with the organic sulphur, cannot be removed by coal washing.

Pyrites has recently been given consideration by Graham as a cause of spontaneous combustion. It was for many years judged to be the principal cause of this wastage of storage coal, but the theory has been modified or practically abandoned during recent years by various authorities. Graham has covered quite thoroughly the question of spontaneous combustion in a recent journal article,¹ and has indicated that the theory which now seems generally accepted holds good. In other words the lower grade, or humic coals, with a high moisture and oxygen content, are more liable to spontaneous combustion than the higher grade coals when stored under similar conditions, and the higher grade a coal is the less liable it is to spontaneous combustion. Segregation of sizes, and unsuitable storage conditions prove a big factor in causing spontaneous combustion, whereas massive or lump pyrites has little or no effect. On the other hand, Graham has clearly demonstrated that finely disseminated pyrites is very liable to cause spontaneous combustion. Unfortunately the present undertaking does not include the determination of that form of pyrites, but it seems possible that some of the high sulphur coals from the Maritime Provinces may contain pyrites in a state of fine division, and therefore be liable to spontaneous combustion.

To the best of the writer's knowledge, the only previous determination of the forms of sulphur in Nova Scotia coal was carried out in 1909, or thereabout, by M'Callum, of Halifax.² He wished to follow the changes in organic sulphur throughout the coking process, and for this purpose selected "a typical Nova Scotia coking coal." This was probably one of the Sydney coals. M'Callum separated the coal into fractions by means of solutions of calcium chloride of different densities, and found that the sulphur in the lightest fraction was 85 per cent organic, whereas that in the heaviest fraction was over 60 per cent inorganic. He called all the sulphur "pyritic" that would combine with the total iron to form pyrites, and the difference between this and the total sulphur he called "organic." In other words, as he himself says, he assumed that no sulphates were present, and that the amount of iron combined as silicate was negligible. He coked the various fractions and determined the sulphur contents of the cokes, and from the relations between these and the coal sulphurs determined the percentages of volatile and fixed sulphur in the various fractions. The amount of volatile sulphur seemed to increase with the quantity of organic sulphur, but M'Callum's figures were irregular and indefinite, and indicate little beyond the fact that, contrary to a theory then largely accepted, the volatile sulphur is derived from the organic as well as the pyritic sulphur.

¹ Jour. Soc. Chem. Ind. —43, 14, 79T—1924.

² Chemical Engineer (U.S.)—11, 1, 27—1910.

The forms in which sulphur occurs in coal and coke have recently been given intensive study by A. R. Powell, for several years on the staff of the U.S. Bureau of Mines. In a bulletin published in collaboration with Parr in 1919¹ he defined the principal forms in which sulphur occurs in coal, and devised methods for their determination. These methods have not been appreciably altered during subsequent years, and have been employed in the present investigation. Powell was principally interested in the correct determination of the forms of inorganic sulphur, more particularly pyritic (iron pyrites and marcasite), as the sulphate was generally unimportant. He showed that the calculation of the pyritic sulphur from the total iron, even after allowance had been made for iron sulphate, was liable to cause a serious error on account of the silicate iron present. By means of experiments with various strengths and mixtures of inorganic acids he gradually determined, by a process of elimination, what strengths of acid would dissolve the various inorganic constituents. Powell also exposed coal to the action of various organic solvents, and eventually deduced methods for determining the organic sulphur forms.

The result of these investigations may be summed up in the following list of sulphur forms in coal, with their solubilities.

Inorganic:

Sulphate—Soluble in 3 per cent hydrochloric acid at 60° C.

Pyritic—Soluble in nitric acid (Sp. Gr. 1.12) at room temperature (this also dissolves sulphate).

Organic:

Resinic—Soluble in phenol.

Humic—Soluble in strong ammonia water after treatment with strong nitric acid (this also dissolves most of, if not all, resinic).

Generally speaking the sulphates in coal are of little account, but Powell showed that pyrites is oxidised to iron sulphate on standing. This oxidation is small as a general rule, but takes place in closed, as well as open, vessels. Powell therefore concluded that bacteria might assist oxidation, and his experiments, while not entirely confirmatory, certainly did not disprove this theory. In an extreme case the sulphate sulphur in a coal with 5 per cent total sulphur increased in 2 years from 0.01 per cent to 1.31 per cent, and that in a tightly stoppered flask. Therefore, in considering the sulphate in the samples analysed during the present investigation, it should be borne in mind that most of these had stood for from 9 months to a year in a finely ground condition before being analysed for the sulphur forms.

In connexion with the determination of pyritic sulphur Powell showed that aqua regia, and to a certain extent concentrated nitric acid, attack organic sulphur. He also proved that attempts to increase the rate of solution, either by boiling or exposing to a steam bath, gave too high results. His conclusion was that "any attempt to hasten the process by heat would result in the oxidation of some of the organic sulphur."

By means of analyses of coals and of the cokes made from them Powell endeavoured to follow each form of sulphur through the coking process. His conclusions were interesting, but, as he has since somewhat modified them, they will not be detailed here. He decided that the sulphur in

¹ Univ. of Illinois Bull. No. 111—Vol. 16, No. 34—1919.

laboratory coke was partly sulphide, while the remainder was probably in combination with carbon. This last was very resistant to strong acids, oxidising agents and heat, but readily gave up sulphur as hydrogen sulphide when treated with nascent hydrogen.

Subsequently Powell¹ followed the sulphur of a variety of coals through all the stages of the coking process. Five-gramme samples of coal were heated to various temperatures in a small electric tube furnace in an atmosphere of carbon dioxide, and the products of distillation collected for the determination of sulphur. The coal sulphur was determined by the methods previously outlined, the sulphide sulphur in the coke by evolution as hydrogen sulphide, the sulphates from the residual solution in the evolution flask and the remaining coke sulphur calculated by difference. By this method of coking only the primary effects were obtained, and not the secondary effects due to the passage through the coke of the hot gaseous products of distillation.

It was noted that between 400° and 500° C. pyrites and marcasite rapidly decomposed into ferrous sulphide and pyrrhotite, the latter a magnetic solution of a small percentage of sulphur in ferrous sulphide, while the sulphur set free was converted into hydrogen sulphide. Sulphates were reduced to sulphides at 500°C. or lower. Organic sulphur began to decompose to form hydrogen sulphide at 300°C., and its percentage in the coking mass continued to decrease according to the amount of pyritic sulphur in the coal. At about 400°C. the original organic sulphur was replaced by a very stable form of "organic" sulphur, which did not respond to the nitric acid plus ammonia test for organic sulphur. With a coal containing a high percentage of inorganic sulphur the "organic" sulphur in the coke increased above 400°C. due to transference of some inorganic sulphur to the "organic" form; with one composed almost entirely of organic sulphur the "organic" sulphur in the coke was found to decrease with rise of temperature, and in some extreme cases showed a tendency to form sulphide sulphur in the coke. In other words, there appeared to be an equilibrium between the hydrogen sulphide evolved and the total sulphur in the coal, so that, in the absence of inorganic, organic sulphur was gradually decomposed. Powell therefore decided that the amount of sulphur in manufactured coke is more dependent upon the total sulphur in the coal from which it was made than upon the relative amounts of organic and inorganic sulphur in that coal. He further decided that the nature of the coal substance proper will largely determine the relation between its total sulphur and that in the coke produced from it, another principal cause being the composition and amount of circulation of the by-product gases through the coke oven.

In a paper² following directly upon the previous one Powell described experiments carried out to determine the desulphurizing action of hydrogen on coke. It had been previously shown that the action of nascent hydrogen removed a considerable amount of sulphur from finished coke, and Powell attempted to improve upon this by passing hydrogen through the tube during the coking process. He used the apparatus described in the previous paper, and heated to a final temperature of 1,000° C., which was maintained for some hours. He found that the passage of hydrogen, which was more effective if slightly moistened, caused the complete decompo-

¹ Jour. Ind. Eng. Chem. 12, 11, 1069—1920.

² Jour. Ind. Eng. Chem. 12, 11, 1077—1920.

sition of pyritic sulphur at 500°C., after which the "organic" sulphur remaining was rapidly decomposed to form hydrogen sulphide. It is upon the latter reaction that the whole desulphurization process is based, and Powell found that hydrogen would remove as much as 80 to 90 per cent of the coke sulphur after 3 hours at 1,000°C., and that without causing any deterioration in the coke. Steam had been suggested for the removal of sulphur and would be equally effective, though with the loss of considerable of the carbon in the coke. Powell further demonstrated that gas containing 50 per cent hydrogen, and of approximately the composition of lean oven gas, would effect a considerable reduction of sulphur during the coking process, though its effect would not be greater than 50 to 75 per cent of that of hydrogen for a corresponding period of time. Prolonged treatment would probably bring about as great a reduction of sulphur as that caused by hydrogen.

Monkhouse and Cobb¹ in the course of investigations of the actions of various gases upon finished coke showed that nitrogen liberated very little sulphur even when a temperature of 1,000° C. was attained. Hydrogen liberated sulphur freely up to and at 800° until about 50 per cent was volatilised, after which the reaction slackened and was very prolonged. On heating to 900° the reaction again became rapid until at 1,000°C. 94 per cent of the sulphur was removed from soft (500°C.) coke. Steam caused the same effect at 800°. With the higher temperature, or harder cokes, considerably less sulphur was removed under similar conditions.

Jolly and Wheeler² investigated the distribution of sulphur in the distillation products of coal coked in a porcelain tube in a current of nitrogen. They divided the distillation into two parts, namely that below 560°C., when little or no hydrogen was evolved from the coal, and that from 560° to 1,000°C., when considerable hydrogen was evolved. They decided that most of the hydrogen sulphide was evolved below 560° when little hydrogen was present. Slow heating, such as would hold in coke oven practice, caused very little increase of hydrogen sulphide evolved below 560° from a coal high in organic sulphur, but increased the amount liberated from one containing a great deal of pyritic sulphur. They also showed that the addition of anthracite to a coking coal would furnish sufficient hydrogen to remove a large proportion of the sulphur during the coking process.

Apart from Powell, no other authority seems to have published more information regarding sulphur and its reactions when heated with carbon, as a guide in coking practice, than Wibaut of Amsterdam. In his earlier work, in collaboration with Stoffel³, he separated 30-mesh coal into two fractions by means of a mixture of benzene and carbon tetrachloride (Sp. Gr. 1.3) and obtained almost entirely organic sulphur in the lighter fraction. He determined sulphates by boiling the coal with 10 per cent hydrochloric acid. The residue from this determination was calcined and then digested with strong hydrochloric, after which the iron in solution was determined. This iron was considered to be that combined as ferric sulphide, and from it the pyrite and marcasite sulphur was calculated. The total inorganic sulphur was determined by treating the finely powdered coal in suspension in water with successive portions of bromine, boiling

¹ Trans. Inst. Gas. Eng.—1922.

² Gas World (Coking Section)—p. 11—May 3rd, 1924.

³ Rec. Trav. Chim.—38, 132—1919.

and determining the total sulphur present as barium sulphate. This method for inorganic sulphur gave comparatively satisfactory results, though a small amount of organic sulphur was oxidised and a little of the ferric sulphide resisted oxidation.

Comparatively recently Wibaut¹ made a study of the behaviour of carbon and sulphur when heated together. He heated carbon in various forms with sulphur in a combustion tube in a current of nitrogen to temperatures averaging 900° to 1,000°C. There were two forms of sulphur produced in the residues, one of which was removed by highly heating in a vacuum or by treating with oxidising agents, and was soluble to a certain extent in toluene. This form Wibaut considered to be free sulphur absorbed in the residue. The other form of sulphur was resistant to the treatments just described, but was, in whatever tests were made, expelled by the action of hydrogen at 800°C. Residues prepared from wood charcoal contained a considerable amount of absorbed sulphur, those from sugar charcoal purified by heating in a current of chlorine contained less of this sulphur, while those prepared from sugar charcoal purified by heating in a vacuum contained only the form of sulphur resistant to oxidising agents. Wibaut described this form as a "physico-chemical complex," and decided that the carbon and sulphur were chemically combined. Subsequent work by Powell seems to have proved otherwise, in fact it seems as if most of Wibaut's "complex" must have been "adsorbed free sulphur."

Powell has recently² made a study of the sulphur in coke and similar substances by application of the phase rule to the composition of the residue obtained by heating together sugar charcoal and sulphur and to coke itself. He has demonstrated that the so-called carbon-sulphur compound is a solid solution of sulphur in carbon, or that sulphur is held by the carbon so as to be indistinguishable from a solid solution. This form, together with adsorbed free sulphur, is present in the residue from the reaction of sugar charcoal and sulphur. Coke made by rapid heating in the laboratory contains in addition ferrous sulphide. Prolonged heating causes the adsorbed sulphur to pass gradually into the solid solution. In freshly manufactured coke the adsorbed free sulphur is not present.

However it is almost impossible to prevent some oxidation during the cooling of coke, and some of the ferrous sulphide is almost invariably oxidised, between 500° and 300°C., to ferric oxide and sulphur. This sulphur, which is only a small percentage of the total sulphur, represents what is removable from finished oven coke by a current of hydrogen, whereas the sulphur in coke prepared by rapid heating is largely in the adsorbed form and is therefore readily removable. Because of the difference between the forms of sulphur, oven gas containing 50 per cent of hydrogen, which removed a large percentage of the sulphur during a rapid coking process in the laboratory, would only remove 20 per cent of the sulphur when passed through a regulation oven during coking.

EXPERIMENTAL

The methods employed for the analyses of coals from the Maritime Provinces are fully described in Powell's "Analysis of Sulphur Forms in Coal"³ and will not be given here in great detail. Only the inorganic forms of sulphur, sulphate and pyritic, have been determined, and the total organic

¹ Rec. Trav. Chim.—41, 153—1922.

² Jour. Am. Chem. Soc.—45, 1, 1—1923.

³ U.S. Bur. of Mines, Tech. Pap. No. 254—1921.

sulphur has been taken to be the difference between the combined inorganic and the total sulphur as determined by Eschka's method. No difficulty was experienced with these methods, and better check determinations were made than with Eschka's method.

The methods are as follows:—

Sulphate—5 grammes of coal were treated in a 600 c.c. beaker with 300 c.c. of 3 per cent hydrochloric acid at a temperature of about 60°C., with occasional stirring. The temperature was maintained by standing the beaker on top of a closed water bath. At the end of 40 hours the contents of the beaker were filtered, and the sulphates precipitated from the almost neutralised filtrate by means of a 10 per cent solution of barium chloride and determined as barium sulphate.

Pyritic—1 gramme of coal was treated in a 150 c.c. beaker with 80 c.c. of nitric acid (Sp. Gr. 1.12). The contents of the beaker were stirred occasionally, and filtered at the end of 24 hours. 2 c.c. of concentrated hydrochloric acid were added to the filtrate, which was then evaporated to dryness. The residue was dissolved in 25 c.c. of water and 5 c.c. of concentrated hydrochloric, and the solution poured into an excess of ammonia. The whole was then brought to a boil, after which the precipitated hydrates were filtered, and washed well with hot water. The filtrate was acidified with hydrochloric acid, after which the sulphur was precipitated by means of the barium chloride solution. The barium sulphate thus obtained represented both pyritic and sulphate sulphur, and the former was calculated by difference.

Powell recommends the determination of iron in each case as a check on the sulphur, but this was not done in the present instance. However, the iron was removed before precipitating pyritic sulphur for fear of contaminating the barium sulphate. This was not considered a serious factor with sulphate sulphur on account of the lesser amount of iron dissolved, any contamination from which would cause almost no error when distributed over 5 grammes of coal.

Yancey and Parr¹ employed a more rapid method for the determination of sulphates in coal. They employed a slightly stronger solution of hydrochloric acid (20 c.c. diluted to 200 c.c. with water) and heated for 2 hours at 80°-90°C., instead of for 40 hours at 60°. Analyses made during the present investigation showed that this method gave results slightly lower than those obtained by the 40-hour treatment, though the difference was not greater than 0.02 per cent of the coal. Although the two methods agree so closely, analyses reported were all carried out by the slower method except that of sample No. 2342.

The same investigators determined the effect of heating coal for 1 hour with nitric acid (Sp. Gr. 1.12) instead of the 24-hour cold treatment for the determination of pyritic sulphur. They employed temperatures of 65° and 90°C., and decided that organic sulphur was attacked to a certain extent at 90° but not at 65°C. In order to ascertain whether this rapid method were advisable three of the coal samples were heated for 1 hour at a temperature of about 75°C., and never higher than 80°. The following results were obtained:—

¹Ind. Eng. Chem.—16, 5, 501—1924.

| Sample No. | Total sulphur | Pyritic-slow | Pyritic-rapid |
|------------|---------------|--------------|---------------|
| 2337 | 7.10 | 3.04 | 3.40 |
| 2339 | 4.52 | 2.92 | 3.07 |
| 2341 | 2.20 | 1.26 | 1.32 |

Powell concluded in the course of his first investigation that any attempt to hasten the action of nitric acid by heat was inadvisable, and the above figures confirm his conclusion, at any rate in so far as coals of high sulphur content are concerned. Accordingly the slower method was adhered to throughout this investigation.

The following table of analyses shows the percentage of sulphur in each coal distributed between the three forms determined, and also the percentage of the total sulphur represented by each form of sulphur. In some instances the figures for total sulphur will appear not to be in accord with those reported in the previous paper, but in such cases the differences are due to alteration in the moisture content of the coal. It will be observed that the analyses are reported to the second place of decimals, which is contrary to the usual practice in these laboratories. This was necessitated by the very small amounts of sulphate sulphur.

| No. | Description | Sulphur determined | | | | Percentages of total sulphur | | |
|------|---|--------------------|---------|---------|-------|------------------------------|---------|---------|
| | | Sulphate | Pyritic | Organic | Total | Sulphate | Pyritic | Organic |
| 2289 | South Minto, N.B. | 0.28 | 4.74 | 1.81 | 6.83 | 4.1 | 69.4 | 26.5 |
| 2642 | North Minto | 0.04 | 5.36 | 1.37 | 6.77 | 0.6 | 79.2 | 20.2 |
| 2485 | Caraquet | 0.42 | 7.88 | 1.92 | 10.22 | 4.1 | 77.1 | 18.8 |
| 2308 | River Hebert, N.S. | 0.94 | 2.18 | 2.28 | 5.40 | 17.4 | 40.4 | 42.2 |
| 2315 | Joggins | 1.26 | 3.58 | 2.34 | 7.18 | 17.5 | 49.9 | 32.6 |
| 2318 | Springhill | 0.06 | 0.60 | 0.84 | 1.50 | 4.0 | 40.0 | 56.0 |
| 2335 | Acadia No. 1, Stellarton | 0.14 | 1.53 | 1.38 | 3.05 | 4.6 | 50.2 | 45.2 |
| 2330 | Albion bankhead, Stellarton | Trace | 0.07 | 0.69 | 0.76 | Trace | 9.2 | 90.8 |
| 2337 | Inverness | 1.63 | 3.04 | 2.43 | 7.10 | 23.0 | 42.3 | 34.2 |
| 2339 | Sydney Mines, Indian Cove | 0.45 | 2.92 | 1.15 | 4.52 | 10.0 | 64.6 | 25.4 |
| 2338 | Sydney Mines, Toronto | 0.72 | 4.43 | 1.43 | 6.58 | 10.9 | 67.4 | 21.7 |
| 2340 | Sydney Mines, Lloyd's Cove | 0.91 | 2.28 | 2.07 | 5.26 | 17.3 | 43.3 | 39.4 |
| 2342 | Sydney Mines, Princess, Main | 0.12 | 1.13 | 0.77 | 2.02 | 5.9 | 56.0 | 38.1 |
| 2575 | Sydney Mines, Princess, Main | 0.04 | 0.37 | 0.57 | 0.98 | 4.1 | 37.7 | 58.2 |
| 2341 | Sydney Mines, Princess and Florence, Main | 0.21 | 1.26 | 0.73 | 2.20 | 9.5 | 57.3 | 33.2 |
| 2348 | Dominion No. 22, Gowrie | 0.25 | 1.68 | 1.07 | 3.00 | 8.3 | 56.0 | 35.7 |
| 2352 | Dominion No. 11, Emery | 0.14 | 0.67 | 0.87 | 1.68 | 8.3 | 39.9 | 51.8 |
| 2356 | Dominion No. 12, Victoria | 0.11 | 1.32 | 0.68 | 2.11 | 5.2 | 62.6 | 32.2 |
| 2576 | Dominion No. 12, Victoria | 0.03 | 0.28 | 0.56 | 0.87 | 3.4 | 32.2 | 64.4 |
| 2357 | Dominion No. 16, Lingan | 0.16 | 1.10 | 0.88 | 2.14 | 7.5 | 51.4 | 41.1 |
| 2349 | Dominion No. 4, Phalen | 0.24 | 2.83 | 1.47 | 4.54 | 5.2 | 62.4 | 32.4 |
| 2359 | Dominion No. 2, Phalen | 0.20 | 1.20 | 0.88 | 2.28 | 8.8 | 52.6 | 38.6 |
| 2383 | Dominion slack | 0.28 | 1.42 | 0.92 | 2.62 | 10.7 | 54.2 | 35.1 |

It will be noted that in nearly all these coals pyritic sulphur is high, though in the few instances where the total sulphur is low it consists mainly of the organic form. The sulphur in 65 per cent of the coals is 60 per cent or more inorganic, a great part of which could be removed by washing. However it should be borne in mind that the investigation was concentrated on high sulphur coals, and few analyses were made of coals from the

Springhill and Pictou areas where the sulphur content is low. The sulphur in all the New Brunswick coals is composed largely of pyrites, as is that in coals from the Sydney area, more especially, apparently, in those from Sydney Mines.

The New Brunswick coals clearly demonstrate that high pyritic sulphur is not necessarily accompanied by high sulphate. An attempt was therefore made to identify some of the sulphate sulphur as calcium sulphate by correlating it with the amount of calcium oxide in the coal ash. Only a few ash analyses were available, and the lime in these showed no particular relation to the sulphates. The ash in River Hebert coal contains a considerable amount of calcium oxide, which is in agreement with the quantity of sulphates obtained, but the Phalen coal also contains a large percentage of calcium oxide, and yet not very much sulphate sulphur. Unfortunately no ash analysis was available for the Inverness coal which contains so much sulphate, and incidentally was freshly mined when sampled. It will be noted that most of the Sydney Mines coals contain a considerable amount of sulphate sulphur. All these samples, with the exception of No. 2575, were taken from storage above ground, though presumably only Nos. 2338-9 had been stored for any length of time, so that it seems probable that most of these coals normally contain a relatively high percentage of sulphate sulphur.

As has been previously stated, pyritic sulphur can be largely removed by washing. The following analyses show the effect of washing an average sample of Dominion slack in the Baum washer at the Sydney coke ovens. In addition there are given the figures obtained by washing a Minto coal on a Wilfley table belonging to the Ore Dressing Division. In order to indicate how large an amount of impurities was removed the ash content is given in each case.

| No. | Description | Ash, dry basis | Sulphur determined | | | | Percentages of total sulphur | | |
|------|------------------------|----------------|--------------------|---------|---------|-------|------------------------------|---------|---------|
| | | | Sulphate | Pyritic | Organic | Total | Sulphate | Pyritic | Organic |
| 2383 | Dominion, slack..... | 8.3 | 0.28 | 1.42 | 0.92 | 2.62 | 10.7 | 54.2 | 35.1 |
| 2384 | Dominion, Baum..... | 5.1 | 0.24 | 0.82 | 0.95 | 2.01 | 11.9 | 40.8 | 47.3 |
| 2642 | North Minto..... | 21.5 | 0.04 | 5.36 | 1.37 | 6.77 | 0.6 | 79.2 | 20.2 |
| 2642 | North Minto, Wilfley.. | 11.8 | 0.04 | 3.40 | 1.80 | 5.24 | 0.8 | 64.8 | 34.4 |

The methods employed for the analyses of sulphur forms in coke were worked out by Powell¹ following his phase rule study, and are briefly as follows:—

Sulphide—1 or 2 grammes (according to the percentage of sulphur present) of coke of 35-mesh size or smaller were placed in a 300 c.c. Erlenmeyer flask (a 200 c.c. flask is recommended, but one was not available in the present case) and covered with 25 c.c. of water. The flask was closed and connected to an absorption bottle containing an ammoniacal solution of cadmium chloride, after which hydrogen was passed through the apparatus. 25 c.c. of strong hydrochloric acid were then run from a stoppered funnel into the flask, the contents of which were gradually heated to the boiling point, and so maintained for some little time in order to expel the last of the hydrogen sulphide. The contents of the absorption

¹ Ind. Eng. Chem. 15, 9, 951—1923.

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bottle were acidified with hydrochloric acid, and the liberated hydrogen sulphide titrated with iodine solution. The iodine had previously been standardised by a similar evolution method, using steel with a standardised percentage of sulphur.

Sulphate—The contents of the Erlenmeyer flask from the sulphide determination were filtered, and barium chloride added to the filtrate. The barium sulphate precipitated represented sulphate sulphur.

Free sulphur—The same amount of coke as was previously used was weighed into a porcelain boat and placed in a tube in a combustion furnace. While the furnace was still cold hydrogen was passed through until all other gases were removed, after which the outlet was closed. The furnace was now brought to red heat, and so maintained for some little time. Periods of 10, 15 and 20 minutes from the time of lighting the furnace all gave the same result. During the heating and subsequent cooling of the coke a slight pressure of hydrogen was maintained in the furnace, and this was best brought about by placing a water seal containing an inch or so of water between the furnace and the source of hydrogen. As soon as the boat was cool enough to handle (about 100°C.) it was removed from the furnace, and its contents rapidly transferred to an Erlenmeyer flask. The evolution method was then carried out as in the determination of sulphide sulphur. In this case the sulphur evolved represented the total of sulphide, sulphate and free or adsorbed sulphur, and the last named was calculated by difference.

Sulphur in solid solution—This was calculated by subtracting the sum of the above three forms of sulphur from the total sulphur as determined by Eschka's method.

There follows the analysis of sample No. 2385, coke made from the Sydney washed coal No. 2384.

| | Per cent in coke | Per cent of total sulphur |
|--|---------------------|------------------------------|
| Sulphide (ferrous and other metallic sulphides)..... | 0.59 | 34.5 |
| Sulphate..... | Nil | Nil |
| Free sulphur..... | 0.13 | 7.6 |
| Sulphur in solid solution..... | 0.99 | 57.9 |

It may be mentioned here that only 0.29 per cent of sulphur was dissolved from the above coke when the rapid method for the determination of pyritic sulphur in coal was employed.

In the Sydney coke the sulphide sulphur is probably a little higher than is usual in metallurgical coke, but nevertheless the above figures demonstrate that the greater part of the sulphur in such coke is present in solid solution in the carbon.

SUMMARY

1. In the first part of this report there is given a review of the recent literature regarding the nature of sulphur in coal and coke, its determination, and the changes that occur or may happen during the coking process. In the second part there is given a description of the application of the information obtained from the review to experimental work on coals and coke from the Maritime Provinces.

2. Conclusions drawn from a study of the literature are as follows:

(a) Coal sulphur is primarily divided into organic and inorganic; the latter is sub-divided into pyritic and sulphate sulphur.

(b) A large percentage of the pyritic sulphur, except that occurring in a finely disseminated condition, can be removed by washing; organic sulphur cannot be so removed.

(c) Sulphate sulphur is, generally speaking, of little account, though prolonged storage may considerably oxidise pyritic sulphur.

(d) The sulphur in a coke is more dependent upon the total amount of sulphur in the coal from which it was made than upon the relative quantities of each form of sulphur present in that coal.

(e) Hydrogen, and to a lesser extent coke oven gas containing 50 per cent hydrogen, will remove nearly all the sulphur from a coke by rapid heating in the laboratory when passed through the retort during the coking process. On the other hand, when the reaction is applied to the slower commercial process, not more than 20 per cent of the sulphur is removed.

(f) Nitrogen will only remove a very little sulphur from coke. Steam will remove the sulphur more readily than hydrogen, but with a considerable loss of carbon. Anthracite if mixed with a coking coal will produce enough hydrogen to remove some of the sulphur during carbonization.

(g) The harder the coke the less readily will its sulphur be removed by any of the above-mentioned gases.

(h) Sulphur may occur in four forms in metallurgical coke, namely:

(1) sulphide, (2) sulphate, (3) adsorbed or free sulphur, principally from oxidation of ferrous sulphide, (4) sulphur in solid solution.

3. Experimental work was confined to 23 coals from the Maritime Provinces, most of which contained a high percentage of sulphur. The sulphur in 15 of these coals was found to be 60 per cent or more inorganic; furthermore the sulphur in 14 of these was 50 per cent or more pyritic. The New Brunswick coals contained a very high percentage of pyrites. Washing ought to remove most of this pyritic sulphur.

The Joggins, Inverness and Sydney Mines coals were found to contain a considerable amount of sulphate sulphur. Very little of this could be identified as calcium sulphate.

Coals containing a low percentage of sulphur had usually very little inorganic sulphur.

Analyses of Sydney and Minto coals before and after washing showed that only pyrites was removed. A considerable amount of this was removed by a Baum washer from Sydney slack, while the amount washed from Minto coal on a Wilfley table was not as large as anticipated.

An analysis of coke from the Sydney washed coal showed that most of the sulphur was present in solid solution, the greater part of the remainder being combined with iron as ferrous sulphide.

IV

GASOLINE SURVEY FOR 1923

ANALYSES OF GASOLINE SAMPLES COLLECTED IN TEN CITIES OF CANADA DURING 1923

P. V. Rosewarne

In order to make available more information concerning the gasoline sold in Canada, a survey was made in August 1923. In a previous report¹ the results of a survey of the gasoline sold in the city of Ottawa were given. The plan adopted for that survey has been expanded to include the larger cities of Canada.

COLLECTION OF SAMPLES AND METHODS OF EXAMINATION

Accordingly, 48 samples of gasoline were collected² during August, 1923, from the wholesalers or distributors in the following cities:—Quebec, Montreal, Ottawa, Toronto, London, Winnipeg, Regina, Calgary, Edmonton, Vancouver. These samples were tested for distillation range, iodine value, and specific gravity. The distillation range was determined according to the method recommended by the United States Bureau of Mines.³ The iodine value was determined by the Hanus method⁴ and the specific gravity was obtained by means of the Westphal balance.

RESULTS OF ANALYSES

The results obtained are given in Table I.

TABLE I
Results of Analyses
QUEBEC, QUE.

| Lab. No. | Distillation Range | | | | | Recover- ed % | Iodine value | Spec. gravity |
|-----------|--------------------|------------|------------|------------|---------------------|---------------------|-----------------|------------------|
| | 1st drop °F. | 20% °F. | 50% °F. | 90% °F. | End point °F. | | | |
| 2374 | 99 | 189 | 257 | 372 | 432 | 97.0 | 23 | 0.734 |
| 2375 | 109 | 171 | 219 | 325 | 397 | 97.2 | 9 | 0.719 |
| 2376 | 111 | 185 | 248 | 345 | 397 | 97.1 | 33 | 0.728 |
| 2377 | 118 | 199 | 272 | 381 | 432 | 97.0 | 25 | 0.739 |
| 2378 | 163 | 219 | 255 | 354 | 439 | 97.9 | 14 | 0.746 |
| Average.. | 120 | 193 | 250 | 355 | 419 | 97.2 | 21 | 0.733 |

¹ Mines Branch, Summary Report of Investigations, 1922.

² The writer wishes to acknowledge the hearty support and co-operation of the Engineering Departments of the various cities in taking the samples.

³ U.S. Bureau of Mines, Technical Paper 208.

⁴ Ellis and Meigs, "Gasoline and other Motor Fuels."

TABLE I—Continued
Results of Analyses—Continued
MONTREAL, QUE.

| Lab. No. | Distillation Range | | | | | Recover- ed % | Iodine value | Spec. gravity |
|-----------|--------------------|------------|------------|------------|---------------------|---------------------|-----------------|------------------|
| | 1st drop °F. | 20% °F. | 50% °F. | 90% °F. | End point °F. | | | |
| 2556 | 120 | 203 | 270 | 378 | 432 | 97.1 | 29 | 0.740 |
| 2557 | 113 | 179 | 232 | 331 | 396 | 97.3 | 5 | 0.718 |
| 2558 | 113 | 192 | 272 | 370 | 423 | 97.0 | 29 | 0.739 |
| 2559 | 153 | 201 | 275 | 374 | 430 | 97.5 | 28 | 0.740 |
| 2560 | 111 | 192 | 275 | 397 | 437 | 97.1 | 4 | 0.751 |
| 2561 | 154 | 221 | 280 | 372 | 424 | 97.6 | 3 | 0.756 |
| Average.. | 127 | 198 | 267 | 370 | 424 | 97.3 | 16 | 0.741 |

OTTAWA, ONT.

| | | | | | | | | |
|-----------|-----|-----|-----|-----|-----|------|----|-------|
| 2394 | 138 | 192 | 234 | 325 | 383 | 97.1 | 2 | 0.725 |
| 2395 | 113 | 199 | 275 | 372 | 428 | 97.2 | 29 | 0.742 |
| 2396 | 160 | 194 | 264 | 369 | 417 | 97.8 | 24 | 0.738 |
| 2397 | 142 | 169 | 214 | 302 | 381 | 97.4 | 3 | 0.718 |
| 2398 | 124 | 214 | 270 | 361 | 432 | 97.4 | 29 | 0.745 |
| Average.. | 135 | 193 | 251 | 346 | 408 | 97.4 | 17 | 0.734 |

TORONTO, ONT.

| | | | | | | | | |
|-----------|-----|-----|-----|-----|-----|------|----|-------|
| 2363 | 115 | 210 | 268 | 356 | 426 | 97.2 | 35 | 0.743 |
| 2364 | 115 | 176 | 217 | 307 | 372 | 96.8 | 3 | 0.721 |
| 2365 | 122 | 181 | 221 | 315 | 386 | 97.1 | 6 | 0.723 |
| 2366 | 95 | 174 | 230 | 356 | 428 | 97.0 | 3 | 0.723 |
| 2367 | 122 | 216 | 268 | 358 | 428 | 97.1 | 26 | 0.744 |
| 2368 | 106 | 181 | 259 | 388 | 451 | 97.0 | 5 | 0.731 |
| Average.. | 112 | 190 | 244 | 347 | 415 | 97.0 | 13 | 0.731 |

LONDON, ONT.

| | | | | | | | | |
|-----------|-----|-----|-----|-----|-----|------|----|-------|
| 2369 | 93 | 147 | 225 | 388 | 435 | 96.5 | 3 | 0.721 |
| 2370 | 118 | 194 | 262 | 363 | 433 | 97.0 | 34 | 0.737 |
| 2371 | 129 | 183 | 230 | 329 | 384 | 95.9 | 2 | 0.727 |
| 2372 | 122 | 205 | 279 | 372 | 424 | 97.1 | 4 | 0.747 |
| 2373 | 120 | 201 | 262 | 363 | 432 | 97.0 | 35 | 0.738 |
| Average.. | 116 | 186 | 252 | 363 | 422 | 96.5 | 16 | 0.734 |

WINNIPEG, MAN.

| | | | | | | | | |
|-----------|-----|-----|-----|-----|-----|------|----|-------|
| 2562 | 122 | 208 | 268 | 369 | 435 | 97.1 | 35 | 0.742 |
| 2563 | 133 | 223 | 272 | 361 | 424 | 98.0 | 34 | 0.747 |
| 2564 | 120 | 191 | 261 | 379 | 449 | 97.1 | 13 | 0.735 |
| 2565 | 95 | 149 | 199 | 354 | 423 | 96.0 | 3 | 0.711 |
| 2566 | 115 | 192 | 252 | 360 | 424 | 97.0 | 30 | 0.736 |
| Average.. | 117 | 193 | 250 | 365 | 431 | 97.0 | 23 | 0.734 |

TABLE I—Continued
Results of Analyses—Concluded

REGINA, SASK.

| Lab. No. | Distillation Range | | | | | Recovered % | Iodine value | Spec. gravity |
|-----------|--------------------|----------|----------|----------|----------------|-------------|--------------|---------------|
| | 1st drop ° F. | 20% ° F. | 50% ° F. | 90% ° F. | End point ° F. | | | |
| 2379 | 122 | 205 | 264 | 363 | 432 | 97.2 | 32 | 0.744 |
| 2380 | 120 | 212 | 270 | 372 | 435 | 97.1 | 35 | 0.746 |
| 2381 | 111 | 181 | 239 | 369 | 441 | 97.0 | 7 | 0.728 |
| 2382 | 108 | 201 | 266 | 369 | 432 | 97.0 | 37 | 0.741 |
| Average.. | 115 | 200 | 260 | 369 | 435 | 97.1 | 28 | 0.740 |

CALGARY, ALTA.

| | | | | | | | | |
|-----------|-----|-----|-----|-----|-----|------|----|-------|
| 2548 | 122 | 212 | 266 | 365 | 435 | 97.3 | 27 | 0.750 |
| 2549 | 120 | 203 | 259 | 360 | 430 | 97.1 | 34 | 0.740 |
| 2550 | 115 | 219 | 268 | 363 | 424 | 97.2 | 23 | 0.753 |
| 2551 | 97 | 163 | 243 | 381 | 424 | 96.1 | 3 | 0.723 |
| 2552 | 127 | 217 | 268 | 360 | 426 | 98.1 | 24 | 0.755 |
| Average.. | 116 | 203 | 261 | 366 | 428 | 97.2 | 22 | 0.744 |

EDMONTON, ALTA.

| | | | | | | | | |
|-----------|-----|-----|-----|-----|-----|------|----|-------|
| 2544 | 127 | 191 | 262 | 361 | 426 | 97.5 | 27 | 0.750 |
| 2545 | 113 | 189 | 237 | 345 | 428 | 97.0 | 4 | 0.724 |
| 2546 | 126 | 212 | 268 | 367 | 437 | 97.3 | 33 | 0.740 |
| 2547 | 115 | 203 | 257 | 360 | 432 | 97.0 | 34 | 0.740 |
| Average.. | 120 | 199 | 256 | 358 | 431 | 97.2 | 25 | 0.739 |

VANCOUVER, B.C.

| | | | | | | | | |
|-------------------------|-----|-----|-----|-----|-----|------|----|-------|
| 2553 | 120 | 205 | 248 | 327 | 388 | 97.1 | 13 | 0.744 |
| 2554 | 113 | 205 | 259 | 367 | 437 | 97.2 | 3 | 0.753 |
| 2555 | 115 | 201 | 266 | 386 | 451 | 97.0 | 4 | 0.752 |
| Average.. | 116 | 204 | 258 | 360 | 425 | 97.1 | 7 | 0.750 |
| Average of all samples. | 120 | 193 | 255 | 358 | 423 | 97.1 | 19 | 0.737 |

TABLE II
Average Results of Analyses by Cities

| District | Distillation Range | | | | | | Iodine value | Spec. gravity |
|----------------------------|--------------------|---------|---------|---------|---------------|-------------|--------------|---------------|
| | 1st drop °F. | 20% °F. | 50% °F. | 90% °F. | End point °F. | Recovered % | | |
| Quebec, Que..... | 120 | 193 | 250 | 355 | 419 | 97.2 | 21 | 0.733 |
| Montreal, Que..... | 127 | 198 | 267 | 370 | 424 | 97.3 | 16 | 0.741 |
| Ottawa, Ont..... | 135 | 193 | 251 | 346 | 408 | 97.4 | 17 | 0.734 |
| Toronto, Ont..... | 112 | 190 | 244 | 347 | 415 | 97.0 | 13 | 0.731 |
| London, Ont..... | 116 | 186 | 252 | 363 | 422 | 96.5 | 16 | 0.734 |
| Winnipeg, Man..... | 117 | 193 | 250 | 365 | 431 | 97.0 | 23 | 0.734 |
| Regina, Sask..... | 115 | 200 | 260 | 369 | 435 | 97.1 | 28 | 0.740 |
| Calgary, Alta..... | 116 | 203 | 261 | 366 | 428 | 97.2 | 22 | 0.744 |
| Edmonton, Alta..... | 120 | 199 | 256 | 358 | 431 | 97.2 | 25 | 0.739 |
| Vancouver, B.C..... | 116 | 204 | 258 | 360 | 425 | 97.1 | 7 | 0.750 |
| Average ¹ | 120 | 193 | 255 | 358 | 423 | 97.1 | 19 | 0.737 |

COMPARISON OF RESULTS

It is interesting to compare the above figures with others obtained in somewhat the same way. Table III gives the average results of twenty samples collected in the city of Ottawa in December, 1922², the average results of the five samples collected in Ottawa in August, 1923, the average of the forty-eight samples collected throughout Canada in August, 1923, the average of 158 samples collected in the United States during July, 1923, as reported by the United States Bureau of Mines,³ and the essential features of the specifications for motor gasoline, adopted by the Specifications Board of the United States³ for the use of the various departments and independent establishments of the United States government. The average of 88 samples collected in Canada, presumably during 1916, and reported by the laboratory of the Department of Inland Revenue,⁴ was calculated by the writer and is also given. In this last case, neither the method of determining the distillation range nor the apparatus used was stated, so that the calculated result can be considered as only approximately correct. Moreover, the results of the distillations are expressed as certain percentages of distillate obtained below several arbitrarily chosen temperatures, as shown in Table VI, whereas the method of showing results which is most often adopted at the present time gives the initial and the final temperatures at which the sample boils, together with the temperature noted when each successive 10 per cent of distillate has been recovered. In order to reduce the results shown in Bulletin 362 to a form in which they can be compared with the figures obtained in the present survey, the average amount of distillate obtained at the different temperatures was calculated and these averages were plotted on squared paper with other graphs, as in Diagram I. A curve was then drawn through the plotted points which was similar in general shape to distillation curves drawn for the other gasolines. From the graph so obtained, the temperature for the initial and end points and for other points was read off as required.

¹ This is the average value for all the samples tested.

² Summary Report of Investigations, Mines Branch, 1922.

³ U.S. Bureau of Mines, Report of Investigations, Serial No. 2525.—"Eighth Semi-annual Motor Gasoline Survey."

⁴ Department of Inland Revenue, Canada, Bulletin 362.—"Gasoline."

DIAGRAM 1.

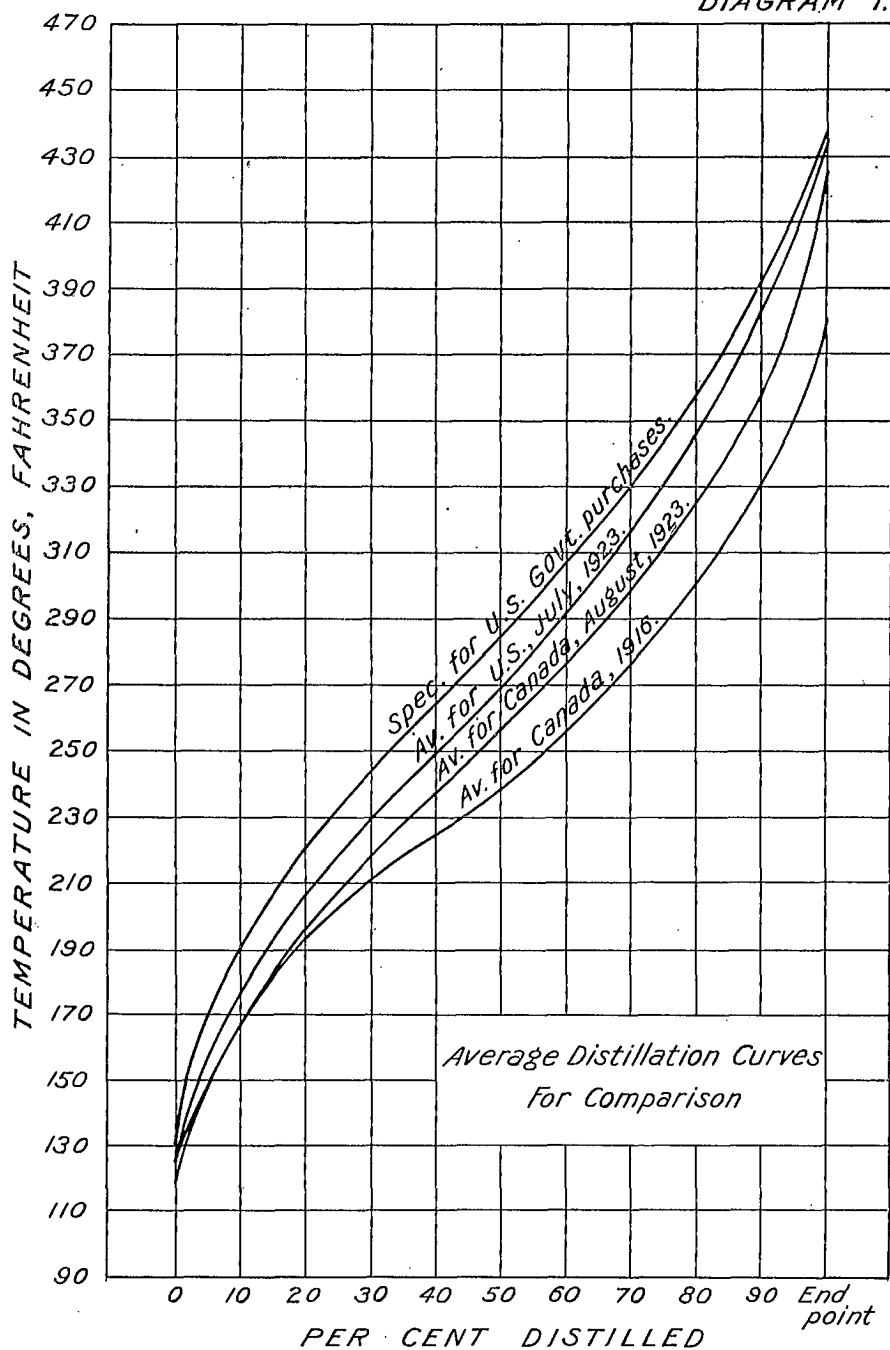


TABLE III
Average Results for Comparison

| | Distillation Range | | | | | Iodine value | Sp. gr. at 60°F. |
|---------------------------------|--------------------|---------|---------|---------|---------------|--------------|------------------|
| | 1st drop °F. | 20% °F. | 50% °F. | 90% °F. | End point °F. | | |
| Ottawa, Dec. 1922..... | 123 | 187 | 246 | 339 | 399 | 16 | 0.727 |
| Ottawa, Aug. 1923..... | 135 | 194 | 251 | 346 | 408 | 17 | 0.734 |
| Canada, Aug. 1923..... | 120 | 193 | 255 | 358 | 423 | 19 | 0.737 |
| Canada, 1916 ¹ | 125 | 192 | 237 | 330 | 380 | 17 | 0.732 |
| United States, July 1923..... | 125 | 206 | 269 | 382 | 436 | | 0.751 |
| U.S. Federal specification..... | 131 | 221 | 284 | 392 | 437 | | |

¹ Department of Inland Revenue, Canada, Bulletin 302—"Gasoline."

It will be observed that the gasoline sold in Canada shows an average of distinctly good quality, when judged by the distillation range, which is the ordinarily accepted standard. This may be due to different causes, among which are the following:—

(a) A general tendency on the part of the user, lacking definite information on the matter, is to demand the best grades.

(b) The prevailing low temperature occurring in the winter months requires a comparatively volatile gasoline in order to operate satisfactorily.

(c) Large stocks of gasoline and crude petroleum have been accumulated and stored above ground during the past year.

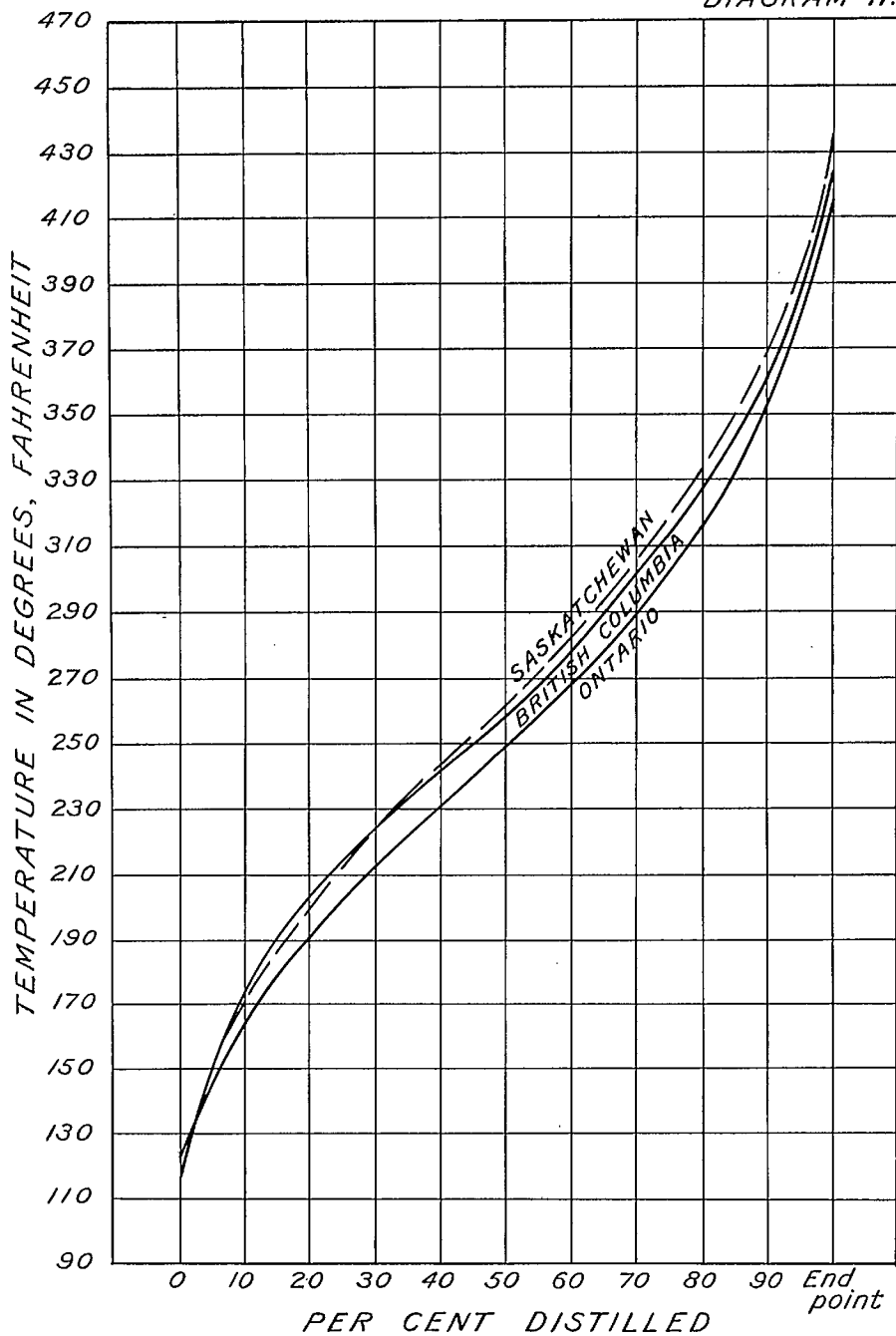
Another point worthy of observation is that the characteristics vary according to the geographical or commercial districts in which the samples were collected. Table IV shows the average distillation ranges and specific

TABLE IV
Average Results by Provinces

| Province | No. of samples | 1st drop °F. | 20% °F. | 50% °F. | 90% °F. | End point | Spec. gravity |
|-----------------------|----------------|--------------|---------|---------|---------|-----------|---------------|
| Quebec..... | 11 | 124 | 196 | 260 | 364 | 422 | 0.737 |
| Ontario..... | 16 | 121 | 190 | 249 | 352 | 415 | 0.733 |
| Manitoba..... | 5 | 117 | 193 | 250 | 365 | 431 | 0.734 |
| Saskatchewan..... | 4 | 115 | 200 | 260 | 369 | 435 | 0.740 |
| Alberta..... | 9 | 118 | 201 | 259 | 362 | 429 | 0.742 |
| British Columbia..... | 3 | 116 | 204 | 258 | 360 | 425 | 0.750 |

gravities of samples from the different provinces. It may be pointed out that samples from Ontario and Manitoba have the lowest specific gravity, and samples from Alberta and British Columbia the highest, samples from Saskatchewan and Quebec having an intermediate value. From that fact it might be assumed that the average distillation range of samples from British Columbia would be much higher than that of samples from Ontario. However, the distillation curves for the two provinces, as shown in Diagram II, show that while the distillation curve for British Columbia is higher than that of Ontario, it is not sufficiently high to explain the difference in gravity. Moreover, the average distillation curve for

DIAGRAM II.



Saskatchewan, also shown in Diagram II, lies above the curve for British Columbia for the greater part of its length, although its specific gravity is considerably lower. It is, therefore, obvious that the specific gravity does not bear a simple and constant relation to the distillation range. It is generally understood¹ that commercial gasoline cannot be compared satisfactorily by reference to specific gravity alone, since different refined products may be blended together, such as a mixture of casing-head gasoline and kerosene, in such proportions as to give almost any specific gravity required. The effect referred to above differs from the effect produced by blending because in the latter case the distillation range is higher when the specific gravity is higher, whereas in the case under discussion, the distillation varies comparatively little while the specific gravity varies a great deal. It is well known that the characteristics and constituents of crude petroleum from different fields vary. It would appear then that the characteristics of various gasolines depend upon the constituents originally present in the crude petroleum, or which may have been formed from the crude during its treatment for the production of gasoline.

The crude petroleum obtained from California, for instance, is of higher specific gravity, as a rule, than that from Oklahoma or from Pennsylvania fields of similar viscosity. Although it is not the purpose here to discuss the distribution of crude petroleum, it seems reasonable to expect to find large quantities of gasoline made in British Columbia from California crudes, on account of the proximity of the two districts and the well-developed coastal trade, which gasoline would therefore show a higher specific gravity for a similar distillation range than gasoline made in Ontario from Oklahoma or Pennsylvania crude oils. The same effect may be observed in the provinces of Quebec and Alberta, where doubtless a certain percentage of California crude oil, or its products, finds a market; in one case because of the low rates for ocean transportation in comparison with either pipe line or rail haul; and in the other case because part of the necessary supplies are obtained from British Columbia.

VARIATION IN QUALITY

In order to estimate the variation in quality of the gasoline being sold, the average of the 10 per cent (five samples) having the highest end point and the average of the 10 per cent having the lowest end point was obtained as shown in Table V. The magnitude of the difference between these two averages, shown in Table VIII, may be accepted for purposes of comparison as a measure of variation in quality. Since no indication of the final boiling temperature of the samples examined is given in Bulletin 362 of the Department of Inland Revenue it is somewhat difficult to compare the results obtained then with those obtained in this survey. However, if the nine samples showing the highest percentage of distillate above 150°C. are accepted as the 10 per cent having the highest end point, and likewise the nine samples showing the lowest percentage of distillate above 150°C. as the 10 per cent having the lowest end point, then the average percentage of each one of the groups as shown in Table VI should show approximately the distillation curve of the maximum and minimum 10 per cent of the samples, respectively. Also the difference between these two values, namely 22 per cent, shows approximately the variation in quality of gasoline sold at that time.

¹ Summary Report of Investigations, Mines Branch, 1922, page 224.

By plotting from Table V the distillation curve as shown in Diagram III of the average maximum and minimum 10 per cent of the samples of the present survey, the difference in volatility at 150°C. (302° F.) can be obtained. It is to be observed that whereas the difference in 1916 was 22 per cent, the difference in 1923 was 17.5 per cent, showing that the general tendency during the intervening years has been towards a more uniform product. Since that is so, it seems logical to expect that the end point of a gasoline obtained in 1916 would be higher than the end point of a gasoline obtained in 1923, if the two gasolines gave an equal percentage of distillate above 150°C. (302°F.). Moreover, if those samples in the present survey be chosen, which give about the same percentage of distillate above 150°C. (302°F.) as the maximum 10 per cent shown in Table VI, and the average end point of these samples be obtained, it follows that the end point so obtained should be not higher than the end point of the maximum 10 per cent in Table VI. Table VII gives the average of samples which have a distillation temperature of 145° C. to 149° C. when 70 per cent of distillate has been recovered. Diagram IV shows the resulting distillation curve and also the curve A, which represents the average maximum 10 per cent of samples examined in 1916.

TABLE V
10 per cent of Samples having Maximum End Point

| Lab. No. | Distillation Range | | | | | | Spec. gravity at 60° F. |
|--------------|--------------------|---------|---------|---------|---------|---------------|-------------------------|
| | 1st drop °F. | 20% °F. | 50% °F. | 70% °F. | 90% °F. | End point °F. | |
| 2368..... | 106 | 181 | 259 | 310 | 388 | 451 | 0.731 |
| 2378..... | 163 | 219 | 255 | 295 | 354 | 439 | 0.746 |
| 2381..... | 111 | 181 | 239 | 295 | 369 | 441 | 0.728 |
| 2555..... | 115 | 201 | 266 | 318 | 386 | 451 | 0.752 |
| 2564..... | 120 | 191 | 261 | 306 | 379 | 449 | 0.735 |
| Average..... | 123 | 195 | 256 | 305 | 375 | 446 | 0.738 |

10 per cent of Samples having Minimum End Point

| | | | | | | | |
|--------------|-----|-----|-----|-----|-----|-----|-------|
| 2364..... | 115 | 176 | 217 | 250 | 307 | 372 | 0.721 |
| 2365..... | 122 | 181 | 221 | 254 | 315 | 386 | 0.723 |
| 2371..... | 129 | 183 | 230 | 246 | 329 | 384 | 0.727 |
| 2394..... | 138 | 192 | 234 | 272 | 325 | 383 | 0.725 |
| 2397..... | 142 | 169 | 214 | 242 | 302 | 381 | 0.718 |
| Average..... | 129 | 180 | 223 | 253 | 316 | 381 | 0.723 |

DIAGRAM III.

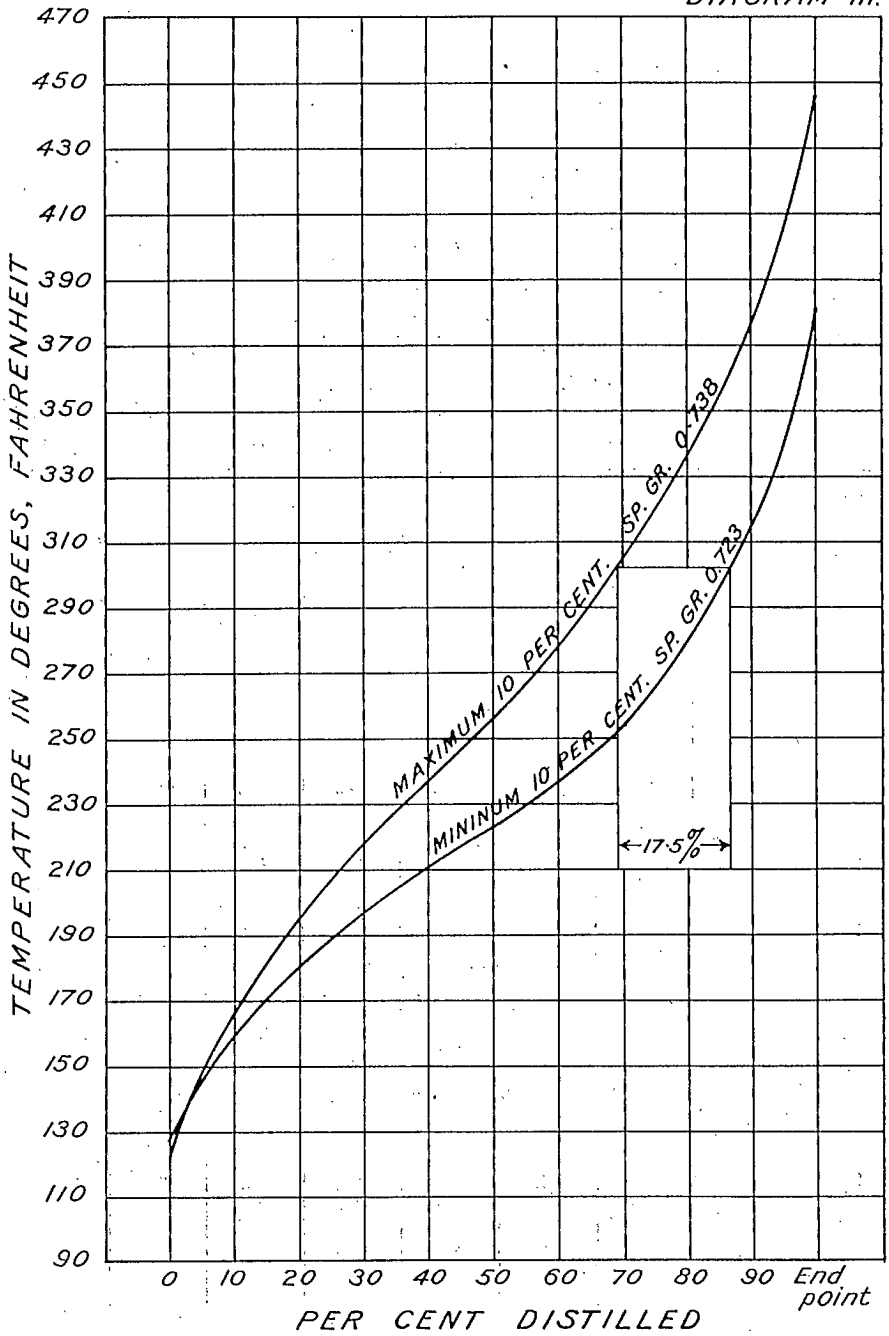


TABLE VI

10 per cent of Samples having highest percentage of distillate above 150° C. taken from Bulletin 362, Dept. of Inland Revenue

| Dept. Inland Revenue No. | % below 70°C. | % 70°C. to 120°C. | % 120°C. to 140°C. | % 140°C. to 150°C. | % above 150°C. | Spec. gravity |
|--------------------------|---------------|-------------------|--------------------|--------------------|----------------|---------------|
| 75462..... | 12.5 | 18.1 | 13.2 | 7.7 | 48.0 | 0.738 |
| 0..... | 12.8 | 18.2 | 16.5 | 12.0 | 36.8 | 0.732 |
| 75463..... | 14.5 | 38.5 | 15.0 | 6.2 | 24.5 | 0.730 |
| 75432..... | 17.5 | 40.0 | 13.0 | 6.1 | 22.5 | 0.720 |
| 75445..... | 5.0 | 46.0 | 17.8 | 7.7 | 23.0 | 0.746 |
| 69507..... | 3.5 | 49.5 | 18.0 | 7.5 | 21.2 | 0.743 |
| 69533..... | 2.0 | 45.5 | 22.0 | 9.5 | 20.0 | 0.743 |
| F..... | 12.7 | 44.9 | 15.2 | 6.2 | 20.0 | 0.730 |
| 75466..... | 5.5 | 45.5 | 20.5 | 8.0 | 20.0 | 0.740 |
| Average..... | 9.6 | 38.5 | 16.8 | 7.9 | 26.2 | 0.736 |

10 per cent of Samples having lowest percentage of distillate above 150°C. taken from Bulletin 362

| | | | | | | |
|--------------|------|------|------|-----|-----|-------|
| 69534..... | 11.5 | 72.0 | 10.0 | 2.0 | 3.4 | 0.719 |
| 69539..... | 11.0 | 71.7 | 10.8 | 2.0 | 4.0 | 0.720 |
| 69531..... | 12.5 | 70.2 | 10.5 | 1.8 | 4.0 | 0.720 |
| 69532..... | 13.5 | 69.2 | 10.8 | 2.0 | 4.0 | 0.720 |
| 69539..... | 9.5 | 71.0 | 11.0 | 4.3 | 4.0 | 0.724 |
| 69538..... | 11.0 | 70.0 | 11.0 | 2.5 | 4.5 | 0.721 |
| 69536..... | 11.5 | 71.2 | 10.0 | 2.8 | 4.5 | 0.720 |
| 69537..... | 12.0 | 67.5 | 12.0 | 3.5 | 4.5 | 0.721 |
| 5433..... | 8.5 | 69.5 | 10.8 | 5.0 | 5.0 | 0.726 |
| Average..... | 11.2 | 70.3 | 10.8 | 2.9 | 4.2 | 0.721 |

Difference between maximum and minimum above 150°C. = 22.0%.

TABLE VII

Samples which yield 70 per cent distillate between 145° C. and 149° C. inclusive

| Lab. No. | 1st drop °C. | 10% °C. | 20% °C. | 50% °C. | 70% °C. | 90% °C. | End point |
|---------------|--------------|---------|---------|---------|---------|---------|-----------|
| 2378..... | 73 | 97 | 104 | 124 | 146 | 179 | 226 |
| 2381..... | 43 | 71 | 84 | 117 | 146 | 187 | 227 |
| 2544..... | 53 | 88 | 99 | 128 | 149 | 183 | 219 |
| 2547..... | 46 | 82 | 95 | 125 | 147 | 182 | 222 |
| 2549..... | 49 | 81 | 95 | 126 | 148 | 182 | 221 |
| 2566..... | 46 | 79 | 89 | 122 | 146 | 182 | 218 |
| Aver. °C..... | 52 | 83 | 94 | 124 | 147 | 182 | 222 |
| Aver. °F..... | 126 | 181 | 201 | 255 | 297 | 360 | 432 |

It was found that the minimum 10 per cent of 1916 was so low that none of the 1923 samples could approximate it. Therefore the distillation range of a special gasoline obtained in 1922 was used to approximate the minimum 10 per cent end point for 1916. This distillation range is shown on Diagram IV, and the curve B which bears the same relation to the minimum curve as the curve A does to the maximum 10 per cent curve.

The results obtained as above are summarized in Table VIII, which shows the difference in degrees Fahrenheit between the maximum 10 per cent and minimum 10 per cent of samples collected in Canada in 1916 and in 1923, together with similar figures obtained by the Bureau of Mines¹ in the United States for July 1923.

TABLE VIII
Difference between Maximum and Minimum End Points

| | Canada 1916 | Canada August 1923 | Ontario and Quebec 1923 | Manitoba, Saskatch- ewan and Alberta, 1923 | British Columbia 1923 | United States July 1923 |
|------------------|----------------|--------------------------|----------------------------------|--|-----------------------------|----------------------------------|
| | °F. | °F. | °F. | °F. | °F. | °F. |
| Maximum 10%..... | 432 | 446 | 442 | 445 | 451 | 471 |
| Minimum 10%..... | 322 | 381 | 379 | 424 | 388 | 400 |
| Difference..... | 110 | 65 | 63 | 21 | 63 | 71 |

DEW POINT AND WEIGHTED INDEX NUMBERS

The dew point of a gasoline is that temperature at which the gasoline vapours begin to condense from a mixture of the vapour with air. It is obvious that the temperature will depend on the proportion of gasoline vapour to air. Since the proportion of 1 to 15 parts respectively gives a satisfactory burning mixture, this proportion has been accepted as the most desirable. Gruse² has developed a very ingenious apparatus for determining the dew point directly. Wilson and Barnard³ have also done some work along the same line. Gruse⁴ has demonstrated that the dew point of a gasoline may be estimated more or less accurately from the results of an ordinary distillation.

The temperature readings in degrees Centigrade at which 10, 20, 50, 70, and 90 per cent of distillate has been recovered are added to the temperature reading at the end point of the distillation and the resultant sum is called the weighted index number. The index numbers for several average distillation ranges are shown on Table IX. It is further shown that the index numbers so obtained are simply related to the dew point, in degrees Centigrade for a wide range of commercial gasolines which have been examined in the Gruse apparatus, and that the relationship can be graphically shown by a straight line. The position and slope of the line can be easily established by the index numbers of two or more gasolines of which the dew point is known.

¹ U. S. Bureau of Mines, Report of Investigation 2525.

² Industrial and Engineering Chemistry, Vol. 15, No. 8, page 796 (1923).

³ Industrial and Engineering Chemistry, Vol. 13, No. 10, page 906 (1921).

⁴ Chemical and Metallurgical Engineering, Vol. 23, No. 22, page 970 (1923).

DIAGRAM IV.

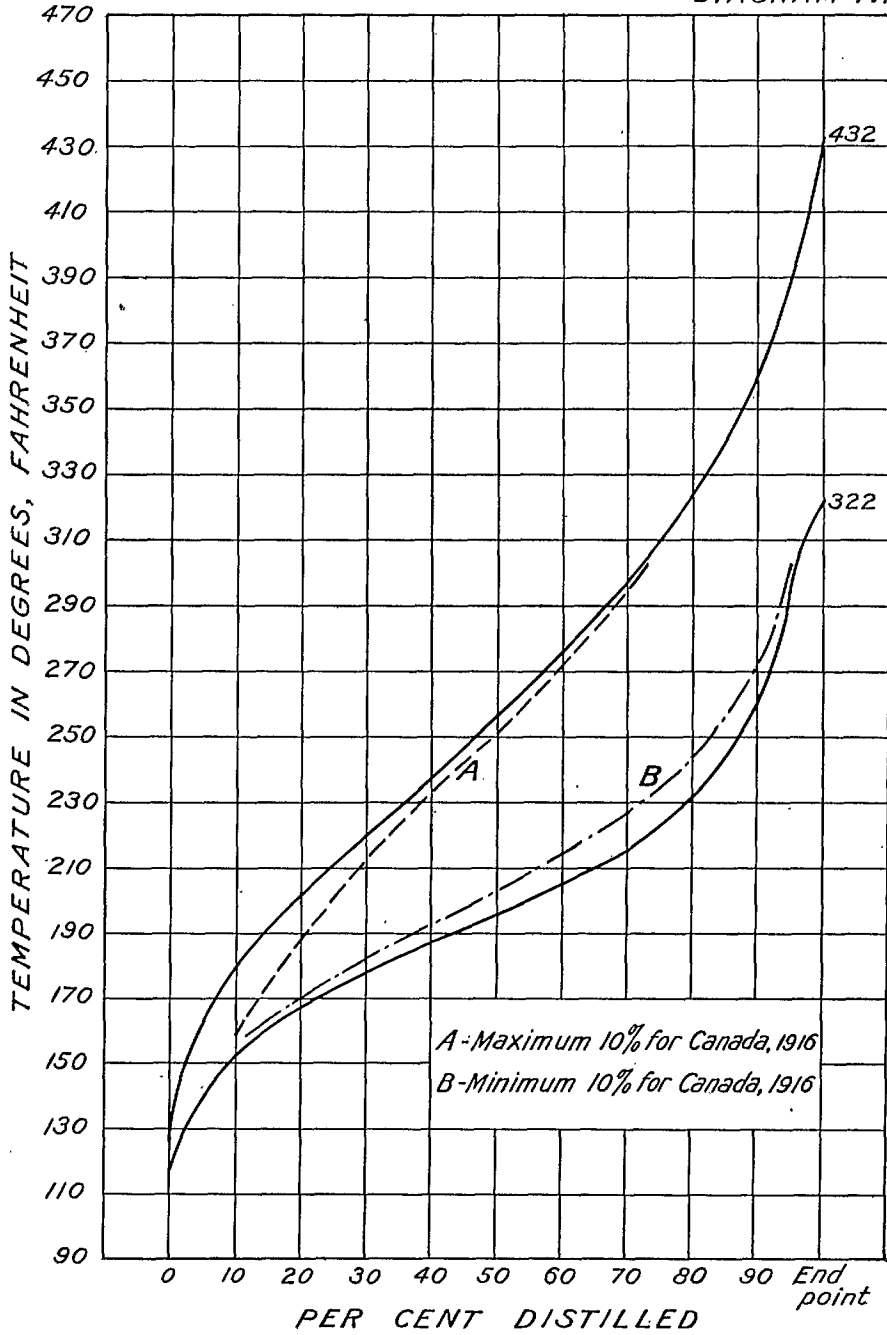


TABLE IX
Calculation of Index Numbers

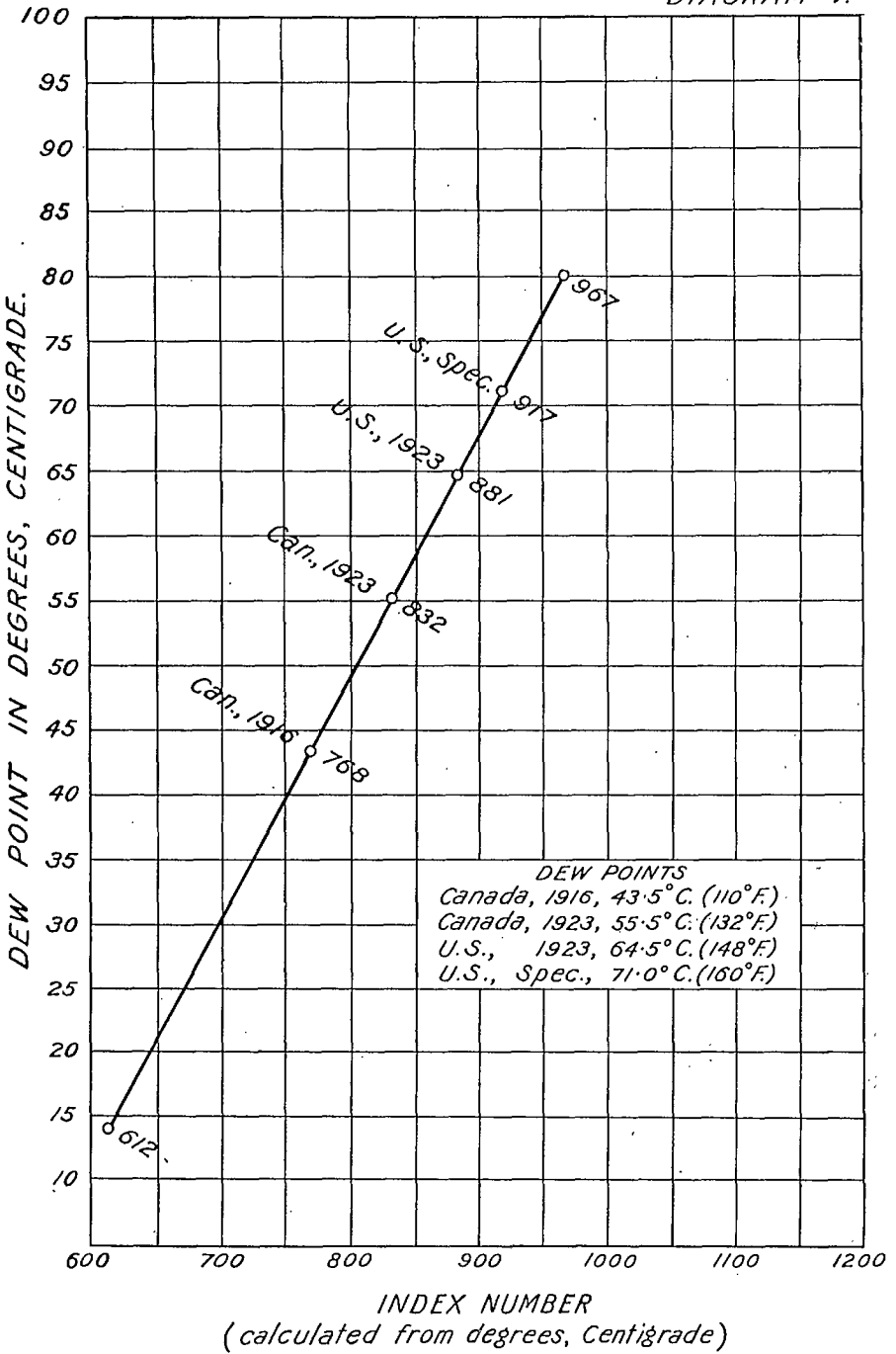
| Distillate | Average for Canada 1916 | Average for Canada 1923 | Average for United States 1923 | United States Federal spec. |
|---------------|-------------------------------|-------------------------------|--------------------------------------|-----------------------------------|
| | °C. | °C. | °C. | °C. |
| 10%..... | 76 | 76 | 80 | 84 |
| 20%..... | 89 | 89 | 96 | 105 |
| 50%..... | 113 | 123 | 131 | 140 |
| 70%..... | 132 | 146 | 156 | 163 |
| 90%..... | 165 | 181 | 194 | 200 |
| End..... | 193 | 217 | 224 | 225 |
| Index No..... | 768 | 832 | 881 | 917 |

Convenient points for establishing the graph are:—Domestic aviation, with index number of 612 and dew point of 14°C. together with co-operative gasoline D, which has an index number of 967 and a dew point of 80°C. The above points are calculated in degrees Centigrade and for a fuel mixture of 15 parts of air to 1 part of fuel.

Diagram V shows the dew point of the average gasoline sold in Canada in the years 1916 and 1923. It is interesting to note that the dew point has increased appreciably during the past seven years.

SUMMARY AND CONCLUSIONS

1. The gasoline sold in Canada during 1923 shows an average of good quality when judged by the distillation range.
2. There are indications which suggest that the constituents present in gasoline from different parts of the country vary.
3. Gasoline sold in Canada during 1923 is more uniform in quality than that sold in Canada during 1916, or that sold in the United States during 1923.
4. The Gruse index number for gasoline was lower for gasoline sold in Canada during 1916 than for that sold during 1923.
5. The average dew point of gasolines sold in Canada has increased from 110°F. in 1916 to 132°F. in 1923, calculated for a gasoline-air mixture of 1 to 15.



THE HARTMAN OIL SHALE RETORT

A. A. Swinnerton

In connexion with the investigational work on Canadian oil shales outlined in previous Mines Branch publications,¹ the Hartman retort for the distillation of oil shale was examined by the writer, and this report has to do with trial runs made in the demonstration apparatus at St. John, N.B., using New Brunswick oil shale from the Albert Mines area.

Canadian Patent No. 223085 granted to Emanuel W. Hartman, August 29, 1922, is entitled, "Shale Handling Apparatus." In the words used in the patent application—"This invention relates to an improvement in Shale Handling Apparatus and more particularly to an apparatus having a retort in which the shale is distilled, and condensing means in which the products of the distillation are treated to separate out the gasoline and heavy oils." No special mention is made as to the precise temperature ranges at which the retort is to be operated, nor is there any reference made to the control of the reactions taking place by the use of steam. The general process used follows regular destructive distillation practice for oil shales, and in this connexion the products obtained may be compared with those obtained by ordinary laboratory distillation methods.

The second to sixth paragraphs of page 2 of the patent application are as follows:—

"One object of this invention is to provide in such apparatus a retort having a cylindrical chamber provided with a series of shelves or decks over which the shale is moved by means of a series of rotating conveyors and in which retort the individual shelves are heated directly so that the temperature under which the shale on each shelf is treated can be controlled.

"Another object of this invention is to provide in the retort of this apparatus a rotatable shaft which is normally supported on bearings beneath its foot but which may be raised under the action of the shale during the operation of the apparatus without disturbing its driving connection.

"A further object of this invention is to provide in the retort a corrugated base plate so that any distortion of the plate due to the extreme heat to which it is subjected during the distillation operation will be taken up by such corrugations without interfering in any way with the operation of the vertical driving shaft.

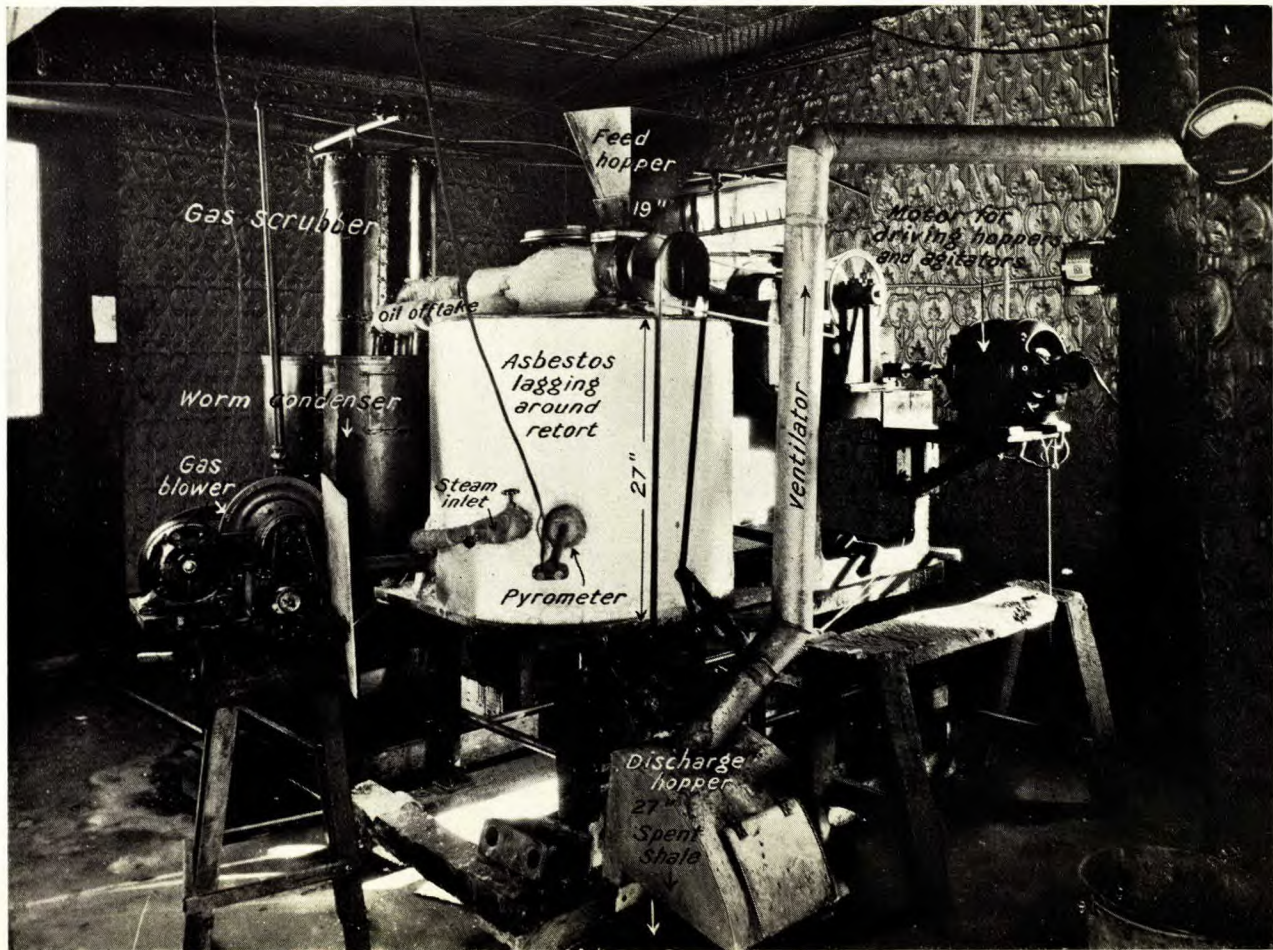
"A still further object of this invention consists in providing in each of the shelves of the retort a plurality of heating elements preferably electrically controlled.

"Another object of this invention consists in the provision of a condensing apparatus the various parts of which are made up of pipes of varying diameters."

DESCRIPTION OF RETORTING APPARATUS

As installed at St. John the Hartman retorting apparatus occupied a space of approximately $6\frac{1}{2}' \times 7\frac{1}{2}' \times 5'$. It consisted of a vertical retort connected to a worm condenser and gas scrubber with the necessary accessories, such as electric motors, gas blower, pyrometer, oil containers, etc. Plate I is a photograph of the apparatus as installed in a garage; Figure 1 is a diagrammatic sketch of the apparatus train; and Figures 2 and 3 are detailed sketches of the retort as given in the patent specification.

¹ Mines Branch Summary of Investigations 1021, page 230, and 1922, page 210.



To face Page 54.

Hartman retorting apparatus, St. John, N.B.

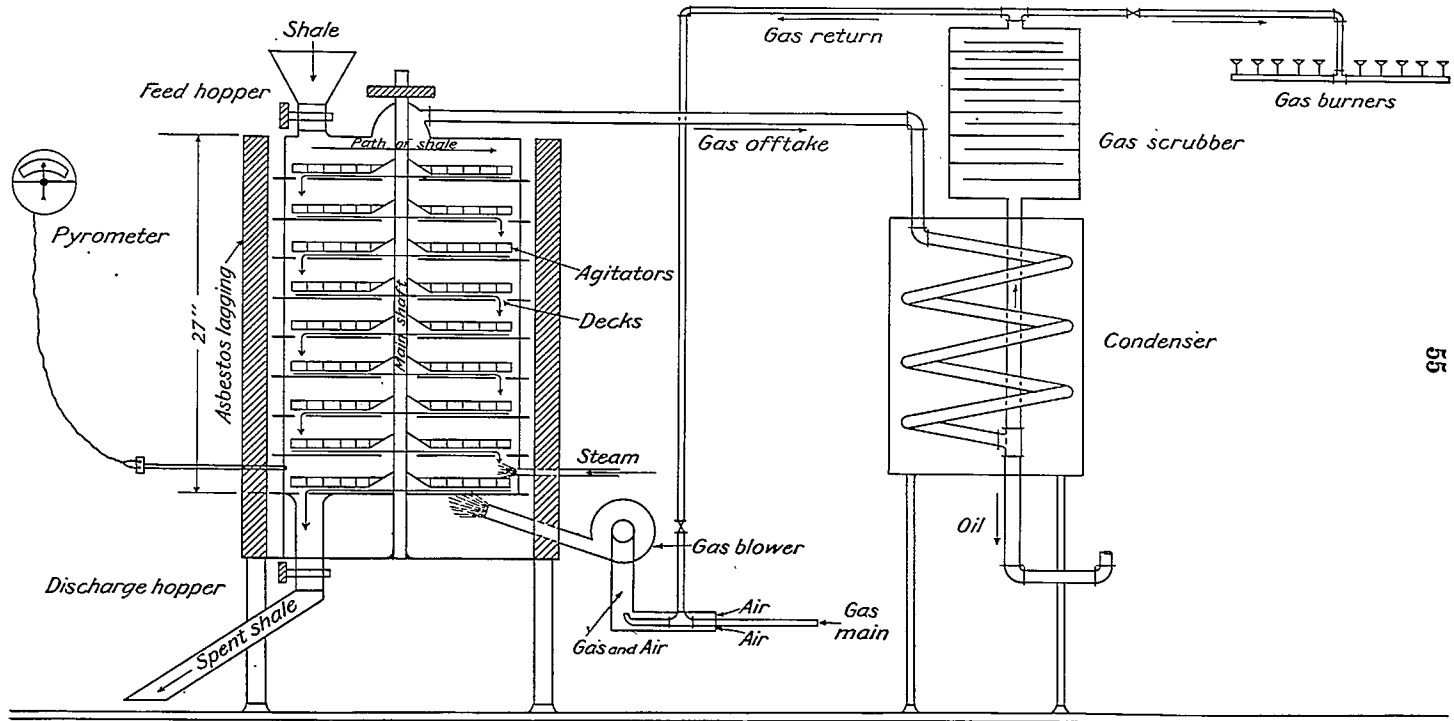


FIGURE 1. Hartman experimental retort, St. John, N.B.

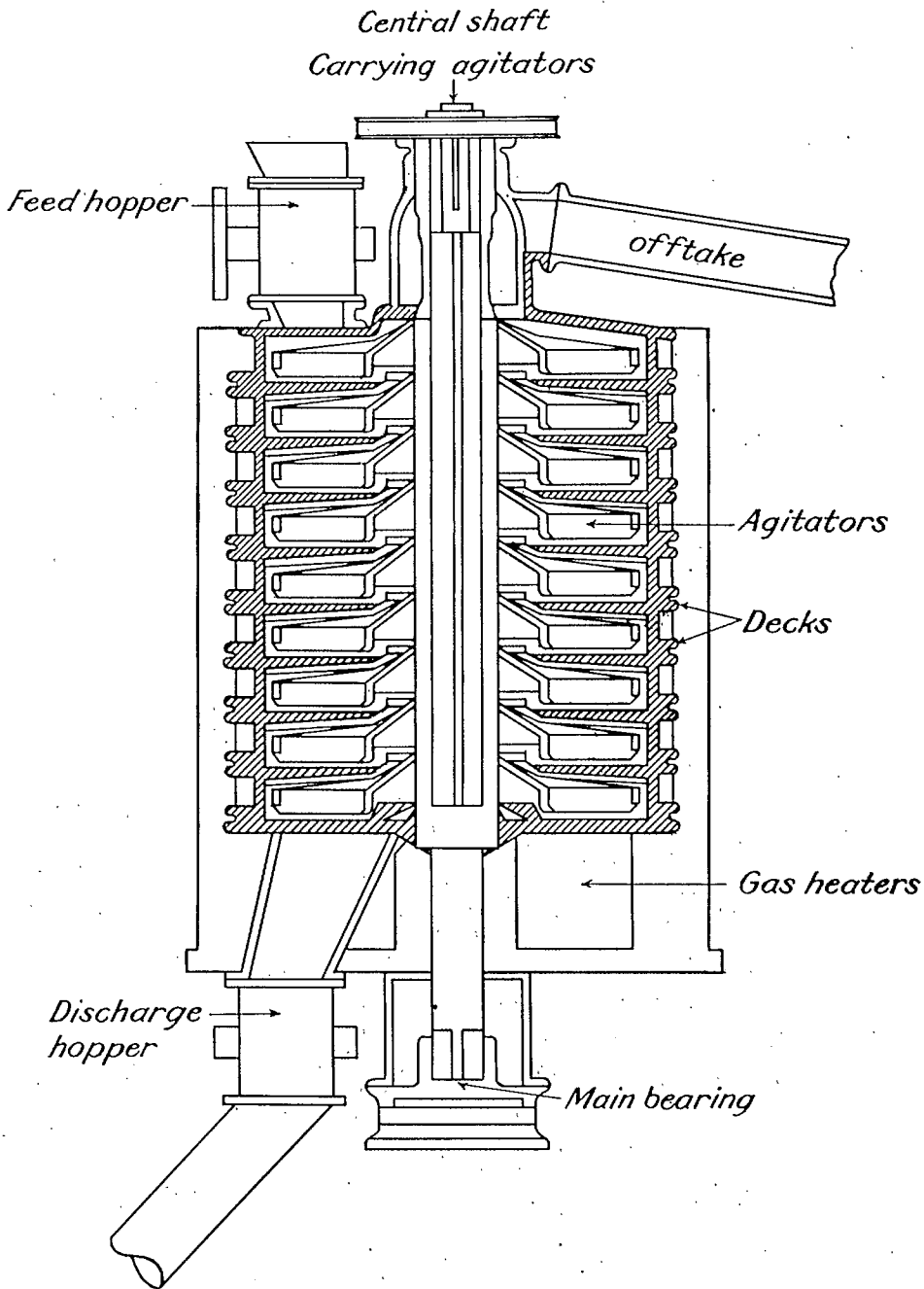


FIGURE 2. Sketch of details of Hartman retort

The retort consisted of a cylindrical chamber provided with a series of ten shelves or decks over which the shale is made to travel by means of a corresponding series of rotating conveyers or agitators which are keyed on to a central revolving shaft as in a Herreshoff-McDougall ore roasting furnace. A special feature of the retort design is a base plate with concentric corrugations, which according to the claims in the patent application, will take up any changes in the dimensions of the plate when subjected to extreme heat. The upper shelves or decks, eight in number, are described as identical in construction and different from the base plate only in that they are plain and not corrugated. Each deck consists of a circular plate with a hole in the centre to permit the escape of the vapours formed and also to allow room for the central revolving shaft which carries the agitators. They are also provided with peripheral flanges on the lower side and these flanges are bolted together to form the outside wall of the

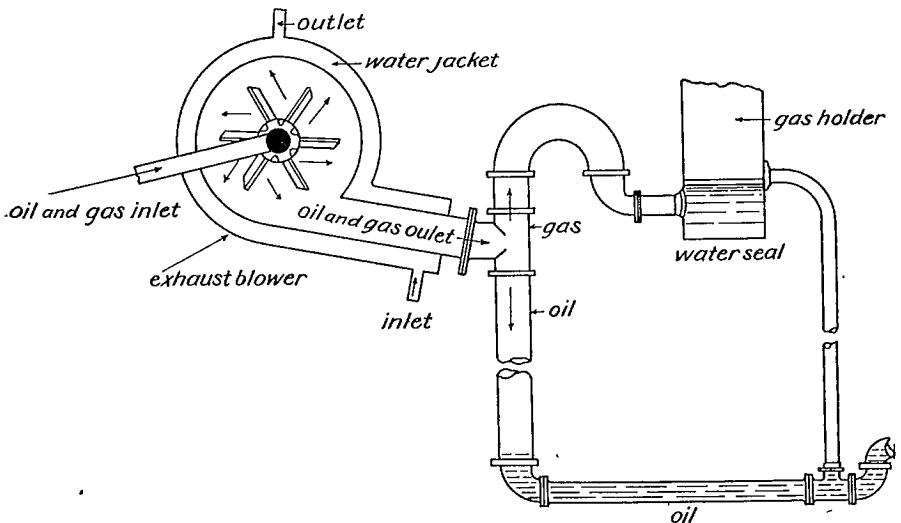


FIGURE 3. Exhaust blower and condenser for Hartman retort

retort. In each deck there is a hole or slot through which the shale may drop on to the next plate. These various openings are staggered so that the shale after dropping on to a plate will be carried round for one revolution before it drops on to the plate below. A hopper with a star wheel feed is attached to the top of the retort and a discharge hopper with a similar feed is attached immediately below the slot in the lowest or corrugated base plate.

The course of travel of crushed shale through the retort is as follows. The shale from the hopper is fed by the star wheel down on the top deck where by means of the first agitator plate it is carried around the complete circumference of the first stationary deck or plate before being dropped through a slot on to the second deck. Here it is again carried around in the same manner to the point where it is dropped through on the third deck and so on, down through the eight decks, and finally through the slot in the base plate into the discharge hopper and out as spent shale.

Mention is made in the patent application of special electric heating elements to be embedded in the stationary plates and the drawings show the whole retort enclosed in a brick, insulation wall. The retort as installed and operated at St. John was however different in that it was heated by a motor-driven blower using city gas, the flame being directed onto the base plate and the flue gases going up and around the retort between the retort walls and the surrounding asbestos insulation as shown in Plate I. As the hot flue gases passed up and around the outside of the retort they came in contact with the projections of the flanged decks which apparently acted as baffle plates. The retort was therefore heated externally and the heat necessary to carry on the distillation of the shale was obtained by radiation and conduction inward, along and through the metal plates.

In the apparatus as demonstrated at St. John no special vapour condensing system was used. The shale gas as it left the retort passed through a worm condenser at the bottom of which by means of a T the uncondensed gas was led off up into an expansion tank and thence to gas burners to be burned. The condensed oil was drawn off at intervals from a swing arm attached to the above-mentioned T as shown in Figure 1.

In the patent application and drawings special mention and claims re a condensing apparatus were made, the main features of which are, (a) a water seal through which the gas must pass before going to gas tank and (b) a special rotary water-cooled exhaust blower as shown in Figure 3. The claim is made that by the admission of air into the shale gas in the blower "the separation of the gasoline and heavy oils in the chamber is furthered by the admission of the air which acts to cool the distillate and also adds oxygen to the mixture thus increasing the amount of the liquid precipitated." The use of cold air admitted and thrown against the water-cooled walls of the rotary blower may be advantageous, but it is evidently the temperature—the cooler the better—that is important and not the use of air as such. It is inconceivable how oxygen (of the air) may be added to the mixture "thus increasing the amount of liquid" as the crude shale oils are unsaturated in nature and require a reducing rather than an oxidizing atmosphere. This special condensing equipment was however not demonstrated in the trial runs here reported and therefore will not be further discussed.

The retort may be operated either intermittently or continuously and the rate of distillation can be controlled both by the rate of feed of the shale and by the intensity of the heat applied. Intermittent runs would not be expected to run as smoothly as continuous runs due to expansion changes while starting and stopping, and runs of short duration would not be as satisfactory as long runs.

TRIAL RUNS IN HARTMAN RETORT

Two test runs only were made at St. John under the writer's supervision, the duration of these being about two hours each. As mentioned above, the retort was heated by city gas and the amount used was measured. By means of a pipe inserted through the side-wall flange just above the base plate steam could be used and was used during the first run. The maximum temperature to which the shale was heated was recorded by a pyrometer inserted in the base plate. In the first run Albert shale, crushed

to about pea size, was used with steam, and in the second run what is known as paper shale (from Albert Mines) was distilled without the use of steam. A summary of the two trial runs is as follows:—

TABLE I

| | Run No. 1 (with steam) | Run No. 2 (without steam) |
|---|---------------------------|------------------------------|
| Shale used— | Albert shale | Paper shale |
| Date of run..... | Feb. 2 | Feb. 3 |
| Retort charged..... | 2.45 p.m. | 2.10 p.m. |
| Retort empty..... | 4.55 p.m. | 4.30 p.m. |
| Amount of shale used..... | 100 lbs. | 100 lbs. |
| Temperature at bottom plate..... | 850-950° F. | 920-970° F. |
| City gas used (c.f. per hour)..... | 115 | 130 |
| Products obtained— | | |
| Shale gas (c.f. per hr. estimated)..... | 50-60 | 50-60 |
| Duration of gas at this rate..... | 1 hr. 30 mins. | 1 hr. 30 mins. |
| Weight of spent shale..... | 75 lbs. 15 oz. | 81 lbs. 5 oz. |
| Spent shale as % of shale charged..... | 75.9 | 81.3 |
| Amount of oil (Imp. qts.)..... | 9½ | 5 |
| Yield of crude wet oils (as measured) (Imp. gals. per 2,000 lbs.)..... | 46 | 25 |
| Dry oil yield (corrected)..... | 32 | 25 |

LABORATORY EXPERIMENTAL WORK

Samples of the crude oil obtained from the Hartman retort were brought back to Ottawa and examined in the Fuel Testing Laboratories. Samples of the oil shales used were also brought back, on which check runs were made in the standard-temperature control lead bath electric-furnace already described in a previous Mines Branch publication¹ and in an electrically heated retort specially fitted for distilling oil shale in the presence of steam.

A proximate analysis of the two oil shales used is as follows:—

TABLE II

| | Albert shale | Paper shale |
|---------------------------------------|--------------|-------------|
| Moisture.....% | 0.7 | 0.5 |
| Ash.....% | 77.4 | 78.7 |
| Volatile matter.....% | 15.9 | 12.0 |
| Fixed carbon (by difference).....% | 6.0 | 8.8 |
| Calorific value (cals. per grm.)..... | 2,740 | 2,095 |

In run No. 1 made in the Hartman retort in St. John, due to the use of steam the crude oil was obtained along with considerable water distillate. Correction was made for this water as it separated by gravity from the oil, but the crude oil measured still consisted of an oil-water emulsion which on further standing separated out more water. By the time the samples reached the laboratory it was found that the oil had separated as a top layer practically free from water. Examination of the dry oil from run 1 in comparison with the dry oil from run 2 was fairly simple, but due

¹ Mines Branch Summary Report of Investigations 1921, p. 241.

to temperature and agitation changes during transit, and due to the nature of the containers in which the oil was shipped, it was difficult to determine accurately the amount of water contained in the crude wet oil as measured in run No. 1. For this reason check runs were made in the laboratory on Albert shale as used in run No. 1, with and without steam. A third run was also made on the paper shale to check the yield obtained at St. John. A summary of these laboratory distillation runs is as follows:—

TABLE III

| | Albert shale | | Paper shale |
|---|---------------|------------|---------------|
| | Without steam | With steam | Without steam |
| Shale used— | | | |
| Spent shale obtained as % of shale charged..... | 77.2 | 76.0 | 80.6 |
| Oil yield (Imp. gals. per 2,000 lbs.)— | | | |
| Crude oil as measured..... | 32.1 | 45.1 | 29.5 |
| Dry oil dehydrated..... | 32.1 | 31.6 | 29.5 |

The crude water-oil emulsion obtained by steam distillation from Albert shale contained an average of 30 per cent water, and correcting for this the yield from the Hartman retort in run No. 1 should be 32.2 instead of 46 Imperial gallons per ton. (See Table I).

EXAMINATION OF SHALE OILS

The specific gravity and distillation range of the dry shale oil samples from the Hartman retort may be compared with that of the dry oils obtained in the laboratory from the same shales as follows:—

TABLE IV

| | Albert shale | | Paper shale | |
|-------------------------------|----------------|-------------------|----------------|-------------------|
| | Hartman retort | Laboratory retort | Hartman retort | Laboratory retort |
| Specific gravity at 60°F..... | 0.883 | 0.861 | 0.854 | 0.886 |
| Distillation range— | | | | |
| 1st drop..... | 75°C. | 74°C. | 69°C. | 75°C. |
| 10% vol..... | 135 | 139 | 126 | 146 |
| 20% vol..... | 174 | 180 | 168 | 195 |
| 30% vol..... | 214 | 223 | 208 | 247 |
| 40% vol..... | 259 | 252 | 249 | 281 |
| 50% vol..... | 298 | 283 | 279 | 322 |
| 60% vol..... | 322 | 320 | 311 | 349 |
| 70% vol..... | 366 | 347 | 337 | 357 |
| 80% vol..... | 384 | 368 | 369 | 375 |
| End point..... | 390 | 300 | 390 | 390 |
| Recovery..... | 89% | 91% | 88% | 94% |

Examination of the above distillation ranges shows that the nature of shale oil obtained by the Hartman retorting process does not differ materially from the oil obtained in the laboratory by ordinary destructive distillation. The gravity of the shale oils from the Hartman retort for each shale differed slightly from that of the oils from the laboratory retort, but this may be accounted for either by rate of the primary destructive distillation or by particles of shale dust being carried over into the crude oil.

GAS YIELD AND WEIGHT BALANCE

The yield of gas during the trial runs in the Hartman retort was only roughly determined. The uncondensed gas produced was burned in standard gas jets the consumption of which was measured. The shale gas figure given in the above table is therefore an estimated yield. A check comparison of the gas yield obtained may however be made by drawing up a weight balance and comparing this with that obtained in the laboratory runs.

TABLE V
Weight balance—Products expressed as percentage of shale charged

| | Laboratory retort | | | Hartman retort | |
|-----------------------------------|-------------------|------------|---------------|----------------|---------------|
| | Albert shale | | Paper shale | Albert shale | Paper shale |
| | Without steam | With steam | Without steam | With steam | Without steam |
| Spent shale.....% | 79.4 | 75.0 | 80.6 | 75.9 | 81.3 |
| Dry oil.....% | 14.1 | 13.8 | 13.0 | 13.9 | 10.2 |
| Water in shale.....% | 0.7 | 0.9 | 0.5 | 0.7 | 0.5 |
| Gas and loss (by diff.).....% | 5.8 | 10.3 | 5.9 | 9.5 | 8.0 |
| Gas yield c.f./ton.....% | 1,285 | 3,540 | 1,030 | (1,650) | (1650) |
| Calorific value B. T. U./c.f..... | 535 | 395 | 450 | | |

Under laboratory retorting conditions where the temperature is controlled and where the maximum temperature does not exceed say 450°C. (840°F.) until all the shale oil is distilled off an oil shale yielding 13.8 to 14.0 per cent of its weight as (dry) oil, will, it has been found, give a gas yield averaging in the neighbourhood of 1,000 cu. ft. per ton. On allowing the temperature to rise appreciably at the end of the run the gas yield reaches as high as 1,500 cu. ft. per ton with the same oil yield. Using steam under these latter conditions will give gas yields ranging from 1,500 to 3,500 cu. ft. per ton, depending on the maximum temperature and the amount of steam coming into contact with the hot carbon of the spent shale. On the same shale the calorific value of the gas apparently varies with the yield. From records it is noticed that gas yields of 1,000 to 1,500 cu. ft. per ton show calorific values ranging from less than 400 to over 700 B.T.U. per cu. ft. with an average of about 550 B.T.U. An 8-lb. sample of oil shale distilled in a laboratory retort at a temperature of 550°C. (1,020°F.) in the shale gave a gas yield of over 3,500 cu. ft. per ton, the calorific value of which was slightly less than 400 B.T.U. per cu. ft.¹

The roughly determined figures of 1,600 to 1,700 cu. ft. per ton as the gas yield obtained in the runs in the Hartman retort corresponds with yields obtained in the laboratory under similar conditions. At this yield (with steam) the heating value of the gas will be in the neighbourhood of 500 B.T.U.

The heating value of city gas such as used for heating the retort also ranges as high as 500 B.T.U. per cu. ft., although the minimum allowable is 450 B.T.U. For practical purposes therefore the shale gas obtained may be considered of equal calorific value to the city gas. The heat units in the shale gas obtained as a ratio of the heat in the city gas used will therefore be according to the rates of each per hour as measured. The

¹ Mines Branch Summary Report 1918, page 84.

average rate at which the shale gas was given off was roughly 55 cu. ft. per hr. as against an average of 123 cu. ft. per hr. for the city gas used. The heat units in the shale gas obtained were apparently only about 45 per cent of the total heat requirements to carry on the distillation as demonstrated in the Hartman retort during the two trial runs made. Either by increasing the throughput or by increasing the efficiency for heating the retort by gas, or both, this figure of 45 per cent may be brought up considerably, but indications are that the claims made to the effect that sufficient shale gas was obtained to carry on the distillation are not substantiated.

COMPARISON OF SHALE OILS

A comparison of the specific gravity and distillation range of the shale oil obtained by the Hartman retort with that obtained by ordinary laboratory distillation has already been given. A summary comparison of the yield of crude oil obtained in the laboratory retort as compared with that in the Hartman retort is as follows:—

TABLE VI

| | Oil yield Imp. gals. per 2,000 lbs. | |
|-----------------------------------|--|----------------------|
| | Hartman retort | Laboratory retort |
| Albert shale— | | |
| Crude wet oil as measured..... | 46 | 45 |
| Crude oil less water content..... | 32 | 32 |
| Paper shale— | | |
| Crude wet oil..... | | 42 |
| Crude dry oil..... | 25 | 29 |

According to the above table it will be noticed that the yield of oil obtained in the laboratory retort from Albert shale checked the yield of crude oil in run 1 Hartman retort remarkably closely, but for the paper shale the laboratory yield was 29 Imp. gallons per ton of dry crude oil as against 25 in the Hartman retort.

From fractional distillation curves plotted from the distillation ranges given in Table IV the crude oils may be divided into light, medium, and heavy oil fractions. The fractions obtained by the laboratory fractionation from the crude oil from the Albert shale and the yield of these fractions per ton may be summed up as follows:—

TABLE VII

| | Hartman retort | | Laboratory retort | |
|---|------------------------------------|--|------------------------------------|--|
| | % dry oil distillate by vol. | Imp. gals. per ton of shale charged | % dry oil distillate by vol. | Imp. gals. per ton of shale charged |
| Light naphtha oils up to 150° C..... | 14.0 | 4.5 | 14.0 | 4.5 |
| Middle illuminating oils 150-300° C..... | 37.0 | 11.8 | 41.0 | 13.1 |
| Heavy lubricating stock 300° C to coke..... | 38.0 | 12.1 | 36.0 | 11.5 |
| Coke, by diff..... | 11.0 | | 9.0 | |

It will be seen from the above that the composition of the oil obtained from the Hartman retort was practically the same as that obtained from the laboratory retort.

THEORETICAL HEAT REQUIREMENTS

The specific heat of oil shale has been reported¹ as 0.265 and the endothermic heat of reaction (at approximately 400°C) as averaging slightly less than 450 cal. per gramme of oil and gas liberated. A single determination on Canadian (New Brunswick) oil shale was 484 cal. per gramme, equivalent to 870 B.T.U. per pound. This means that for every pound of shale oil and gas produced, the heat required to carry out the reaction at 400° to 410° C. was 870 B.T.U. for a 13.8 per cent yield of dry oil, and a minimum of 3.5 per cent² yield of gas, making a total of 17.3 per cent for every pound of shale distilled, the theoretical heat requirements are 0.173×870 B.T.U. equal to 150.5 B.T.U. On the assumption that the specific heat for the spent shale is the same as reported for the raw shale, viz., 0.265, the heat required to raise the spent shale to a temperature in the neighbourhood of 900°F., as obtained in the Hartman retort trial runs, would be 238.5 making a total of 389 B.T.U.

The capacity of the retort as demonstrated at St. John was approximately 75 pounds per hour with a gas consumption of 115 cu.ft. and 130 cu.ft. per hour respectively for the two runs. With the average consumption of 125 cu.ft. of 475 B.T.U. per cu.ft. gas the heat required to distill a pound of shale was about 790 B.T.U. Hence the distillation of the oil from the shale within the retort took place at an efficiency of slightly less than 50 per cent based on the theoretical heat requirements. This would indicate that when the efficiency of heating the shale in the retort could be raised to say 66 to 67 per cent, the throughput of the retort as demonstrated may be brought up to 100 lbs. per hour as claimed.

DEVELOPMENT OF CAPACITY OF RETORT

Special claims are made in the patent application for electric heating elements to be installed in the bottom plate and in the upper decks or shelves; and in the blue prints accompanying the prospectus—promoting the Hartman retort—a commercial plant size retort is draughted with the same design as the small retort in the patent drawings. It is not the intention of this report to discuss any other retort than that demonstrated at St. John, but it should be pointed out that the development of a new retort from a small (laboratory) scale to a semi-commercial or commercial scale generally proves difficult. In order to heat by electricity at less cost than with gas, low price current used in a cheap and simply designed retort would be necessary, and while it is conceivable that oil shale may be retorted on a large scale by electricity, it is difficult to see how the Hartman apparatus can be developed to a commercial size retort with an efficient electrical heating system. If the elements are cast in the shelves or decks as indicated in the patent drawings, the cost of replacing the burnt out elements would be high. It may be concluded that the development of the Hartman retort is distinctly in the preliminary stages, and that the

¹ The Thermal Decomposition of Oil Shales—by McKee and Lyder. The Journal of Industrial and Engineering Chemistry Aug. 1921.

² See Weight Balance, page 244, Mines Branch Summary Report for 1921.

development of an efficient heating system is still somewhat of a problem. It would appear that for a retorting operation in New Brunswick coal would be the cheapest fuel, with a combination of crude shale oil and producer gas (made by passing steam into hot spent shale) as second place.

THE USE OF STEAM

The Hartman retort is designed so that steam can be used. On the laboratory scale the use of steam greatly increases the yield of crude wet oil, but does not materially increase the yield of dehydrated or water-free oil.

One of the claims made for the use of steam is that it improves the quality of the oil by reducing the vapour pressure within the retort, and while no doubt the efficiency with which the heat may be conducted to different parts of the retort by convection currents may be improved, the most important use of steam is apparently to increase the gas yield. At temperatures of 550°C. and above the steam reacts on the hot carbon, resulting in the spent shale from the cracking of the hydrocarbons, to produce gas rich in carbon monoxide and hydrogen. The oil vapour present in the system will give a gas with heating value equal to or above that of ordinary water-gas.

Knowing the value of extra gas yield due to steam, and correlating this with the cost of steam, the extent to which steam can be used to obtain economical results can be calculated and the steaming operation so regulated.

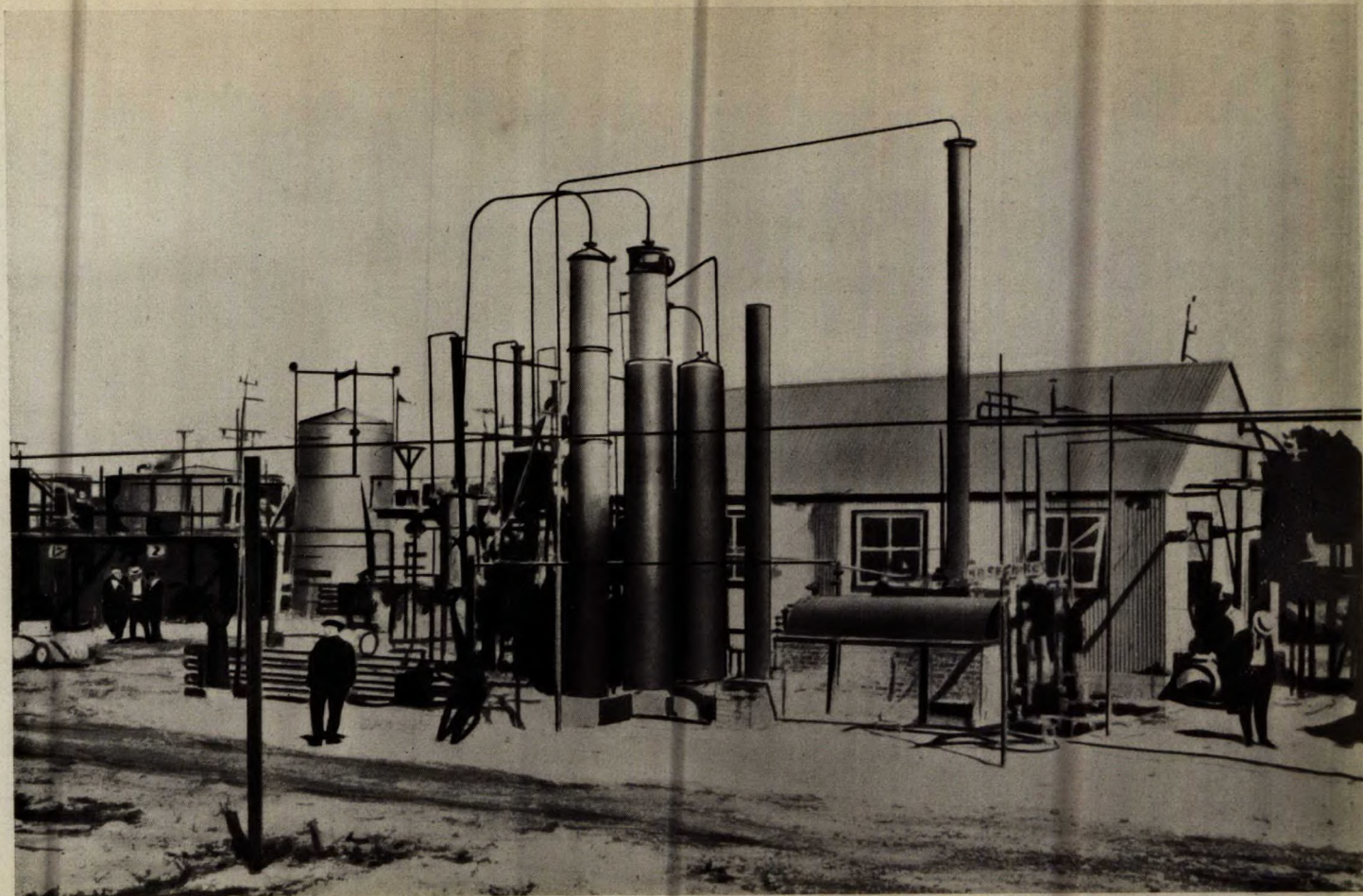
SUMMARY AND CONCLUSIONS

1. The Hartman retort is a shale handling apparatus for the recovery of crude shale oil from oil shales by destructive distillation. The retort as demonstrated at St. John, N.B., can be described as a continuous large scale laboratory apparatus, the capacity of which was from 75-100 lbs. of crushed shale per hour.

2. The crude wet oil produced by the Hartman retort, using steam, equivalent to a yield of 46 Imperial gallons per short ton, was a water-oil emulsion containing approximately 30 per cent of water. The yield of dry oil was 32 gallons per ton and this result is in close agreement with that obtained by the Standard (temperature control) method used in this laboratory. The specific gravity and distillation range of the dry oil from the Hartman retort were not materially different from those of the crude obtained in this laboratory.

3. In operating the retort at a temperature for optimum oil yield the amount of heat in the gas given off was less than 50 per cent of the total available heat in the gas used to carry on the retorting process.

4. The Hartman retorting process demonstrated at St. John had not progressed beyond the laboratory experimental stage and the development of the retort to a semi-commercial or commercial stage will probably not be free from mechanical difficulties. Furthermore, in the development of a shale oil industry in Canada, the retorting is not the greatest problem. The cost of mining a uniformly high-grade shale and the refining of crude oil into marketable products will in all probability be more important controlling factors. It is therefore not feasible to express an opinion as to the possibility of developing and operating the Hartman retort on a commercial scale.



VI

REPORT ON THE RAMAGE PROCESS

R. E. Gilmore and P. V. Rosewarne

Under instructions from the Chief of the Fuels and Fuel Testing Division the writers visited the plant of The Nations Oil Refineries at Montreal East in August, 1923. The purpose of this visit was to observe the Ramage process in operation in order to determine its merits as a technical process.

The Ramage process was originated by Alexander Sydney Ramage of the United States, and has been developed from the laboratory scale to the present stage, apparently under the inventor's personal direction. It is a process for the conversion of heavy petroleum oils into lighter oil products suitable for use as fuel in automobile, or other internal combustion engines, and is covered by several patents both in Canada and the United States. The process is said to be in operation on a commercial basis at the petroleum refinery of the Barnsdall Corporation, at Bigheart, Oklahoma. The raw materials in stock at The Nations Oil Refineries were light fuel or gas oil and heavy Mexican fuel oil. This report has to do only with gas oil as the raw material and covers three runs made in the experimental plant at Montréal.

After a preliminary survey of the plant a general plan for carrying out the experimental runs was adopted, namely, that the Chief Engineer and his assistants should proceed to make a series of runs under the observation of the representatives of the Fuel Testing Laboratories, who checked all oil measurements and operation data. Data sheets are attached for the complete runs, as well as volume and weight balance sheets. A photograph of the plant and diagrams of the process are given in order to make the descriptions more easily understood.

DESCRIPTION OF PROCESS

The Ramage process is a process for converting the heavier petroleum oil products into lighter oil products suitable for use as fuel in internal combustion engines. The term "Hydrogenation" has been applied to the process, but the writers have not yet sufficient data to enable them to express an opinion as to whether the process may be properly so described or not.

Briefly the process consists of passing petroleum vapour mixed with steam in certain proportions over ferric oxide heated to a temperature of about 1,000° F., then fractionating, and condensing the resultant product.

In most processes for the production of motor fuel from heavy petroleum oils the reaction takes place with the production of uncondensable gases and free carbon. This free carbon if allowed to accumulate often gives serious trouble. In the Ramage process the accumulation of free carbon is apparently prevented by the use of steam. It is to be noted, too, that the reaction in the Ramage process takes place at pressures that are only slightly above atmospheric pressure, whereas some processes require upwards of one hundred and fifty pounds pressure per square inch.

The chemical reactions taking place in the Ramage process are complex. It is claimed that the ferric oxide is reduced to ferrous oxide at the temperature and under the conditions prevailing in the converter; that the oxygen so liberated combines with the carbon formed due to the disassociation of the hydrocarbons, which takes place at the temperature maintained; that the steam also disassociates at this temperature, liberating oxygen which oxidizes the iron back to the ferric condition and so leaves it ready for further reaction on additional heavy oil vapour. It is not the purpose of this report to enter into a discussion of the theory of these reactions, nor is a sufficient amount of data available at the present time to warrant such a discussion, but the absence of any appreciable deposit of carbon in the apparatus is worthy of note, as well as the presence of hydrogen, of carbon monoxide and carbon dioxide in the uncondensed gases. It may be pointed out, too, that the ferric oxide before being used in the process is of a characteristic red colour and the lumps are rather easily crumbled; after use the oxide is dull black in colour and can be crumbled only with difficulty. The black oxide appears to react satisfactorily in the converter for an indefinite length of time.

DESCRIPTION OF PLANT

The plant of The Nations Oil Refineries at Montreal East may be described under three headings, namely:—

- (a) Power House.
- (b) Experimental Plant.
- (c) Tank and Accessory Equipment.

Power House

The Power House was a large building which housed steam boilers and accessories, designed to supply power and steam for a refinery which was in process of construction. The boilers were heated with oil fuel. An air compressor, driven by electric power, supplied air for the burners. A small laboratory was fitted up in a corner of the power house.

Experimental Plant

The Experimental Plant was a semi-commercial unit and may be considered in three parts, namely:—

- (a) The Converter System.
- (b) The Scrubber System.
- (c) The System for Chemical Treatment.

Converter System.—The Converter System was composed of the following units suitably joined together:—

- i. A cylindrical still or tank, heated with oil burners.
- ii. A converter furnace composed of four tubes to contain the oxide, with provision made so that the temperature of the tubes could be controlled within narrow limits.
- iii. Two upright fractionation towers.
- iv. A condenser cooled with water.
- v. A separator, or receiver for the condensate.

Scrubber System.—The Scrubber System was made up of the following units suitably joined together:—

- i. An ordinary gas holder and direct reading gas meter.
- ii. A steam-driven compression pump, a cooler, ice condenser, and pressure tank.
- iii. Two scrubber towers.
- iv. An oil-fired still, reflux tower, a water-cooled condenser, and a separator or receiver for the condensate.
- v. A heat exchanger to preheat the incoming scrubber oil and to cool the effluent still bottoms.

System for Chemical Treatment.—The system for chemical treatment consisted of two conical agitator tanks with suitable connexions to draw off the sludge after treatment and to handle the semi-refined light oil.

Tank and Accessory Equipment

Eight iron storage tanks for crude or gas oil were conveniently located. The tanks were cylindrical and set on end. Proper connexions were made for filling and emptying them. A separate large cylindrical iron tank, set horizontally, was used as storage for fuel oil for the oil burners. Double action steam-driven pumps of various sizes were used wherever necessary to transfer oil to the various parts of the plant. The miscellaneous equipment included such necessary articles as, thermometers, gauges, measures, steel drums, pails, tools, supplies, etc.

CYCLE OF OPERATIONS

The cycle of operations as carried out at Montreal may be described under three headings, namely:—

- (a) Converter System.
- (b) Scrubber System.
- (c) System for Chemical Treatment.

In order that the description may be followed more readily, a diagram of the process and a flow sheet of the products is shown in Figures 4 and 5 respectively.

Converter System

Gas oil or fuel oil is pumped from storage tank 4 into still 11, until the still is about one-half full. The oil burners under the still are then lighted and the oil heated until a pressure of eight to ten pounds per square inch is developed within the still. The petroleum vapour is then admitted to the converter manifold where it is mixed with steam from the power house at about twenty pounds pressure per square inch. The mixed vapours are then forced through nozzles into the converter tubes, which have been heated to a temperature of about 1000° F. The mixed vapours pass through the tubes over the ferric oxide cubes, where part of the heavy petroleum vapours of the gas oil is altered or converted into vapours of lighter oils.

The vapours resulting from the converter reaction pass into tower 1 at the base and flow upward. From the top of tower 1 the vapours are led to the base of tower 2, where they flow upward again, through tower 2 and thence to condenser 1. In condenser 1 the greater part of the

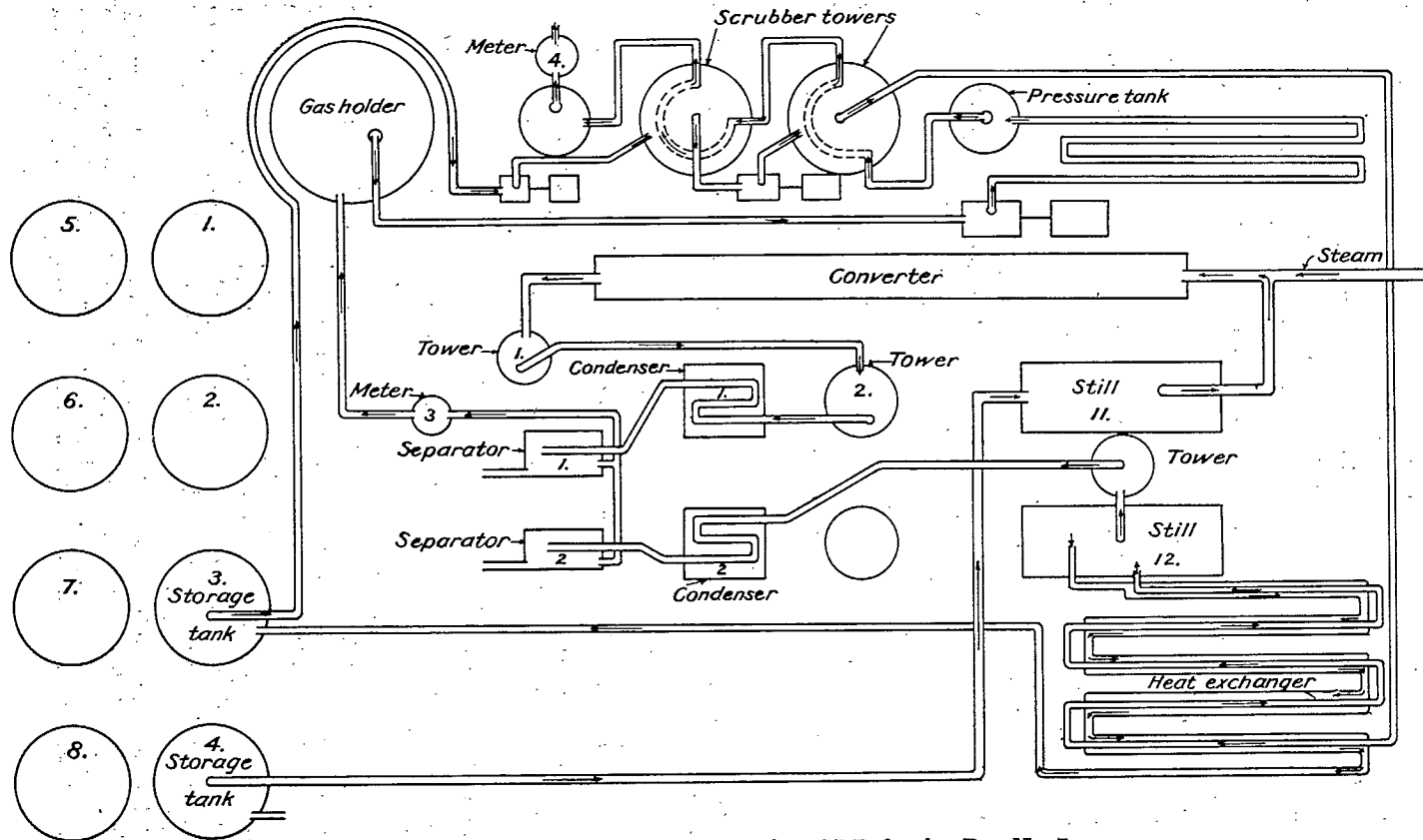


FIGURE 4. Ramage process, The Nations Oil Refineries, Run No. 7

vapours is condensed and passed on to separator 1, which has previously been charged with thirty to thirty-five gallons of any light petroleum oil, such as kerosene, gasoline, or the product of a previous run. This is to prevent a sudden rush of vapour through the condenser into the gas lines. The condensate which is caught in separator 1 contains a certain amount of water which settles to the bottom of the separator and can be drawn off separately from the light oil condensate. The uncondensed gases rise to the top of the separator and pass on through meter 3 to the gas holder.

In both tower 1 and tower 2 a certain amount of unconverted oil condenses on the walls and falls to the bottom of the towers. This oil which is called tower bottoms, is returned to still 11 continuously to be vapourized and again passed through the converter tubes. By mixing with these still bottoms the necessary amount of gas oil to replace what has been converted, the process may be made practically continuous in so far as the converter system is concerned.

Scrubber System

The gas compressor draws the gas from the gas holder, compresses it to about forty pounds pressure per square inch, forces it through the water-cooling system and ice condenser into the pressure tank, in which a certain amount of liquid condenses due to the pressure and low temperature. The residual gas then passes up through one scrubber tower, is brought down to the base of the other tower, passes up through it, then through meter 4 and is discharged into the air as stripped gas. It is intended to use the gas as fuel when the plant is completed.

While the gas passes up through the scrubber tower gas oil from the storage tanks is pumped to the top of the second tower and allowed to flow down counter current to the gas. As fast as it collects at the base of the second tower it is pumped to the top of the first one and again flows down to wash the rising gas free of light oils. This scrubber oil with its charge of absorbed light oils is then pumped through the inner part of the heat exchanger into still 12. In still 12 the absorbed light oil is distilled off through tower 3, and the hot residual gas oil is pumped out of still 12 through the outer part of the heat exchanger back to the storage tanks again. The light oil vapours pass up through tower 3 to condenser 2 where it is condensed and passes on to separator 2. Any uncondensed gas rises to the top of separator 2 and passes out to unite with the gas from separator 1. The mixed gas then passes through meter 3 and so to the gas holder again.

Chemical Treatment

The mixed light oil condensate from separator 1 and separator 2 is transferred to one of the agitator tanks and treated first with concentrated sulphuric acid and then with caustic soda-litharge solution. After the resultant sludge has been drawn off the light oils are distilled from still 12 at intervals.

RAW MATERIALS

The raw materials used in the Ramage process may be included in three groups, namely:—

- (a) Gas Oil.
- (b) Ferric Oxide.
- (c) Fuel Oil.

Gas Oil

The oil used as the basis of the process at Montreal was what is known as gas oil, which may be described as a partially refined petroleum product intermediate between illuminating oils and the heavier fuel oils. It may be also described as a light fuel oil. The oil used for the runs observed had a specific gravity of 0.890 at 60°F. and an initial boiling point of about 320°F. The lower part of the average distillation curve is shown in Diagram VI.

Ferric Oxide

The ferric oxide used for filling the converter tubes was a good grade of powdered red oxide of iron. Analysis showed only a trace of sulphur as impurities. The powdered oxide was mixed into a paste with water, cut into cubes and allowed to air dry. When dry they were rather friable but were nevertheless hard enough to stand ordinary handling. The cubes so prepared were placed in the converter tubes.

Fuel Oil

The fuel oil used to generate steam and to heat the stills and converter furnace was ordinary fuel oil with a specific gravity of 0.877 at 60°F., a flash point of 140°F., and an initial boiling point of 295-300°F. A heavier oil was also used but with less satisfaction, apparently because it was difficult to atomize it properly with compressed air.

FINAL PRODUCT

The product examined by the writers at Montreal was a clear, colourless, mobile liquid with a peculiar aromatic-like odour. It was called, Super Gas, at The Nations Oil Refineries.]

A sample of a run previous to the runs witnessed by the writers had a specific gravity of 0.770 at 60°F., and a distillation range of from 111°F., initial point, to an end point of 334°F. The light oil product from runs 6, 7, and 8, before chemical treatment, showed a specific gravity of 0.809 at 60°F., and a distillation range of from 153°F. to 514°F. It was yellowish brown in colour. Both the refined and the semi-refined product gave an iodine number of over 31.

It will be seen from the above analysis that super gas differs from ordinary gasoline as sold on the Canadian market, especially in regard to the specific gravity. Ordinary gasoline with a similar distillation range would have a specific gravity of approximately 0.710 to 0.750 at 60°F. Super gas also is different from ordinary gasoline in that it is miscible to a large extent with denatured ethyl alcohol, while gasoline is miscible to a very small extent.

The super gas produced at the plant at Montreal appeared to be a very satisfactory fuel for internal combustion engines, such as the automobile motor. The motors ran smoothly, but no definite test was made. Diagram VII shows 3 distillation curves for comparison.

SUMMARY OF OPERATING DATA

Three complete runs were made in the plant described above. These were called runs 6, 7, and 8 respectively. The following is a summary of the operating data.

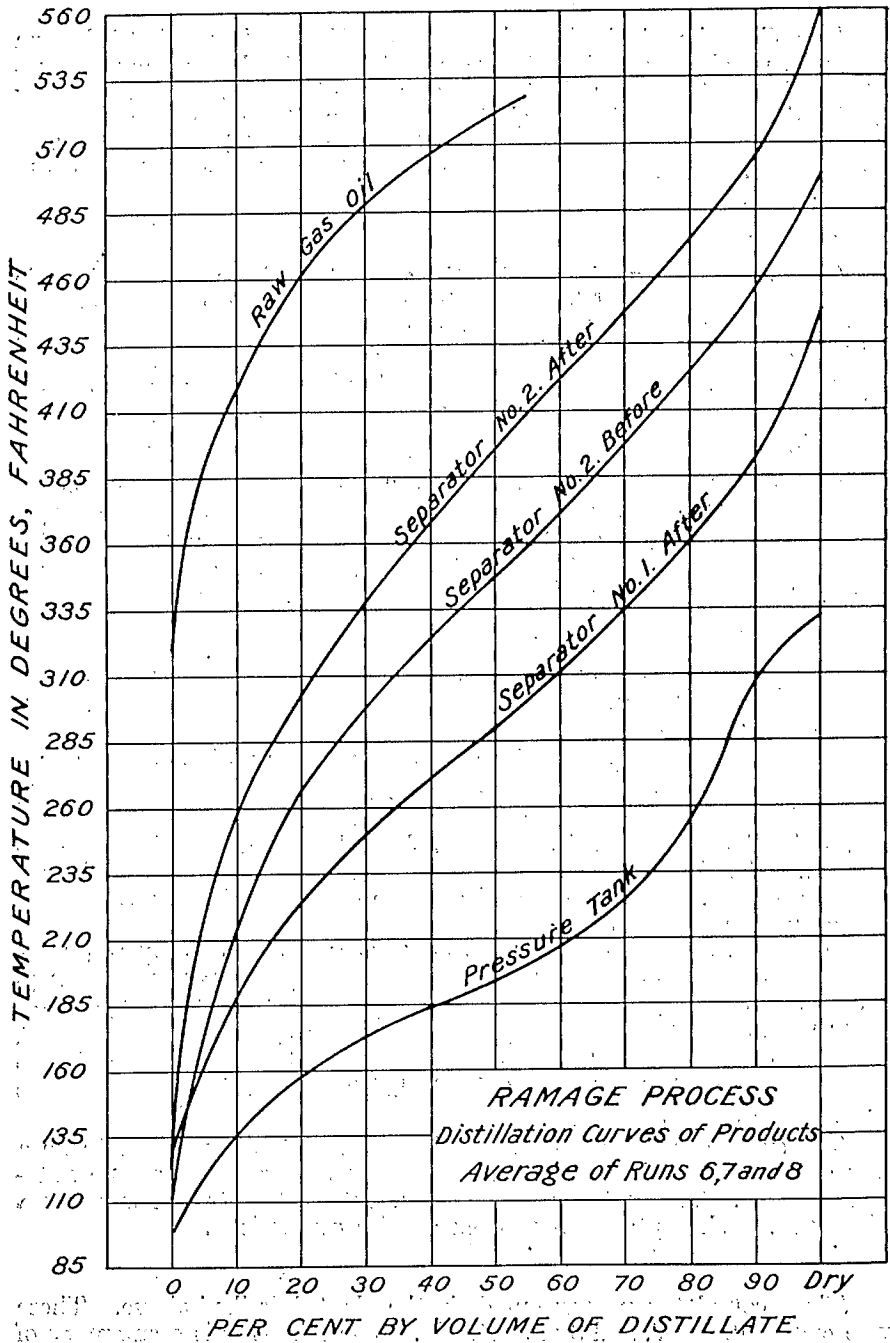
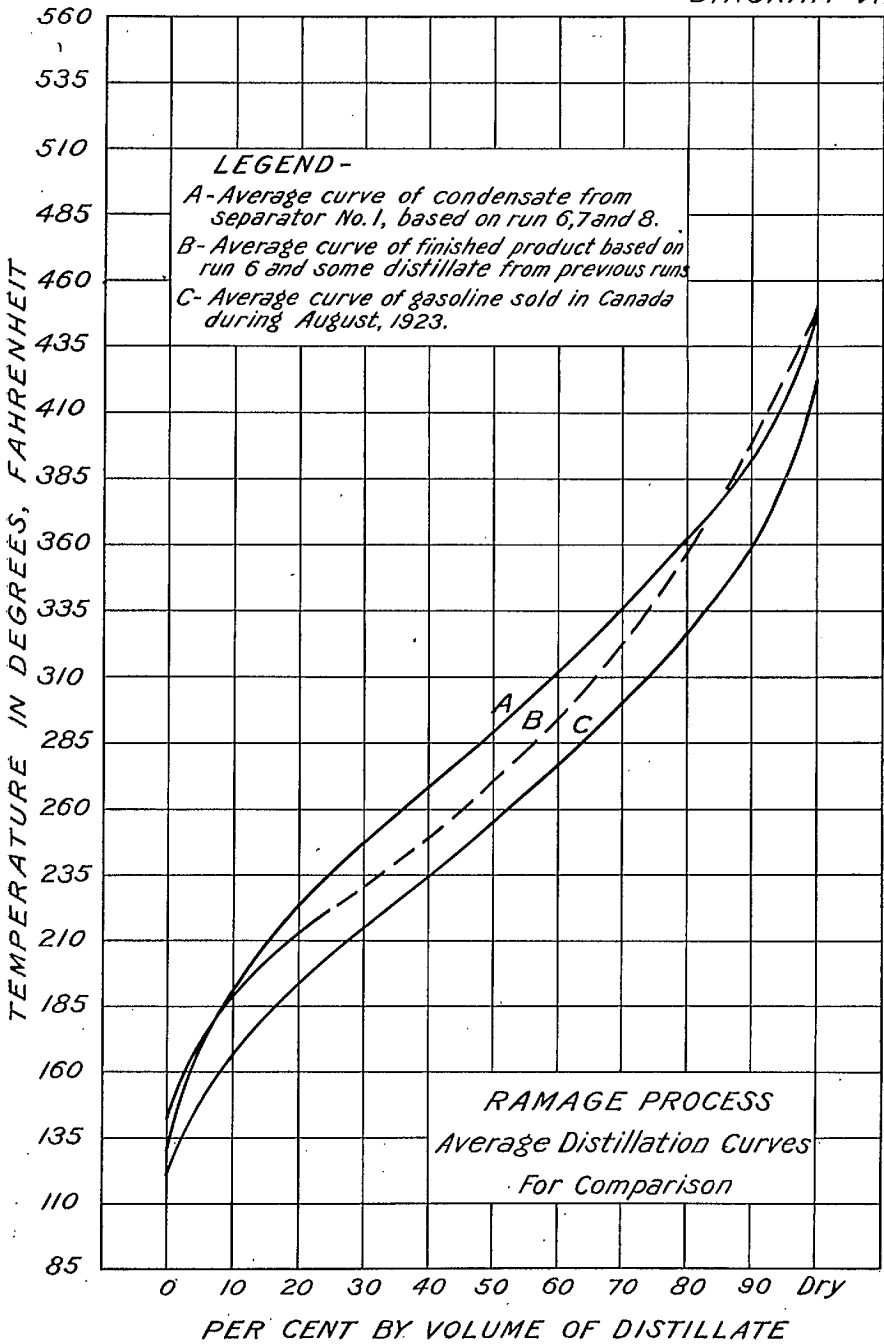


DIAGRAM VII.



| | Run 6 | Run 7 | Run 8 |
|--|---------|---------|---------|
| Date of experimental run..... | Aug. 16 | Aug. 22 | Aug. 23 |
| Actual duration of run, hours..... | 7 | 5½ | 5½ |
| Average pressure at steam orifice, pounds..... | 23.3 | 20.4 | 19.5 |
| Average pressure at oil still, pounds..... | 9.5 | 10.2 | 11.2 |
| Average temperature, still No. 11, (°F.)..... | 544 | 564 | 526 |
| Average temperature front of converter, (°F.)..... | 1,105 | 1,040 | 1,050 |
| Average temperature rear of converter (°F.)..... | 1,120 | 1,081 | 1,082 |
| Average pressure rear of converter, lbs..... | 1.4 | 0 | 1.3 |

SUMMARY OF RESULTS

Overall Efficiency

The purpose of the process is to obtain light oils suitable for use as motor fuels from heavy petroleum oils. The total amount of light oil obtained in the three runs was 165 gallons, as shown in Table IV. If a final product with an end point of 450°F. be considered a satisfactory fuel for motors, it is to be observed from Tables XVII-XIX that the light oil condensate from the separators requires a further fractionation in order to remove the heavy ends. It is shown in Tables IV, V, and VI that only 76 per cent of the total light oil, or 126 gallons for the three runs, was obtained which distilled over below 450°F. The total amount of oil charged in the three runs was 3,274 gallons, as shown in Table I. Thus the overall efficiency for the three runs was slightly less than 3.9 per cent. As by-product, also shown in Table I, there was produced 415 gallons of heavy fuel oil, and 6,718 cubic feet of gas, which had an average calorific value of 610 British thermal units per cu. ft. of gas.

Efficiency of the Converter System

The object of the converter system is to produce a light oil, which after suitable treatment may be used as a motor fuel. As shown in Table II, the total amount of light oil obtained from separator 1, during the three runs, was 32.6 gallons. In order to obtain that amount, over 586 gallons were treated in still 11. Table V shows that 99 per cent of the condensate from separator 1 had a distillation end point below 450°F., that is, approximately 32 gallons. Thus the converter system yielded 5.6 per cent of light oil boiling below 450°F. from 586 gallons of raw oil. The by-products obtained were 415 gallons of fuel oil somewhat heavier than the raw oil used to charge the still, as shown in Table I, and part of 15,100 cubic feet of unstripped gas with a calorific value of 705 British thermal units per cu. ft., as shown in Table II.

Efficiency of the Scrubber System

The purpose of the scrubber system is to recover in liquid form as much oil as possible from the uncondensed gases from separator 1. The design of the plant, however, makes any attempt to determine the efficiency of the scrubbers very difficult, because the gas measured by meter 3 comes from two sources, namely:—(a) the Converter System, (b) the Scrubber System. If this fact is kept in mind it may be possible to form some estimation of the degree of efficiency of the system, but such estimation can only be considered as approximation.

From the two sources 15,100 cubic feet of gas were obtained. The total amount of light oil from the three runs, boiling below 450°F., as shown in Table VI, was 88.3 gallons from separator 2 and 4.4 gallons from the pressure tank, making a yield of 92.7 gallons from the scrubber system. In order to obtain that amount of light oil, 2,687 gallons of scrubber oil was handled, as shown in Table II, showing a percentage yield of 3.5. It seems obvious from Table IX and Diagram VI that a large proportion of the above yield is due to the low initial boiling point of the scrubber oil. Table XI shows the oil used or obtained per 1,000 cubic feet of gas treated.

Efficiency of Converter

The purpose of the converter is to convert or alter a heavy petroleum oil into light oils suitable for motor fuel. It is to be observed from Table IX that the gas oil used as raw material had an average initial boiling point of 320°F. It follows then that only that portion of the condensate boiling below 320°F. may be taken as a whole as a converted product. However, it is likely that some of the higher boiling fractions of the gas oil are converted to lower boiling fractions, which, although lower in boiling point than the fraction from which they have been split off, are nevertheless higher in boiling point than those fractions which boil at the initial boiling point of the gas oil. The amount so converted could not be ascertained with any degree of exactness. The writers therefore assumed that the light oil boiling below 350°F. was equivalent, for all practical purposes, to the amount of light oil converted by the converter. The converter efficiency may be obtained therefore by determining the amount of light oil boiling below 350° F. in the products coming through the converter and expressing that amount of light oil as per cent of the heavy oil entering the converter. In the runs under observation, Tables XII and XIII show that 298.7 gallons of oil passed through the converter, and that 78.1 gallons of light oil, boiling below 350°F., were obtained. The yield was, therefore, 26.1 per cent.

COMMENTS

Design of the Plant and Apparatus

The writers observed several details in design of the plant which seemed of doubtful value for experimental purposes. One of them was the connexion between separator 1 and separator 2 to which reference has already been made. This connexion resulted in gases from the scrubber system mixing with those from the converter system and made it practically impossible to estimate the efficiency of the scrubber system. It also left the writers at a loss in regard to interpretation of the analyses of the gases since neither the relative amounts nor difference in composition of the gas produced by the two systems could be determined. It also permitted during run 7 the absorption of some of the gas by the charge of light oil in separator 2 without those gases passing through meter 3.

The latter result suggests inefficient operation of condenser 1, or of separator 1, and the writers incline to the opinion that the design of the separator might be improved. As mentioned earlier in this report the uncondensed gases and condensed liquid from condenser 1 were led to the bottom of separator 1 and below the surface of the liquid in the separator.

This operation resulted in the uncondensed gases being saturated with vapours of the condensate and possibly carrying over some liquid mechanically.

Operating Difficulties

The writers also foresee a certain number of operating difficulties, for instance, the possible blocking of the converter tubes. In the converter as operated at Montreal partial or complete plugging of a tube may take place without being detected until an appreciable decrease in the output is apparent. The liberal use of pressure gauges will of course aid in locating trouble of this kind but will not prevent its occurrence. The practice of blowing compressed air through the tubes at intervals was adopted at Montreal after the trouble was encountered and appeared to remove the sulphur compounds from the catalyst. It would also tend to remove any free carbon which might be formed. Whether or not this practice would prevent plugging of the tubes completely can only be proved by trial. Another difficulty likely to appear would be that of filling the tubes with the oxide in such a way that equal amounts of vapour could pass through each tube. This difficulty will likely increase in proportion to the number of tubes operated from one manifold. It is possible that radical improvements in the tube design and in the catalyst carrier may be developed, such as impregnating sponge iron with the ferric oxide and supporting it in the tube in such a way that the resistance to the flow of the vapours is decreased and the transference of heat improved without lessening the intimate contact of the vapours and the catalyst.

CONCLUSIONS

1. The Ramage process, using heavy petroleum as raw material, yields a product which has characteristics differing somewhat from those of ordinary gasoline on the market at present.

2. A yield of 165 Imperial gallons of semi-refined light oil was obtained as the main product from approximately 3,300 gallons of gas oil in three runs at the experimental plant in Montreal.

3. The by-products obtained from the three runs consisted of 415 gallons of fuel oil heavier than the gas oil used and approximately 6,700 cubic feet of gas with a heating value of over 600 B.T.U. per cubic foot.

4. The estimated amount of redistilled motor fuel to be obtained from the above 165 gallons is 126 gallons, no allowance being made in that estimation for chemical treatment and subsequent refining losses.

5. One gallon of motor fuel, boiling below 450°F., was obtained by handling 4.7 gallons of oil in the vapourizing still, and an additional 21.3 gallons in the scrubber system.

6. The converter efficiency was 26.1 per cent, which means that for every 100 gallons of gas oil, boiling above 350°F., that passed through the converter, only a little more than 26 gallons of light oil, boiling below 350°F., were produced.

7. The writers consider the process was still in the development stage at the time of their visit. The development of the converter tubes in particular as catalyst carriers will have to proceed with care in order to overcome the difficulties now foreseen.

8. The economical development of the process will necessarily depend upon the market conditions of the particular products obtained in comparison with the heavy oils used as raw material.

TABLE I.
Summary of Volume Balance

| | | |
|--|-------|------------|
| Gas oil used:— | | |
| As raw material..... | 587 | gallons |
| As scrubber oil..... | 2,687 | " |
| Total..... | 3,274 | " |
| Gas oil recovered:— | | |
| As heavy fuel oil ¹ | 415 | gallons |
| As spent scrubber oil..... | 2,564 | " |
| As intermediate oil (over 450°F.)..... | 39 | " |
| Total..... | 3,018 | " |
| Loss of gas oil ² | 256 | gallons |
| Motor fuel ³ obtained (below 450°F.)..... | 126 | " |
| Stripped gas..... | 6,718 | cubic feet |

¹The fuel oil and scrubber oil recovered had a higher specific gravity and a higher initial boiling point than the raw material used.

²To this figure must be added the fuel oil necessary to heat the stills, boiler, and converter and to provide power to operate oil pumps and air compressors.

³No allowance has been made for loss through chemical treatment and subsequent refining losses.

TABLE II
Volume Balance

| Run No. | Imperial gallons at 60°F. | | | |
|--------------------------------------|---------------------------|------------|------------|-------------|
| | 6 | 7 | 8 | Total |
| Oil Charged— | | | | |
| (a) Raw oil charged to still 11..... | 217.5 | 192.0 | 177.0 | 586.5 |
| (b) Raw oil charged to scrubber..... | 1,061.0 | 815.0 | 811.0 | 2,687.0 |
| Total oil charged..... | 1,278.5 | 1,007.0 | 988.0 | 3,273.5 |
| Products Obtained— | | | | |
| (a) From converter system— | | | | |
| Still 11 bottoms..... | 113.6 | 66.0 | 70.0 | 249.6 |
| Drips from converter..... | 6.2 | 15.7 | 16.3 | 38.2 |
| Tower 1 bottoms..... | 18.4 | 17.4 | 29.2 | 65.0 |
| Tower 2 bottoms..... | 10.5 | 38.9 | 12.8 | 62.2 |
| Separator 1 gain..... | 14.1 | 12.7 | 5.8 | 32.6 |
| Unstripped gas..... | 7,910 c.f. | 4,357 c.f. | 2,840 c.f. | 15,107 c.f. |
| Total oil from converter system..... | 162.8 | 150.7 | 134.1 | 447.6 |
| (b) From scrubber system— | | | | |
| Compression oil..... | 2.3 | 1.1 | 1.0 | 4.4 |
| Separator 2 gain..... | 56.0 | 7.3 | 64.8 | 128.1 |
| Oil recovered..... | 1,016.0 | 823.0 | 725.0 | 2,564.0 |
| Stripped gas..... | 3,766 c.f. | 2,194 c.f. | 758 c.f. | 6,718 c.f. |
| Total oil from scrubber system..... | 1,074.3 | 831.4 | 790.8 | 2,696.5 |
| Total oil from converter system..... | 162.8 | 150.6 | 134.1 | 447.6 |
| Total oil from scrubber system..... | 1,074.3 | 831.4 | 790.8 | 2,696.5 |
| Total oil products obtained..... | 1,237.1 | 982.0 | 924.9 | 3,144.1 |
| Total oil charged..... | 1,278.5 | 1,007.0 | 988.0 | 3,273.5 |
| Total oil products obtained..... | 1,237.1 | 982.0 | 924.9 | 3,144.1 |
| Loss..... | 41.4 | 25.0 | 63.1 | 129.4 |
| Per cent loss..... | 3.3 | 2.3 | 6.4 | 4.0 |

TABLE III
Weight Balance

| Run No. | Weight in pounds | | | |
|---|------------------|----------------|----------------|-----------------|
| | 6 | 7 | 8 | Total |
| Oil Charged— | | | | |
| (a) Raw oil charged to still 11..... | 1,931 | 1,707 | 1,576 | 5,214 |
| (b) Raw oil charged to scrubber..... | 9,443 | 7,253 | 7,112 | 23,808 |
| (c) Unstripped gas..... | 472 | 260 | 170 | 902 |
| Total oil charged..... | 11,846 | 9,220 | 8,858 | 29,924 |
| Products Obtained— | | | | |
| (a) From converter system— | | | | |
| Still 11 bottoms..... | 1,038.3 | 617.1 | 650.3 | 2,305.7 |
| Drips from converter..... | 53.9 | 130.1 | 143.6 | 336.6 |
| Tower 1 bottoms..... | 168.4 | 164.1 | 260.0 | 592.5 |
| Tower 2 bottoms..... | 90.2 | 345.8 | 108.4 | 544.4 |
| Separator 1 gain..... | 119.3 | 100.3 | 47.3 | 266.9 |
| Unstripped gas..... | 472.0 | 260.0 | 170.0 | 902.0 |
| Total from converter system..... | 1,942.1 | 1,626.4 | 1,379.6 | 4,948.1 |
| (b) From scrubber system— | | | | |
| Compression oil..... | 18.3 | 8.7 | 7.6 | 34.6 |
| Separator 2 gain..... | 466.0 | 54.8 | 540.0 | 1,060.8 |
| Oil recovered..... | 9,032.2 | 7,325.0 | 6,467.0 | 22,824.2 |
| Stripped gas..... | 169.0 | 121.0 | 41.0 | 331.0 |
| Total from scrubber system..... | 9,685.5 | 7,509.5 | 7,055.6 | 24,250.6 |
| Total from converter system..... | 1,942 | 1,626 | 1,380 | 4,948 |
| Total from scrubber system..... | 9,686 | 7,510 | 7,056 | 24,252 |
| Total products obtained..... | 11,628 | 9,136 | 8,436 | 29,200 |
| Total oil charged..... | 11,846 | 9,220 | 8,858 | 29,924 |
| Total products obtained..... | 11,628 | 9,136 | 8,436 | 29,200 |
| Loss..... | 218 | 84 | 422 | 724 |
| Per cent loss..... | 1.8 | 0.9 | 4.8 | 2.5 |

TABLE IV
Gallons of Light Oil Obtained

| Run No. | 6 | 7 | 8 | Total |
|--------------------|-------------|-------------|-------------|--------------|
| Pressure tank..... | 2.3 | 1.1 | 1.0 | 4.4 |
| Separator 1..... | 14.1 | 12.7 | 5.8 | 32.6 |
| Separator 2..... | 56.0 | 7.3 | 64.8 | 128.1 |
| Total..... | 72.4 | 21.1 | 71.6 | 165.1 |

TABLE V
Per cent of Light Oil Boiling Below 450° F.

| Run No. | 6 | 7 | 8 | Average |
|--------------------|-----|-----|-----|---------|
| Pressure tank..... | 100 | 100 | 100 | 100 |
| Separator 1..... | 97 | 100 | 100 | 99 |
| Separator 2..... | 69 | 95 | 77 | 80.3 |

TABLE VI
Gallons of Light Oil Boiling Below 450° F.

| Run No. | 6 | 7 | 8 | Total |
|--------------------|------|------|------|-------|
| Pressure tank..... | 2.3 | 1.1 | 1.0 | 4.4 |
| Separator 1..... | 14.4 | 12.7 | 5.8 | 32.9 |
| Separator 2..... | 37.6 | 6.4 | 44.3 | 88.3 |
| Total..... | 54.3 | 20.2 | 51.1 | 125.6 |

Total light oil obtained = 165 gallons.

$$\text{Percentage of light oil below 450°F.} = \frac{126 \times 100}{165} = 76\%.$$

TABLE VII
Light Oil from Separator 2 (Below 450° F.)

| Run | Oil drawn | | | Oil charged | | | Net light oil |
|------------|----------------|---------------------|--------------------|----------------|---------------------|--------------------|--------------------|
| | Gals. measured | % Vol. below 450°F. | Gals. below 450°F. | Gals. measured | % Vol. below 450°F. | Gals. below 450°F. | Gals. below 450°F. |
| No. 6..... | 39.0 | 69 | 61.4 | 33.0 | 72 | 23.8 | 37.6 |
| No. 7..... | 40.6 | 95 | 38.6 | 33.3 | 97 | 32.2 | 6.4 |
| No. 8..... | 98.1 | 77 | 75.6 | 33.3 | 94 | 31.3 | 44.3 |
| Total..... | | | | | | | 88.3 |

TABLE VIII
Light Oil from Separator 1 (Below 450°F.)

| Run | Oil drawn | | | Oil charged | | | Net light oil |
|------------|----------------|---------------------|--------------------|----------------|---------------------|--------------------|--------------------|
| | Gals. measured | % Vol. below 450°F. | Gals. below 450°F. | Gals. measured | % Vol. below 450°F. | Gals. below 450°F. | Gals. below 450°F. |
| No. 6..... | 47.1 | 97.5 | 45.9 | 33.0 | 95.5 | 31.5 | 14.4 |
| No. 7..... | 46.0 | 100.0 | 46.0 | 33.3 | 100.0 | 33.3 | 12.7 |
| No. 8..... | 39.1 | 100.0 | 39.1 | 33.3 | 100.0 | 33.3 | 5.8 |
| Total..... | | | | | | | 32.9 |

TABLE IX
Initial Boiling Point of Oils

| Run No. | 6 | 7 | 8 | Average |
|--------------------------|-----|-------|-----|---------|
| Gas oil °F..... | 304 | 331 | 325 | 320 |
| Scrubber oil °F..... | 322 | | 329 | 325 |
| Still 11 bottoms °F..... | 392 | 525 | 491 | 469 |
| Still 12 bottoms °F..... | 374 | | 361 | 368 |

TABLE X
Gas and Oil Produced from Scrubber System

| Run No. | 6 | 7 | 8 | Total |
|--|-------|-------|-------|--------|
| Volume of gas through meter 3 in cubic feet..... | 7,910 | 4,357 | 2,840 | 15,107 |
| Volume of gas through meter 4, in cubic feet..... | 3,766 | 2,194 | 758 | 6,718 |
| Volume of scrubber oil used in gallons..... | 1,061 | 815 | 811 | 2,687 |
| Oil obtained from pressure tank, in gallons..... | 2.3 | 1.1 | 1.0 | 4.4 |
| Light oil from separator 2 (below 450°F.) in gallons.... | 38.6 | 7.1 | 49.9 | 95.6 |
| Total light oil from separator 2 and pressure tank..... | 40.9 | 8.2 | 50.9 | 100.0 |

TABLE XI
Oil Used or Obtained per 1,000 cu. ft. of gas

| | Gallons per 1,000 c.f. of gas |
|-----------------------------------|-------------------------------|
| Total light oil from gas..... | 6.6 |
| Light oil from pressure tank..... | 0.3 |
| Scrubber oil handled..... | 149.6 |

TABLE XII
Converter Efficiency

| | Imperial gallons at 60°F. | | | | | |
|--|---------------------------|------------------------|-------------------|------------------------|-------------------|------------------------|
| | Run 6 | | Run 7 | | Run 8 | |
| | Gals. as measured | Light oil below 350°F. | Gals. as measured | Light oil below 350°F. | Gals. as measured | Light oil below 350°F. |
| Raw gas oil— | | | | | | |
| Charged to still 11..... | 217.5 | 0 | 192.0 | 0 | 177.0 | 0 |
| Still 11 bottoms..... | 113.6 | 0 | 66.0 | 0 | 70.0 | 0 |
| Drips not entering converter..... | 6.2 | 0 | 15.7 | 0 | 16.3 | 0 |
| Net raw oil through converter..... | 97.7 | 0 | 110.3 | 0 | 90.7 | 0 |
| Products— | | | | | | |
| Tower 1 and 2 bottoms..... | 28.9 | 1.55 | 56.3 | 5.45 | 42.0 | 3.45 |
| Separator 1 net oil..... | 14.1 | 10.76 | 12.7 | 6.21 | 5.8 | 4.18 |
| Oil from pressure still..... | 2.3 | 2.30 | 1.1 | 1.10 | 1.0 | 1.00 |
| Separator 2 net oil..... | 56.0 | 12.91 | 7.3 | 6.43 | 64.8 | 14.58 |
| Scrubber oil increase..... | | | 8.0 | 8.23 | | |
| Total light oils— | | | | | | |
| From converter..... | | 27.52 | | 27.42 | | 23.21 |
| As % of net oil through converter..... | | 28.0% | | 24.8% | | 25.6% |

TABLE XIII
Summary of Converter Efficiency

| Run No. | Imperial gallons at 60°F. | | | |
|------------------------------------|---------------------------|-------|-------|-------|
| | 6 | 7 | 8 | Total |
| Net raw oil through converter..... | 97.7 | 110.3 | 90.7 | 298.7 |
| Net light oil obtained..... | 27.5 | 27.4 | 23.2 | 78.1 |
| Percentage net light oil..... | 28.0% | 24.8% | 25.6% | 26.1% |

TABLE XIV
Analyses of Samples. Run No. 6

| Lab. No. | Name of product | Imp. gals. at 60°F. | Sp. Gr. at 60°F. | Deg. Be. | Light oil below 350°F. | |
|----------|---------------------------|---------------------|------------------|----------|------------------------|------------|
| | | | | | % Vol. | Imp. gals. |
| 2424 | Raw gas oil charged..... | 217.5 | 0.888 | 27.7 | 0 | 0 |
| 2433 | Still 11 bottoms..... | 113.6 | 0.914 | 23.2 | 0 | 0 |
| 2428 | Drips from converter..... | 6.2 | 0.869 | 31.1 | 0 | 0 |
| 2429 | Tower 1 bottoms..... | 4.0 | 0.915 | 23.0 | 5 | 0.92 |
| | Tower 2 bottoms..... | 14.4 | | | | |
| | Tower 2 loss..... | 10.5 | 0.859 | | 6 | 0.63 |
| | Separator No. 1..... | | | | | |
| 2427 | Oil charged..... | 33.0 | 0.836 | 37.5 | 68.0 | 22.44 |
| 2430 | Oil withdrawn..... | 47.1 | 0.839 | 36.9 | 70.5 | 33.20 |
| | Net oil—separator 1..... | 14.1 | | | | 10.76 |
| 2438 | Compression oil..... | 2.3 | 0.797 | 44.1 | 100.0 | 2.30 |
| | Separator No. 2— | | | | | |
| 2424 | Oil charged..... | 33.0 | 0.829 | 38.0 | 31.0 | 10.23 |
| 2431 | Oil withdrawn..... | 89.0 | 0.831 | 38.5 | 26.0 | 23.14 |
| | Net oil—separator 2..... | 56.0 | | | | 12.91 |
| | Scrubber oil— | | | | | |
| 2435 | Tank 4, before run..... | 1,061 | 0.890 | 27.3 | 0 | 0 |
| 2436 | Tank 3, after run..... | 1,016 | 0.889 | 27.5 | 0 | 0 |

Unstripped gas = 7,910 cu. ft. Density = 0.74 (air = 1).

Stripped gas = 3,766 cu. ft. Density = 0.56 (air = 1).

TABLE XV
Analyses of Samples. Run No. 7

| Lab. No. | Name of product | Imp. gals. at 60°F. | Sp. Gr. at 60°F. | Deg. Be. | Light oil below 350°F. | |
|----------|---|---------------------|------------------|----------|------------------------|------------|
| | | | | | % Vol. | Imp. gals. |
| 2448 | Raw gas oil charged..... | 192.0 | 0.889 | 27.5 | 0 | 0 |
| 2449 | Still 11 bottoms..... | 06.0 | 0.935 | 19.7 | 0 | 0 |
| 2450 | Drips from converter..... | 15.7 | 0.886 | 28.0 | 0 | 0 |
| 2460 | Tower 1 bottoms..... | 17.4 | 0.943 | 18.5 | 0 | 0 |
| 2461 | Tower 2 bottoms..... | 38.9 | 0.889 | 27.5 | 14.0 | 5.45 |
| | Separator No. 1— | | | | | |
| 2451 | Oil charged..... | 33.3 | 0.796 | 45.9 | 78.0 | 29.97 |
| 2452 | Oil withdrawn..... | 46.0 | 0.796 | 45.9 | 78.0 | 36.18 |
| | Net oil—separator 1..... | 12.7 | | | | 6.21 |
| 2455 | Compression oil..... | 1.1 | 0.766 | 52.8 | 100.0 | 1.10 |
| | Separator No. 2— | | | | | |
| 2453 | Oil charged..... | 33.3 | 0.805 | 43.9 | 77.0 | 25.64 |
| 2454 | Oil withdrawn..... | 40.6 | 0.795 | 46.1 | 79.0 | 32.07 |
| | Net oil—Separator 2..... | 7.3 | | | | 6.43 |
| | Scrubber oil— | | | | | |
| 2456 | Tank 4, before run..... | 815.0 | 0.890 | 27.3 | 0 | 0 |
| 2457 | Tank 3, after run..... | 823.0 | 0.890 | 27.3 | 1.0 | 8.23 |
| | Unstripped gas ¹ = 4,357 cu. ft. | | | | | |
| | Stripped gas = 2,194 cu. ft. Density = 0.685 (air = 1). | | | | | |

¹ Sample bottle broken in transit.

TABLE XVI
Analyses of Samples. Run No. 8

| Lab. No. | Name of product | Imp. gals. at 60°F. | Sp. Gr. at 60°F. | Deg. Be. | Light oil below 350°F. | |
|----------|--|---------------------|------------------|----------|------------------------|------------|
| | | | | | % Vol. | Imp. gals. |
| 2462 | Raw gas oil charged..... | 177.0 | 0.890 | 27.3 | 0 | 0 |
| 2463 | Still 11 bottoms..... | 70.0 | 0.929 | 20.7 | 0 | 0 |
| 2466 | Drips from converter..... | 16.3 | 0.881 | 28.9 | 0 | 0 |
| 2475 | Tower 1 bottoms..... | 29.2 | 0.890 | 27.3 | 0 | 0 |
| 2476 | Tower 2 bottoms..... | 12.8 | 0.847 | 35.3 | 27.0 | 3.45 |
| | Separator No. 1— | | | | | |
| 2470 | Oil charged..... | 33.3 | 0.797 | 45.8 | 79.0 | 26.31 |
| 2471 | Oil withdrawn..... | 39.1 | 0.800 | 45.0 | 78.0 | 30.49 |
| | Net oil—separator 1..... | 5.8 | | | | 4.18 |
| 2474 | Compression oil..... | 1.0 | 0.762 | 53.7 | 100.0 | 1.00 |
| | Separator No. 2— | | | | | |
| 2472 | Oil charged..... | 33.3 | 0.795 | 46.1 | 77 | 25.64 |
| 2473 | Oil withdrawn..... | 98.1 | 0.821 | 40.5 | 41 | 40.22 |
| | Net oil—separator 2..... | 64.8 | | | | 14.58 |
| | Scrubber oil— | | | | | |
| 2464 | Tank, 4 before run..... | 811 | 0.891 | 27.1 | 0 | 0 |
| 2465 | Tank, 4 after run..... | 725 | 0.892 | 27.0 | 0 | 0 |
| | Unstripped gas ¹ = 2,840 cu. ft. (170 lbs.) | | | | | |
| | Stripped gas ¹ = 758 cu. ft. (41 lbs.) | | | | | |

¹ Samples not taken.

TABLE XVII
Analyses of Oils. Run No. 6

| | Sp. Gr. at 60 °F. | Distillation Range | | | | |
|-------------------------|-------------------------|--------------------|------------|------------|------------|------------|
| | | 1st drop | 20% °F. | 50% °F. | 90% °F. | End °F. |
| Raw gas oil..... | 0.888 | 304 | 460 | 520 | | |
| Tower 1 bottoms..... | 0.915 | 286 | 417 | 462 | 576 | 628 |
| Tower 2 bottoms..... | | 149 | 228 | 300 | 419 | 482 |
| Separator 1 before..... | 0.836 | 149 | 232 | 298 | 404 | 474 |
| Separator 1 after..... | 0.839 | 392 | 502 | | | |
| Still 11 bottoms..... | 0.914 | 113 | 302 | 408 | 505 | 534 |
| Separator 2 before..... | 0.829 | 117 | 324 | 414 | 513 | 563 |
| Separator 2 after..... | 0.831 | 115 | 167 | 196 | 291 | 333 |
| Pressure tank..... | 0.797 | 374 | 457 | 513 | | |
| Still 12 bottoms..... | 0.890 | | | | | |

TABLE XVIII
Analyses of Oils. Run No. 7

| | Sp. Gr. at 60 °F. | Distillation Range | | | | |
|-------------------------|-------------------------|--------------------|------------|------------|------------|------------|
| | | 1st drop | 20% °F. | 50% °F. | 90% °F. | End °F. |
| Raw gas oil..... | 0.891 | 331 | 462 | 516 | | |
| Tower 1 bottoms..... | 0.943 | 367 | 457 | 500 | | |
| Tower 2 bottoms..... | 0.889 | 221 | 379 | 437 | 527 | |
| Separator 1 before..... | 0.796 | 163 | 230 | 286 | 383 | 424 |
| Separator 1 after..... | 0.796 | 113 | 219 | 280 | 383 | 428 |
| Still 11 bottoms..... | 0.935 | 525 | 574 | | | |
| Separator 2 before..... | 0.804 | 97 | 248 | 295 | 401 | 462 |
| Separator 2 after..... | 0.795 | 104 | 232 | 282 | 399 | 459 |
| Pressure tank..... | 0.767 | 95 | 162 | 194 | 298 | 324 |
| Still 12 bottoms..... | | | | | | |

TABLE XIX
Analyses of Oils. Run No. 8

| | Sp. Gr. at 60 °F. | Distillation Range | | | | |
|-------------------------|-------------------------|--------------------|------------|------------|------------|------------|
| | | 1st drop | 20% °F. | 50% °F. | 90% °F. | End °F. |
| Raw gas oil..... | 0.890 | 325 | 464 | 523 | | |
| Tower 1 bottoms..... | 0.890 | 399 | 451 | 484 | 563 | |
| Tower 2 bottoms..... | 0.847 | 205 | 333 | 385 | 455 | |
| Separator 1 before..... | 0.797 | 131 | 223 | 282 | 387 | 424 |
| Separator 1 after..... | 0.800 | 131 | 225 | 286 | 392 | 448 |
| Still 11 bottoms..... | 0.929 | 491 | 554 | | | |
| Separator 2 before..... | 0.795 | 108 | 234 | 284 | 408 | 464 |
| Separator 2 after..... | 0.821 | 129 | 282 | 378 | 500 | 559 |
| Pressure tank..... | 0.762 | 85 | 147 | 187 | 338 | 338 |
| Still 12 bottoms..... | 0.892 | 361 | 460 | 525 | | |



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